

THERMAL, OXIDATIVE AND HYDROLYTIC STABILITY OF SELECTED  
FRYING SHORTENINGS EVALUATED BY NEW AND CONVENTIONAL  
METHODS

BY

Nabil A. Bati

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APPROVED:

-----

George J. Flick, Jr., chairman

-----

William L. Baran

-----

James K. Palmer

-----

William E. Barbeau

-----

Paul P. Graham

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(ABSTRACT)

The thermal, oxidative and hydrolytic stability of several frying shortenings were studied via chemical, physical and sensory analyses. Corn, cottonseed and peanut oils, and cottonseed and soybean liquid shortenings were tested under static heating conditions, while peanut oil, and cottonseed and soybean oil liquid shortenings were evaluated under commercial frying conditions.

The research had two objectives: to evaluate the relative stability of the various shortenings under both heating condition; and to evaluate new or modified quality assessment methods which would provide early prediction of heat abuse for the fast food industry.

Six of the conducted analyses were conventional or modified: free fatty acids; polar components; gas chromatograph volatile profiles; viscosity; FoodOil-Sensor; and sensory. Three were new: contact angle; high temperature; and high temperature gas chromatographic analysis of triglyceride; and polar component% as determined by high performance thin layer chromatography (HPTLC).

Under static heating conditions, varying heating periods or shortening types had significant ( $P < 0.0001$ ) effects on the resulting

data of the following tests: free fatty acids; polar component; total volatiles; dielectric constant; viscosity; polar component% measured by HPTLC; contact angle; and sensory analysis; but heating time had no significant effect on triglyceride profiles.

Under commercial frying conditions of chicken nuggets and filets, heating time had significant effects on changes in the dielectric constant; free fatty acid%; viscosity; contact angle; and sensory rating; also it had a significant effect on the polar component% under chicken nugget frying conditions only. Furthermore, heating time had no significant effect on polar component% under chicken filet frying conditions and on polar component% by HPTLC under both frying conditions.

Cottonseed oil liquid shortening had sensory scores equal to peanut oil under static and commercial frying conditions even though peanut oil exhibited a greater chemical and physical stability. Soybean oil liquid shortening had an objective quality identical to peanut oil, however, its subjective quality was lower. Cottonseed oil liquid shortening had better flavor but less objective stability than soybean oil liquid shortening.

The cut-off quality level for the shortenings was not reached, because all the shortenings were discarded after seven days of use which was before the onset of significant quality deterioration.

The best on-site index of shortening stability was the FoodOil-Sensor reading (dielectric constant) which was followed by the free fatty acid test.

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## CHAPTER I

### INTRODUCTION

Deep fat frying is one of the most important processes used worldwide for the preparation of foods just prior to their consumption.

The constant search for interchangeable, competitively priced frying oils has been one of the major concerns of restaurants. The use of hydrogenated shortenings or vegetable-animal fat blends has resulted in the improvement of frying medium stability. However, the presence of either trans and saturated fatty acids as a result of hydrogenation, or the presence of cholesterol and saturated fatty acids due to blending with animal fat has resulted in a decrease in consumer acceptability. Consequently, restaurants have realized that a failure to provide consumers with products that have desirable lipid compositions will result in product rejection and reduced profitability.

Assessment of frying oil deterioration has been a subject of much concern because of the high volume of fried food products being produced. It is important for the food industry to have simple, low cost objective methods for evaluating frying oil quality, as premature discarding of frying oil is an economic disadvantage while over use of

the frying fat can affect product quality, and consumer acceptability.

Desirable textures and flavors can be generated during the frying operation as a result of Maillard reactions, dehydration and oil absorption (Stevenson et al., 1984b). However, degradation can occur due to hydrolysis, autoxidation, thermal polymerization, thermal oxidation and isomer cyclization reactions (McNeill et al., 1986) when the oil is used repeatedly at high temperatures in the presence of air and moisture. The rate of fat deterioration is directly proportional to the specific surface area - ratio of oil surface to oil volume - (Rock and Roth, 1964). Food components may enhance the degradation processes. The oxidative stability of oil was influenced by iron (Min and Wen, 1983), Phospholipids (Min and Stasinopoulous, 1983 and Hildebrand et al., 1984), free fatty acids and mono-and diglycerides (Mistry and Min 1987a,b). Thompson and Aust (1983) reported that the quantity of food fried contributes to the decreased quality of a frying oil. Baran (1984) suggested that the amount of moisture, oxygen, salts, metal contamination and types of foods may have an effect on fat breakdown. Furthermore, Billek (1983) claimed that the rate of fat deterioration will vary with the type of operation; industrial (continuous) deep-fat frying manifests no problem with fat deterioration as compared to household or commercial

(batch) deep fat frying. High frying temperatures (above 325°F), in addition to their effect on the degradation reactions, may also influence chicken yield (Yang and Chen, 1979). According to McGill (1980), fatty foods, wet foods and foods that easily break apart will accelerate the chemical changes of a frying oil. White and Wang (1986) reported that breaded vegetables likely enhanced the deterioration of soybean oil even though it contained BHA, BHT, citric acid and methyl polysiloxane. Thompson and Aust (1983) also reported that the quantity of food fried contributes to the decreased quality of a frying oil.

During frying, the oil itself will undergo a number of physical and chemical changes, such as darkened color, increased viscosity and foaming, and decreased smokepoint accompanied by the formation of free fatty acids, hydroperoxides, carbonyls and high molecular weight polymers (Tangle et al., 1977). These compounds may cause an increased absorption of oil into the fried food; hence decreasing food quality (Meltzer et al., 1981) and resulting in decreasing the concentrations of these compounds, particularly polymers (Alim and Morton, 1974).

Furthermore, it is important to realize that as a result of oxidation, polymerization and browning, shortening color changes from light yellow to an orange or brown color. Roth and Rock (1972a) reported that oil darkening is due to the presence of certain unsaturated

molecular groups. Traces of iron and some other metallic contaminants (King and Wharton, 1949), phenolic antioxidants (Sherwin and Thompson, 1967) and products of caramelization (Orthoefer, 1988) have been known to cause undesirable darkening of fat and oil during frying.

In general, both volatile and non-volatile compounds are formed by the degradation reactions. Orthoefer (1988) suggested that flavor compounds are generated from the heat effects on proteins, carbohydrates, lipids and minor constituents present in the fried food. However, most of the volatile compounds are distilled during the frying process (May et al., 1983). The non-volatile decomposition products are produced primarily by thermal oxidation and polymerization of unsaturated fatty acids (Paulose and Chang, 1978) and triglycerides (Perrin et al., 1983). These polymers may accumulate in the frying oil to promote further degradation and may be absorbed by the fried food and enter the diet of the consumer (Stevenson et al., 1984b). The lipid content of various deep-fat fried foods was reported by Smith et al. (1985). The possible toxic effects of degradation products from several oils was reported by many researchers (Crampton et al., 1952 and 1956; Ohfuji and Kaneda, 1973; Landers and Rathmann, 1981; and Hurrell and Nielsen, 1987), even though deep-fat fried foods had little or no mutagenic activity (Taylor et al., 1983).



Several analytical procedures to evaluate the extent of lipid oxidation were reviewed by Gray (1977).

Stevenson et al. (1984b) reported that regular cleaning and maintenance of equipment and utilization of proper conditions are required to decrease the chemical and physical changes due to frying and to prolong the useful life of the shortening. Weiss (1983) suggested that oil should be filtered at least daily. Food particles, if not removed by filtration, may burn and develop undesirable flavors and odors and increase the rate of oil deterioration which would affect the flavor of the finished products (Jacobson, 1967). Moreover, Weiss (1983) indicated that 15-25% fresh fat should be added daily to extend shortening frying life. Furthermore, additives are used to improve the frying life of shortenings, such additives are BHA, BHT, propyl gallate, methyl polysiloxane and citric acid (Sherwin, 1972), TBHQ (Sherwin, 1967), methyl polysiloxane (Freeman et al., 1973), ascorbyl palmitate (Gwo et al., 1985) or ascorbic acid (Cort, 1973) and (Unda et al., 1986). Also, Franckel et al. in 1985 reported that TBHQ, citric acid and dimethylpolysiloxane, had a synergistic effect in lowering the rancid odor score of soybean oil. Asap and Augustin (1986) found that the four breakdown products of TBHQ had an antioxidant effect, even though the Refined, Bleached and Deodorized (RBD) olein was darker in color. Walting and Zmachinski (1970)

claimed that cooking a food in frying oil produce a steam layer which can decrease the deterioration rate of a frying oil. Frying methods may affect the rate of oil replenishment hence its stability; Mostert and Stadelman, in 1964, found that pressure-fried chicken samples tended to have a higher moisture percentage and a lower fat percentage than crispy, open-vat fried samples. The objective and subjective performance of different oils may vary with their degree of unsaturation. Erickson (1983) did not recommend the use of highly polyunsaturated oils such as safflower, sunflower, corn, rapeseed and soybean oils, for heavy-duty deep-fat frying. Nevertheless, Huang et al. (1981) considered corn and sunflower oils suitable for cooking or frying without hydrogenation, because they contain practically no linolenic acid when compared with soybean oil.

The first question is, "Is there a low cost substitute oil for the following situations?".

1. Expensive oils.
2. Variable supply due to environmental conditions such as droughts or floods.
3. Consumer concerns about report on nutritional quality and food safety.
4. International conflicts which may disrupt distribution channels.

The second question is, "How does one know when the frying fat needs to be discarded?". It is difficult to answer this question for several reasons:

1. There is no adequate On-Site test to evaluate oil degradation.
2. It is much easier to conduct an experiment in the laboratory as compared to the real world (restaurant).
3. Different foods have different effects on oil deterioration, hence, the cut-off level.
4. Different oils and variations in the same oil, which are due to differences in growing seasons and processing operations.
5. Different fryer designs, which may effect a specific surface area and heat transfer efficiency.
6. Different operating conditions.

#### OBJECTIVES:

The major objectives of this study were to:

1. Investigate whether different oils and liquid shortenings are interchangeable under different frying conditions.
2. Evaluate chemical, physical and sensory properties of different frying shortenings in order to determine their maximum useful life.
3. Study the possible exchange between frying oil and chicken fat and the effect of this change on oil stability.

4. Identify and compare the effects of heating time, heating time and product volume, and product form under commercial frying conditions on the stability of selected frying media.

5. Develop rapid- or economical- On-Site quality tests for frying shortenings and compare the results with conventional tests.

In the present study, five types of commercially used frying shortenings were heated under static conditions and evaluated for chemical stability and sensory perception. Three of these oils have been previously used for the commercial frying of chicken nuggets and filets.

A total of ten conventional and new tests were performed on the shortenings under static heating conditions, and nine of these tests were studied under commercial frying conditions.

## CHAPTER II

### LITERATURE REVIEW

The useful life of a frying shortening is directly influenced by the its rate of degradation. Therefore, the chemical and sensory quality of the shortening used in frying will affect the quality of the cooked product and its ultimate acceptability. The useful life of a shortening is reflected through the interaction of heating conditions, shortening type, product type and volume and quality evaluation procedures.

#### 2.1 EFFECT OF STATIC HEATING AND COMMERCIAL FRYING CONDITIONS ON SHORTENING QUALITY.

The importance and popularity of deep-fried foods are due essentially to the basic distinctive structure and flavor imposed on them by the way in which they are cooked, and to the high efficiency and speed of their preparation. The necessity of using a good quality frying medium and of maintaining it in that state as long as possible becomes obvious when one considers that some of the oil is absorbed by every piece of food fried in it. Therefore, it is necessary to investigate some of the changes, which occur

in frying oils during their use in order to establish On-Site test(s) which can provide more accurate information about the oil quality in a restaurant.

During frying, numerous and complex chemical reactions such as polymerization, hydrolysis, oxidation and pyrolysis occur in the frying medium which lead to the production of degradation products (Fritsch, 1981). These decomposition products affect the quality of both frying oil and fried foods. Fig. 1 shows some of the known changes that occur under deep fat frying conditions. These products are probably more complex under commercial frying conditions as compared to static heating conditions. Although considerable attention has been given to the evaluation and assessment of oil degradation, little is known about the mechanism of oil degradation under different frying conditions. A summary of known lipid degradation mechanisms was reviewed by Frankel (Frankel, 1980).

It would be expected that factors affecting the rate of deterioration of deep frying shortenings used to prepare chicken products, such as nuggets and filets, are different from those affecting the shortening under static heating conditions. During the cooking of coated (battered or breaded) chicken nuggets or filets, carbohydrates, proteins, lipids, minerals, water, air and other ingredients are introduced into the shortening. Longer cooking periods at a specific temperature might be an

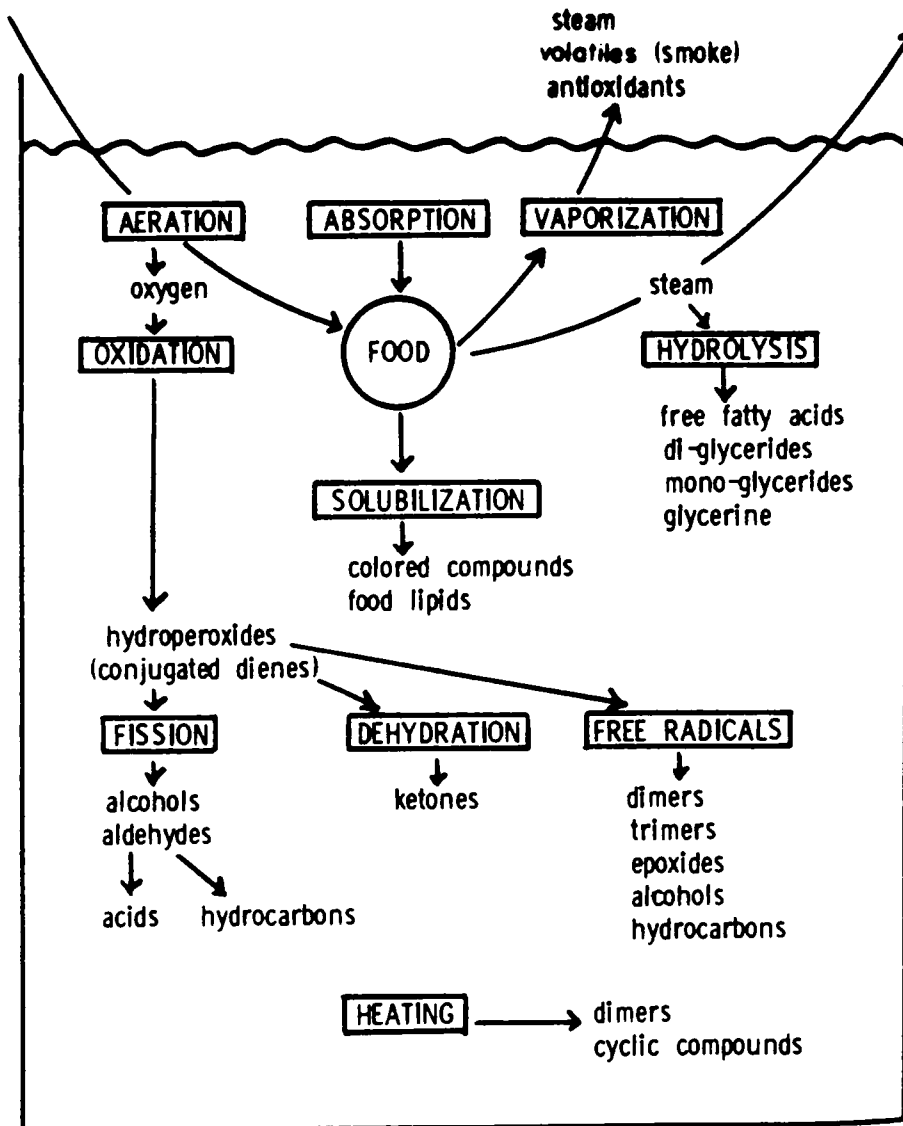


Fig. 1. Changes occurring during deep fat frying (Fritsch, 1981).

important factor in controlling the rate at which these added ingredients participate in the decomposition and deterioration of the frying material, particularly triglycerides which are the major constituents of any shortening.

Orthoefer (1988) reported that the deterioration of frying fats is familiar to all users of frying oils. According to Erickson, primary oil deterioration includes hydrolysis, oxidation and caramelization (Orthoefer, 1988). Perkins noted that further degradation of the oil results in oxidation and polymerization to promote further darkening, off-flavor development and foaming (Orthoefer, 1988).

During static heating, temperature of the medium, heating period and, to a lesser extent, air and surface to volume ratio of the fryer are probably the only factors that affect the rate of deterioration. Consequently, the degradation products might be less complex than those of the commercial frying situation. Moreover, none of the degradation products are reduced in concentration by absorption since no food or shortening is added. This particular point could be important when a new method is needed to evaluate the quality changes of a frying shortening, because heating time is the only independent variable affecting changes in oil quality. Stevenson et al. (1984b) claimed that there is a need for simple,



reliable methods to rapidly identify frying oil quality. Commercially, used frying shortening is a dynamic system with many complex reactions. The accumulation of particular reaction products depends upon their rates of synthesis, absorption by food, as well as, their rate of participation in further reactions. Alim and Morton (1974) and Shahasrabudhe and Bhalerao (1963) have found that the oil removed from a fried food contains greater amounts of polymers than does the oil in which it was fried. Hence, any objective analysis could be under or overestimating these particular degradation products reducing the effectiveness of laboratory type On-Site evaluations, and creating difficulties in finding one universal test that can be applied on different foods and oils. Consequently, the detection method on the sample used may no longer be representative of the actual population (heating medium).

To avoid this problem, all analyses must be conducted in the restaurant immediately after sampling which is not feasible to do.

#### QUALITY CONTROL TESTS.

Various criteria may be used to determine when to discard the oil in a frying vessel (Table 1).

Although TBA and peroxide values are considered among the more popular tests to evaluate rancidity in food products, they are not recommended to evaluate progress of

rancidity in shortenings during frying. Peroxides are very unstable at frying temperatures; their concentration will peak then decrease over the entire frying period (Gray, 1978). Also, TBA tests provided poor correlation with the length of deep frying time (Stevenson et al., 1984a). Conjugated Diene and Triene was not considered because the increase in UV light absorption is difficult to relate to the degree of oxidation because the rates of oxidation vary with the different types of polyunsaturated fatty acids (Jackson, 1981). Furthermore, the greatest changes in Diene content was found during the first day of the test (Fritsch, 1981). Foam hight could not be used to evaluate oil degradation since dimethylpolysiloxane (antifoaming agent) was added to suppress foaming (Chang et al., 1978). Smoke point was not considered because any variation in room temperature may cause up to a 26<sup>o</sup>C variation for single fat (Stevenson et al., 1984). Other analytical methods were not considered due to their cost, relative complexity, inappropriateness, or their ability to provide additional information over the selected procedures.

There are several reasons for developing new procedures to evaluate frying oil quality:

1. Although polar component percent evaluated by the open column chromatography procedure is considered the most reliable test to measure the extent of fat deterioration, the test has major disadvantages since it requires lengthy

procedures and high solvent consumption. These problems were eliminated by the application of Quantitative High Performance Thin Layer Chromatography.

2. The change in triglyceride concentrations in frying oils during heating was investigated because of a possible exchange between the frying medium and the product being prepared. It is possible that different triglycerides will have varying thermal stabilities.

3. The contact angle test was developed as an On-Site test to evaluate frying oil quality in a short period of time since On-Site tests which are currently available to the food service industry cannot be used under all frying conditions.

4. The FoodOil-Sensor procedure (measures dielectric constant) was modified to more accurately indicate shortening freshness, as well as, the dielectric constant of the frying oil. Furthermore, this test is considered to be one of the most recommended On-Site procedures in fast food restaurants.

Table 1. Quality control tests of deep-frying oils available to restaurants and food service institutions.

| Physical tests  | References                              |
|---|---|
| Without laboratory facilities <sup>1</sup> :  |   |
| Color, foaming, smoking and odor of frying fat  | :Landers and Rathman (1981).            |
| Changes in dielectric constant  | :Graziano (1979).                       |
| Length of fat use, and sensory evaluation of cooked product for flavor, odor and texture. | :Ylimaki and Vaisey-Genser (1981).      |
| With laboratory facilities <sup>2</sup> :   |   |
| Smoke point and color   | :Weiss (1983).                          |
| Foam height   | :Fritsch et al. (1979).                 |
| Viscosity and Refractive Index  | :Alim and Morton (1974).                |
| Sensory evaluation of cooked product for flavor, odor and texture                         | :Jacobson (1967).                       |
| Chemical tests  | References                              |
| With laboratory facilities <sup>2</sup> :   |   |
| FFA   | :Weiss (1983).                          |
| TBA   | :Jacobson (1967).                       |
| Peroxide value and petroleum ether insoluble  | :Billek et al. (1978).                  |
| Iodine number, Active Oxygen Method and Non-Urea adduct fraction                          | :Alim and Morton (1974).                |
| Dienes  | :Fritsch et al. (1979).                 |
| Carbonyls and Anisidine value   | :Fritsch (1981).                        |
| Total polars  | :Paquot (1982).                         |
| <sup>1</sup>  | Restaurants, food service institutions. |
| <sup>2</sup>  | Food processors.                        |

### 2.1.1 FREE FATTY ACIDS (FFA-% OLEIC ACID).

Free fatty acids are among the degradation products that accumulate in shortenings during heating or frying processes (Meltzer et al., 1981), and are responsible for many physical and chemical changes that occur, such as polymerization (Paulose and Chang, 1978). The content of free fatty acid is measured by titration of the tested shortening with sodium hydroxide of specific normality and expressed as oleic acid percent.

Considering that FFA are one of the polar components found in heated shortening, Stevenson et al. (1984b) reported a high correlation between the percent FFA or polar compounds and length of frying time. FFA are formed during frying due to the presence of moisture in the foods. A free fatty acid level of 0.05-0.08% is normal for most frying fats (Weiss, 1983).

Bauerlen et al. (1968) reported that FFA appears to be the most favored method for quality control evaluation of frying fats. However, this method not only reflects the FFA formed by hydrolysis and oxidation during the frying process but also the level of FFA initially present in the fat before heating.

### 2.1.2 POLAR COMPONENT TEST (OPEN COLUMN CHROMATOGRAPHY).

Polar compounds are generated as a result of shortening degradation in the presence of air, heat and/or moisture (McNeil et al., 1986) and can be isolated from used frying shortening as a brownish, transparent, viscous liquid (Chang et al., 1978). A collaborative study on the determination of polar compounds in frying fats sponsored by the International Union of Pure and Applied Chemistry (IUPAC) published by Wessels (1983) reported that thermal degradation leads to thermal dimeric triglycerides, polymers and cyclic acids. Oxidative degradation may produce oxidized triglycerides containing hydroperoxide-, epoxy-, hydroxy- and keto- groups and also dimeric fatty acids or dimeric triglycerides linked by one or more oxygen atoms. Volatile degradation products are saturated and monounsaturated, hydroxy-, aldehydic-, keto- and dicarboxylic-acids; hydrocarbons; alcohols; aldehydes; ketones; and aromatic compounds. Hydrolytic degradation processes yield diglycerides and free fatty acids.

The polar component percent can be determined by removing the nonpolar fraction from the original sample using a relatively nonpolar solvent. Silica gel inside the column will retain the polar fraction after elution is completed. By calculating the difference between the weight of the sample added to the column and that of the nonpolar fraction eluted, the percent of polar components is determined. The advantage is that only one fraction,

which contains the nonpolar compounds, is eluted. Direct determination of polar compounds results in an underestimation of their concentration because of incomplete elution of the highly polar compounds (Wessels, 1983). The polar component test is one of the more complicated, time consuming tests, and tends to monitor quality changes more accurately than other procedures. Billek et al. (1978) concluded from a comparison study that this method provided the best reproducibility when compared with: petroleum ether insoluble oxidized fatty acids; gel permeation chromatography; and liquid chromatography. In the same study, they reported that 15% polymeric triglycerides determined by gel permeation chromatography corresponded to 27% polar compounds. Paradis and Nawar (1981a,b) recommended that 27% polar components from column chromatographic analysis be the maximum level for maintaining frying oil quality. Also, Wu and Nawar (1986) reported that the polar components in frying fats are highly correlated with FoodOil-Sensor (FOS) readings. Fritsch (1981) reported that the determination of total polar components is a complex procedure, requires highly skilled personnel, is time consuming, and that the test is more suited for use by regulatory agencies where precision is important.

### 2.1.3 GAS CHROMATOGRAPHY - VOLATILE PROFILE ANALYSIS (GC-VPA).

Volatile oxidation products in lipids are derived from the breakdown of monohydroperoxides and the secondary reactions of unsaturated aldehydes. Furthermore, the secondary nonvolatile products of hydroperoxides can decompose and contribute significant amounts of volatile oxidation products (Frankel, 1982).

Autoxidation of oils is affected by atmospheric oxygen and the oxidation process is initiated by radical reactions involving unsaturated fatty acids. The primary products formed are hydroperoxides and the secondary products include alcohols and carbonyl compounds (Gunstone, 1984; Frankel, 1984). These products can subsequently be oxidized further to carboxylic acids (Loury and Forney, 1969).

Many volatiles are formed during autoxidation of unsaturated lipids as a result of heat and their odor and taste will influence the flavor of fried foods and the frying fat. Weiss (1983), Robertson (1968) and May et al. (1983) have reported that most of the volatiles are removed from the frying medium by steam generated during frying.

Various gas chromatographic (GC) methods have been reported to analyze oxidation volatiles produced from the oxidative deterioration of vegetable oils (Evans et al.,



1969; Selke et al., 1970; Selke and Frankel, 1987; Dupuy et al., 1971a,b; Hartman et al., 1971). These methods have been reviewed by Waltring and Goetz (1983) and Snyder et al., (1985), who indicated a serious lack of quantitative quality studies using fats of different origins.

Consequently, they attempted to develop a reproducible capillary GC method to analyze headspace volatiles in commercially processed canola, corn, cottonseed, olive, peanut, safflower, soybean and sunflower seed oils, and to identify specific volatile components using GC/MS analysis. They found that the volatile compounds present in each stored vegetable oil sample were related to the main fatty acid components of the oil. Pentane and hexanal are particularly produced in high amounts from safflower, sunflower seed, corn and cottonseed oil because they have a high quantity of linoleate. Canola and soybean oils, which contain linolenate, both formed measurable amounts of 2, 4-heptadienal. Olive oil, with the largest quantity of oleate, produced the most octanal and nonanal. Warner and Frankel (1985) tested a direct GC method to predict flavor stability of soybean oil by increasing induction periods based upon the time required for rapid formation of volatile compounds and compared their results with a headspace technique (Warner et al., 1974; Fiority et al., 1974). The direct GC method was preferred for the early detection of changes in flavor deterioration.

The advantages and disadvantages of static versus dynamic headspace techniques have been reviewed recently by McNally and Grab (1985). In general, both dynamic headspace and static headspace have identical detectable limits. However, the dynamic headspace technique is more sensitive than the static headspace method, while, the static headspace technique is two or three times faster than the dynamic headspace method.

The American Oil Chemists' Society (AOCS) Flavor Nomenclature Committee conducted a collaborative study to evaluate GC volatile profiles for chromatography magnitude of oil flavor (Waltking, 1982). The study concluded that the GLC procedures were more precise than flavor panels. However, separate correlation equations must be developed for each type of oil and for each type of storage or abuse procedure.

#### 2.1.4 DIELECTRIC CONSTANT.

The FoodOil-Sensor System (FOS) measures changes in the dielectric constant of an oil or shortening, however, it does not provide absolute values which are more representative of polarity% in a frying shortening. Stevenson et al. (1984a) suggested that the FOS is suited to monitoring operations where frying time is the only

variable. Fritsch et al. (1979) reported that "changes in the dielectric constant of oil is a good measure of oil deterioration during frying or exposure to high temperatures for extended periods. Oxidation products were primarily responsible for this change.. ."

Graziano (1979) reported that oil degradation during the frying process results in a dielectric constant increase as a result of polar compound accumulations. Paradis and Nawar (1981a,b) and Augustin et al. (1987) showed high correlations between polar compound accumulation and the FoodOil-Sensor. However, they added that water and food fats may lead to spurious instrumental readings. A similar finding was reported earlier by Stevenson et al. (1984b). For this reason, samples should not be taken from the fryer while the food is being prepared (Fritsch et al., 1979).

Griffith et al. (1986) found good correlations ( $R=0.84$ ) between frying times and increases in the dielectric constant under actual operating conditions for 100 interrupted hours of frying with oil replacement. Their dielectric constant readings were also correlated with polar materials ( $R=0.95$ ) and free fatty acids ( $R=0.92$ ).

Also, Smith et al. (1986) found that frying times were highly correlated with increases in dielectric constants, polar materials and FFA. There were high correlations

among increases in dielectric constants, percentages of polar materials and FFA for partially hydrogenated soybean oil used to fry battered chicken parts and French fries.

Paradis and Nawar (1981a) reported that FOS instrument values represent the net balance between polar and nonpolar materials. Positive changes were assumed to be caused by moisture, oxygenated dimeric material, polar volatiles and diglycerides; negative values resulted from free fatty acids, hydrodimer acids and nonpolar volatile compounds.

#### 2.1.5 VISCOSITY.

Thermal oxidation and polymerization of unsaturated fatty acids (Perkins, 1967; Paulose and Chang, 1978) and triglycerides (Perrin et al., 1983) result in the formation and accumulation of nonvolatile decomposition products which are responsible for an increase in viscosity. Paradis and Nawar (1981a) suggested that the higher molecular weight compounds are more reliable indicators of fat abuse as their accumulation is steady and they have low volatility.

The presence of polymers in fat is often shown by the non-urea adduct forming fraction. Published values range from less than 1% to over 7% for frying fats from commercial sources as measured by Rock and Roth (1966)

using the method of Firestone et al. (1961) or Shahasrabudhe and Bhalerao (1963). Furthermore, Rock and Roth (1966) correlated the percent non-urea adducting fraction with viscosity. Thus, the use of relative simple viscosity tests would enable the processors to monitor their fats for non-adducting material with a minimum of time, equipment and technical training (Jacobson, 1967).

Several researchers have studied the nature of the polymerization reaction products formed during thermal treatment. From early studies, Chang and Kummerow (1953) and Witting et al. (1957), proved the formation of significant amounts of polar and polymeric materials from the autoxidation of linoleate and linolenate.

Perkins and Van Akkeren (1965) investigated the effects of deep fat frying processes on the formation of polymeric and oxidized materials in cottonseed oil under practical conditions. Their results indicate that the amount of polymeric material formed increased regularly as heating time increased. Also, they confirmed that foaming is related to the formation of oxidation products of high molecular weight compounds. Perkins (1967) obtained fats from fryers in several restaurants and examined them for their "polymer" content. The non-distillable methyl esters of these fats served as an indication of the percentage of non-volatile high-molecular-weight material in the used fat. A "polymer" content of 3-9% was common in corn oil,

lard, hydrogenated shortening and animal fat shortening. The hydroperoxidation of unsaturated fatty esters has been reviewed in detail by Frankel (1962; 1979). Monohydrogen oxides undergo further oxidation and condensation to produce a very complex mixture of monomeric and polymeric polar and nonpolar materials. Ohfuji and Kaneda (1973) reported that a highly toxic material formed in thermally oxidized soybean oil is a dimer of triglyceride molecules.

Paulose and Chang (1978) separated polymers from the nonvolatile decomposition products of treated trilinolein, and through depolymerization techniques they concluded that trimers with carbon to carbon linkages represent 8.4%, dimers and trimers joined through carbon to carbon or carbon to oxygen linkages in the same molecule at 4.9%, and also trimers in which all three monomeric units were joined through carbon to oxygen linkages.

Perkins and Wantland (1973) have attempted to characterize the nonvolatile compounds formed during thermal oxidation of 1-linoleyl-2,3-distearin. They found evidence to indicate the presence of both cyclic and noncyclic dimer species which differ in molecular weight but which have similar structures. Combe et al. (1981) could separate three polymeric acids from heated soybean oil using quantitative thin layer chromatography.

White and Wang (1986) separated polymers formed in heated soybean oils within a 700 to 6000 Dalton range by

using high performance size exclusion chromatography. Similar results were obtained by Perrin et al. (1983) who determined triglyceride polymers from fried sunflower oil using identical methods.

Several researchers have conducted studies concerning the possible absorption of frying oils and their degradation products into the fried food. Stevenson et al. (1984b) suggested that the void in a cooked food created by loss of moisture from the crust is filled largely by absorption of the frying fat. Melnick et al. (1958) concluded that the oils used in the production of potato chips are free of thermal polymers and there was no reason to question the wholesomeness of the oil being absorbed by the chip. However, Meltzer et al. (1981) reported that the formation of free fatty acids, hydroperoxides, carbonyls and high molecular weight polymers cause an increased absorption of oil into the fried food, hence decreasing food quality. From these references, we may suggest that viscosity is not always a good indicator of heat abuse of frying oils. Also, polymer accumulation is not always a good indication of shortening heat abuse.

#### 2.1.6 POLAR COMPONENT% AS DETERMINED BY QUANTITATIVE HIGH PERFORMANCE THIN LAYER CHROMATOGRAPHY (QHPTLC).

Thin layer chromatography (TLC) is considered the most general technique for obtaining a qualitative profile of a total lipid extract (Kuksis, 1978). Usually, the polar and nonpolar lipids are profiled separately using appropriate solvent systems. However, in most nonpolar solvent systems, total polar lipids may be estimated from the material left at the origin during a neutral (non-polar) lipid separation. Freeman (1974) used quantitative TLC to follow the progress of oxidation in frying oils by charring the bands with chromic acid. However, a good correlation with polar component% was obtained with homemade TLC plates only. Furthermore, the use of benzene and diethyl ether as a solvent system to develop the plates is undesirable since they both are carcinogenics. Nevertheless, neither the type of shortenings nor the frying conditions were reported. Charring of separated zones followed by densitometry for quantification is widely practiced (Touchstone et al., 1980). However, effective quantitation of all lipid fractions by ordinary TLC has not been entirely satisfactory. This is partly due to a variability in the resolution of different lipid classes and partly to the variability in response of different lipid classes to charring. Privett and Blank (1962) used TLC to quantitate mono-, di-, and triglycerides using charring procedures. They observed that equal amounts of these compounds produced spots of different intensities on charring far out



of proportion to the difference in their carbon densities. Furthermore, unsaturated glycerides gave much darker spots than saturated glycerides. However, Adams and Selke (1971) sprayed 20% ammonium bisulfate onto the plate and then charred the lipids by heating on a hot plate at 179°C for 1 hr. Scanning with dual-beam spectrodensitometer showed that a linear relationship from 0-10µg of neutral lipids was obtained. Nevertheless, Bitman and Wood (1982) reported that, in practice, it has been difficult to control reaction conditions to produce complete quantitative carbonization.

#### 2.1.6.1 SEPARATION OF POLAR AND NON-POLAR COMPOUNDS.

Review articles concerning developments in the thin layer chromatography of lipids have been written by Hamilton (1986) and Malins (1966). Both of these articles referenced different methods to separate different lipid classes using a variety of techniques. However, the Malins (1966) article is outdated and both articles did not cover the application of (TLC) in evaluating frying oil quality.

Macala et al. (1983) indicated that although High Performance Thin Layer Chromatography (HPTLC) has provided much improved resolution of the lipid classes, the problem of quantitation has not been absolutely solved. Malins (1966) reported that dyes, such as 2',

7'-dichloro-fluorescein and rhodamines, are quite suitable for the nondestructive detection of most lipids. However, no literature citations were found on the application of these dyes in quantitative TLC. In order to obtain reproducible separation of polar and nonpolar fractions of frying shortenings, minimum diffusion (band broadening) should be maintained. Hauck et al. (1987) reported that precoated High Performance Thin Layer Chromatography (HPTLC) plates provide better separation, accuracy and reproducibility than regular Thin Layer Chromatography (TLC) plates because the silica gel used on HPTLC plates have a narrow-particle size distribution, small mean particle diameters and better chemical purity. These characteristics could be advantageous when rapid separation is needed.

Quantitation of polar fractions on thin layer plates require:

1. The selection of suitable solvent systems to separate the nonpolar from the polar fraction;
2. Conversion of the separated colorless bands into charred or fluorescent products; and
3. Quantitative determination of these fractions by densitometric scanning.

Kolaronic et al. (1985) reported that a common technical problem in separating polar lipids (e.g. phospholipids) in thin layer chromatography is always

achieving the same degree of chamber saturation so that reproducibility of results is assured. Yavin and Zutra (1977) and Bens et al. (1982) have shown that chromatographic properties of HPTLC silica gel plates depend upon storage conditions which are usually subject to the variability in atmospheric humidity. Hamilton (1986) showed that under a relative humidity of 50%, an activated plate will lose 50% of its activity in 3 minutes. It is therefore desirable to determine the degree of activity of the silica gel (e.g. by adjusting it to a defined relative humidity) and thus influence the strength of retention of a given sample substance with a defined solvent (Hauck et al., 1987).

#### 2.1.6.2 HPTLC PLATES PREPARATION.

Eng et al. (1964) reported that lipids are particularly susceptible to oxidation when spread as a thin film over a surface. Also, Kohlschutter and Unger (1969) have reported that silica gel itself is an acid, and the possibility cannot be excluded that silicic acid catalyzed hydrolysis of the triglyceride is occurring. In contrast, Badings (1964) has shown that unsaturated fatty acid esters containing from two to four double bonds undergo some oxidation when chromatographed in air, a practice commonly employed in many laboratories. However, no significant

losses were obtained when the same acids were chromatographed in an atmosphere of nitrogen using a specially designed chromatobar. Trappe (1940) and Borgstrom (1954) have reported that aluminum oxide is rarely used for chromatography of lipids because it causes hydrolysis of ester linkages and isomerization of double bonds. Consequently, silica gel plates are still considered as the best choice for lipid chromatography.

#### 2.1.7 CONTACT ANGLE.

Hundreds of volatiles and non-volatiles are formed during frying or heating of shortenings and each of these compounds may increase or decrease the shortening free energy (surface tension) hence its contact angle measurement (Fig. 2 in Chapter III).

Many studies have now been published on the nature of dimeric and high molecular weight materials that are formed by thermal decomposition of oxidized lipids (Evans et al., 1965; Frankel et al., 1960; Perkins and Wantland, 1973; Wheeler and White, 1967). This subject has also been reviewed by Artman (1969) and Perkins (1967).

More recently, Combe et al. (1981) could separate three polymeric acids from heated soybean oil using quantitative thin layer chromatography. Wessels (1983)

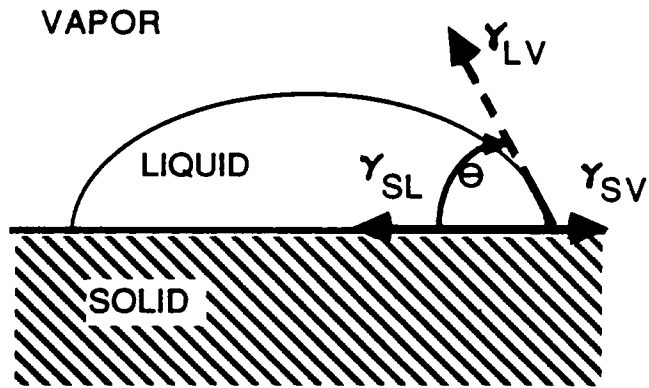


Fig. 2. Scheme of sessile drop of fresh frying oil and its contact angle with a solid surface.

reported that thermal dimeric triglycerides, polymers and cyclic acids can be formed by thermal degradation of fats and oils. However, oxidized triglycerides containing hydroperoxide-, epoxy-, hydroxy-, and keto- groups and also dimeric fatty acids or dimeric triglycerides linked by one or more oxygen atoms may be produced by oxidative degradation. Moreover, several saturated and monounsaturated volatile degradation products (aldehydes; ketones; hydroxy-, aldehydic-, keto-, and dicarboxylic-acids; hydrocarbons; alcohols; and aromatic compounds) were produced in the shortenings. Diglycerides and free fatty acids were produced from hydrolytic degradation processes of triglycerides.

Good (1979), reported that misleading conclusions can result from contact angle measurements of liquid mixtures since adsorption isotherms are never linear over wide ranges of mixture compositions.

Recently, White and Wang (1986) used high performance size exclusion chromatography to separate polymers within a 700 to 6000 Dalton range from heated soybean oils. These materials are extremely difficult to characterize because of their complex composition. No literature was available on the application of contact angle technique in food science for evaluating the effects of heating or frying time upon shortening quality. Polymer formations in used shortening may result in lowering surface free energy and

increasing contact angle measurements ( $\theta$ ). However, the polar fraction in the heated shortenings may result in increasing the surface free energy and decreasing contact angle on a particular surface. Due to antagonistic effects, the qualitative and quantitative domination of polar components or polymers may govern the interaction of the shortening with the surface, hence decreasing or increasing the contact angle.

The selection of an appropriate surface for the contact angle measurements requires two major accomplishments:

1. Good (1979) reported that "contact angle" can only be employed with regard to systems in which the solid phase is effectively flat.

2. In order for a particular surface to show significant differences between unheated and heated frying shortening, its surface free energy should be essentially equal to that of unheated frying shortening (Wightman, 1988).

#### 2.1.7.1 EFFECT OF MOLDING CONDITIONS ON FT-120-WW SURFACE.

The FT-120-WW mix is molded into a step pellet form at 300°C (Fig. 14 in chapter IV) and slowly cooled in ambient air. During cooling (Bortolini, 1988), unfavorable modification(s) such as oxidation or crystallization of the

FT-120-WW surface may occur, which, may result in less surface selectivity and increased variation in the contact angle measurements. Hence, annealing of this surface under controlled conditions is a necessary process in order to increase surface homogeneity and improve selectivity.

2.1.7.2 A SURFACE STUDY BY ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS (ESCA) AND SCANNING ELECTRON MICROSCOPY (SEM) OF THE POLYPROPYLENE MIXED WITH MONO-DISTEARIN (1.25%) AND CA STEARATE (30 ppm): THE EFFECTS OF VARIOUS SURFACE TREATMENTS:

X-ray photoelectron spectroscopy or Electron Spectroscopy for Chemical Analysis (XPS or ESCA) is considered the most powerful technique for the investigation of structure, bonding, and reactivity of polymeric systems (Dilks, 1981). Its many applications have ranged from the structural elucidation of simple homopolymers (Clark et al., 1973; Clark and Thomas, 1976; Clark and Thomas, 1978) to the determination of copolymer composition and morphology (Thomas and O'Malley, 1979). This procedure also can be used with more complex areas of highly crosslinked materials (Clark and Shuttleworth, 1979) and mechanistic studies of surface modification processes occurring on a monolayer depth scale 50A (Clark and Dilks, 1979).



ESCA experimentation involves the measurement of binding energies of electrons photoejected by interaction of a molecule with a monoenergetic nondestructive beam of soft X-rays (0.1 millirads  $S^{-1}$ ). The sample usually possesses an overall positive charge which is a valuable extra source of information (Dilks, 1981). The information provided by ESCA such as bonding energy is relatively easy to interpret and is related directly to the molecular configuration.

The possibility of quantifying absolute and relative binding energies for polymers has been demonstrated on a variety of surfaces (Clark et al., 1973; Clark and Thomas, 1976; Clark and Thomas, 1978).

#### 2.1.8 HIGH TEMPERATURE GAS CHROMATOGRAPHIC ANALYSIS OF TRIGLYCERIDES.

Frying oils and liquid shortenings are complex mixtures of triglycerides (TGs) (Wada and Koizumi, 1957; Petersson et al. 1981; El-Hamdy and Perkins, 1981). These triglycerides contain from 70-95% of unsaturated fatty acids (Langstraat, 1976; Industry News, 1980). Polyunsaturated fatty acids are much more prone to degradation during heating than the saturated fatty acids. However, Thompson et al. (1967) indicated that shortening

deterioration was independent of its degree of saturation but was dependent upon usage and condition. The physical and chemical nature of any frying media therefore can be determined from the quality and quantity of their triglycerides. The main requirements for any frying media are stability and good resultant sensory attributes to the fried food. During frying, the thermal and oxidative decomposition of shortenings produce volatiles (Chang et al., 1978) and non-volatiles (Combe et al., 1981), which have adverse effects on consumer acceptability and experimental animals (Alexander, 1981). Raughveer and Hammond (1967) suggested that the triglyceride structure might affect relative rates of oxidation of acyl groups in a triacylglycerol and that the acyl groups in the sn-1 and 3 positions should oxidize faster than those in sn-2. Yoshida and Alexander (1984) investigated the effects of heating on changes in triglycerides (TGs) in soybean oil and concluded the following:

1. Longer heating periods resulted in a reduction of absolute amounts of the (TGs). However, residual triacylglycerols were still the major component of the heated oil.

2. After 100 hr of heating, TGs with one to four double bonds increased from 42 to 71%, while those containing five to seven double bonds decreased from 58 to 29%.

3. Triglycerides with unsaturated fatty acids located in the second position contributed significantly less to thermal oxidation decomposition. A similar conclusion was reported by Wada and Koizumi (1983). However, they further reported that the carbon chain length of saturated fatty acids have essentially no influence on the oxidation rates of unsaturated fatty acids esterified in the same glycerol.

Analysis of mixtures of triglycerides in frying oil are time consuming and very difficult to accomplish. Kimmey and Perkins (1984) applied High Performance Liquid Chromatography (HPLC) equipped with acetadecyl bonded ( $5\mu$ ) spherical silica to separate triglycerides from confectionary fat (as cocoa butter), fractionated vegetable oil (palm and others) and from fractionated hydrogenated vegetable oil (soy and cottonseed). The premium quality replacement shortenings and cocoa butter had very similar (TG) compositions. The composition of the triglycerides was determined by gas chromatography of the corresponding fatty acid methyl esters.

Yoshida and Alexander (1984) used column chromatography and Thin Layer Chromatography (TLC) to separate triglycerides, which were identified by argentation (TLC), (separate triglycerides by the number of double bonds), and lipase hydrolysis. The resulting triacylglycerols were converted into fatty acid methyl esters and analyzed by gas liquid chromatography (GLC).

This technique required six steps to identify the general structure of different triglyceride classes. Also, the stereochemistry of each triglyceride remained undefined.

Takano and Kondoh (1987) used argentation nonaqueous reverse phase (NARP) high performance liquid chromatography HPLC equipped with an infrared detector for peak trapping. Each triglyceride fraction was analyzed with NARP chromatography using the glyceride-selective post-column reactor detector. However, complete separation of all triglyceride species could not be accomplished even with a gradient elution. Therefore, no satisfactory results could be obtained from the previous methods, because they are either complex, time consuming and/or could not provide complete separation of triglyceride classes.

Geeraert and Sandra, (1984; 1985) described capillary columns which contain a polar liquid phase (a methylphenylsilicone polymer) and possess high temperature stability comparable to that of the methyl silicone polymers. Kuksis and Myher (1986) reported that this procedure is well suited for a qualitative and quantitative assessment of total lipid extracts.

Lee and Dawson (1973) have studied changes in corn oil and chicken lipids as a result of frying. Linoleic acid decreased from 59% in fresh corn oil to 50% in oil heated for 48 hr. Losses of arachidonic, eicosatrienoic, oleic, stearic, and palmitoleic acids and a large increase in

linoleic acids were found in total muscle lipids after chicken was cooked in fresh corn oil. Also, Heath et al. (1970) found evidence indicating that an exchange of fat from the product to the cooking medium and from the oil to the batter coated chicken parts could occur to the extent of changing the fatty acid composition of the products, and the cooking oils determined the fatty acid profile in the batter samples

#### 2.1.9 SENSORY ANALYSES.

A great variety of volatile compounds are formed during autoxidation of unsaturated fatty acids in frying shortenings. Because of their odor and taste properties, they decisively influence the flavor of fried foods (Frankel, 1982; 1985). Also, Jackson (1981) reported that sensory evaluation will always be the final judgment on flavor and odor, however, there is also a need for more objective methodology to support decisions from sensory analyses and, at the same time, supply information specific enough to understand and offer a solution to flavor and odor problems.

CHAPTER III  
EXPERIMENTAL

3.1 EFFECT OF STATIC HEATING AND COMMERCIAL FRYING  
CONDITIONS ON SHORTENING QUALITY.

ESTABLISHING STANDARD OF REFERENCES.

One of the factors which contributes significantly to used frying oil stability is the quality of the fresh oil at the point of purchase. In general, shortening is considered fresh if it has a light color, bland flavor and odor, and its free fatty acid content does not exceed 0.08%.

The accumulation of oxidation products in shortenings heated over extended period of time has been reported to have adverse effects on consumer health. Moreover, the development of undesirable odor and flavor in frying shortening, and fried food, may result in consumer unacceptability of the food product causing a reduction in total sales volume which can impact firm profitability. Therefore, it is necessary to establish standards of reference, to be used for the determination of cut-off levels in frying shortenings. A used shortening is considered deteriorated if:

1. A sensory evaluation of its odor and flavor indicated

- the sample as not acceptable; or
2. The concentration of polar component% in used shortening is equal to or greater than 27%.

#### EXPERIMENTAL DESIGN:

The stability of the frying shortenings were evaluated in duplicate under static heating and commercial frying conditions. Two product forms (chicken nuggets and filets) were tested under the commercial frying conditions while no product added under static heating conditions. Five oils were tested under static heating conditions and three of the five were tested under commercial frying conditions. From each replicate, one sample was collected and tested in duplicate using the following procedures: polar component (open column chromatography), FoodOil-Sensor, free fatty acid, viscosity, triglycerides and sensory evaluation. Each oil sample was tested in duplicate on two pellets to measure contact angles, and in triplicates on two QHPTLC plates to evaluate polar component percent. Volatile profile analysis of the statically heated samples was conducted without sample duplication. Statistical procedures were reported in sections 3.1.1.11 for the static heating conditions and 3.1.2.3 for the commercial frying conditions.

### 3.1.1 EFFECT OF STATIC HEATING TIME ON SHORTENING QUALITY.

#### SHORTENINGS:

Five types of refined, bleached, winterized and deodorized shortenings were used in this study: (RBWD) corn oil; (RBD) peanut oil; (RBWD) cottonseed oil; (RBWD) cottonseed oil liquid shortening; and (RBWD) soybean oil liquid shortening (both liquid shortenings contained 6-10% fat crystals) (LOU ANA Foods, Inc, Opelousas, LA). All five shortenings contained the same amount of tertiary butylhydroquinone (TBHQ), propyl gallate, citric acid, and dimethylpolysiloxane to lengthen the oxidation induction period.

#### FRYERS:

The ten 5-quart deep fat fryers used in the study were purchased from K. Mart (Robeson brand) in Blacksburg, VA.

#### STATIC HEATING:

Four pounds of randomly selected shortening was placed in each of two fryers and heated at 360°F for 8 hr/day. A pair of fryers (two replicates) was added to the experiment at three day intervals, so that at the end of the heat treatment studies, samples with six heating periods (0, 2, 5, 8, 11 and 14 days) were obtained for each oil or liquid shortening used. Approximately, 200 ml of both the heated and fresh oil were withdrawn in duplicate, from each



replicate, for analysis. The samples were stored under nitrogen in 200 ml glass bottles in a freezer at 0°C until analyzed. The remaining oil was subjected to sensory analysis evaluations, 3.1.1.9 and 3.1.1.10. The four remaining oils were heated in a similar fashion in random order. The volume of the heated oils for each treatment was maintained constant throughout the experiment.

#### ANALYTICAL PROCEDURES:

Ten analyses were conducted to evaluate quality changes in frying shortenings under static and commercial frying conditions. Six of these methods were conventional and included free fatty acid, polar component%, gas chromatographic volatile profile analysis, viscosity, flavor evaluation of chicken nuggets prepared in each frying shortening, and odor evaluations of the tested shortenings. Three rapid tests were evaluated for their effectiveness in evaluating frying oil quality and included quantitative high performance thin layer chromatography, contact angle measurement, and gas chromatographic analysis of triglycerides. This latter test will be used to investigate the effects of heating periods and frying conditions, such as the type of shortening and cooked product, on quality changes. A modified procedure for the FoodOil-Sensor was developed to obtain absolute values of dielectric constant changes.

### 3.1.1.1 FREE FATTY ACID (% OLEIC ACID).

Percentages of free fatty acids (FFA-%oleic) were determined by titration with sodium hydroxide using the AOCS official method Ca 5a40 (1977).

### 3.1.1.2 POLAR COMPONENT TEST (OPEN COLUMN CHROMATOGRAPHY).

The chromatographic method of Paquot (1982) was used. This test involves elution of the non-polar portion of fresh or heat abused oils or fats from open chromatographic columns. The polarity% is calculated from the difference in weight between the sample and the eluted non-polar fraction.

### 3.1.1.3 GAS CHROMATOGRAPHY - VOLATILE PROFILE ANALYSIS (GC-VPA).

#### 3.1.1.3.1 GAS CHROMATOGRAPHIC CONDITIONS.

A Hewlett Packard (HP) gas chromatograph (5890A series) equipped with a flame ionization detector was used to identify volatile flavor profiles according to the method of Dupuy et al. (1985). Flow rates for nitrogen were 1.2 ml/min through the column and 30 ml/min for the

auxiliary make-up gas. Hydrogen and air flow rates were 30 and 240 ml/min, respectively. A HP ultra performance capillary column (Altra2) was used (50 m x 0.31 mm ID column coated with 0.52 micron film of crosslinked 5% phenyl methyl silicone). An external closed inlet device (Scientific Instrument Service) designed from the inlet system previously described (Legendre et al., 1979) was interfaced at the carrier gas arm of the insert Weldment assembly of the GC to facilitate direct gas chromatography. The inlet temperature of this device and the six-port rotary valve were set at 180°C and 200°C, respectively. The detector was set at 275°C. The column oven was lowered to -30°C with dry ice during an initial hold period of three min, and the six-port valve was positioned in the "inject" mode to direct the carrier gas through the sample and purge the volatiles onto the head of the cold column. The six-port valve was then positioned in the "run" mode to divert the carrier gas directly into the column. The oven temperature was programmed immediately from -30° to 30°C at 10 C/min, from 30° to 150°C at 2.5 C/min and from 150° to 250°C at 5 C/min. Final hold was 250°C for 10 min.

A HP Lab Automation System 3356 was used for data acquisition and analysis. A HP 7221T plotter was used to plot the GC curves.

#### 3.1.1.3.2 SAMPLE PREPARATION.

An 85 mm length of 9 mm OD borosilicate glass tubing was packed with volatile-free glass wool (conditioned for 16 hr at 200°C) loose enough to permit diffusion of oil, yet tight enough to prevent oil seepage from the liner onto the GC column. A clearance of about six mm was allowed at the bottom of the liner and 20 mm at the top. About 300 mg of the oil sample was added on top of the glass wool plug, and the liner with sample was secured in the heated inlet device for three min.

#### 3.1.1.3.3 CAPILLARY GC/MS.

A Finnegan-Mat Model 4000 Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS) was used in the identification of the volatile components of the oils. A SE-54 capillary column having the same physical dimensions and characteristics as the column described above was used for the GC/MS analysis.

Only specific flavor compounds (markers) which were produced in significant concentration (peak area) and total volatile peak areas were used to evaluate frying oil quality. These marker compounds are pentane, pentanal, hexanal, t-2-heptenal, t,c-2,4-decadienal and t,t-2,4-decadienal.

The objective of this test is to establish a high correlation between total volatiles or any of the marker volatiles with heating time and other tests particularly

the sensory evaluation in order to be able to predict oil abuse accurately.

#### 3.1.1.4 DIELECTRIC CONSTANT.

A FoodOil-Sensor (FOS) model NI-21A (Northern instruments Corp., Lino Lakes, MN.) was used to measure the dielectric constant of the frying oils. The operational procedure provided with the instrument was followed with some modifications. The purpose of the modifications was to obtain absolute FOS readings in addition to relative changes in the dielectric constant of the shortenings. The procedure utilized was as follows:

1. The FoodOil-Sensor reading (dielectric constant) was adjusted to zero using the 0.00 test oil-provided with the instrument, rather than fresh shortening. The 0.00 test oil is a synthetic and consists of isopropylmyristate and maintains excellent stability after four years of storage at room temperature. This modification provides relative changes in the dielectric constant as well as absolute values which are more representative of the percent polarity in the shortening than conventional methods. It has been stated (Stevenson et al., 1984) that the quality of the fresh shortening at time of purchase contribute

significantly to the deterioration rate of fat during frying.

2. The FOS readings were taken 30 seconds after the test light turned on to allow sufficient time to dissolve any fat crystals present in the liquid shortening.

3. A fixed volume (0.4ml) of the shortening was placed in the sensor cup (oil would cover the inner rim), using a disposable pipette, in order to avoid variations in FOS readings.

#### 3.1.1.5 VISCOSITY MEASUREMENT.

A Cannon-Fenske calibrated viscometers (Fisher capillary # 300 and # 400) were used for oil and liquid shortening viscosity measurements, respectively (Fisher, 1989). The efflux time was conducted at a constant temperature of 40°C. The densities of the oils were determined with a pycnometer and the viscosity in centipoise was obtained by multiplying the efflux time in seconds by the viscometer constant and oil density in grams per ml.

#### 3.1.1.6 POLAR COMPONENT% AS DETERMINED BY QUANTITATIVE HIGH PERFORMANCE THIN LAYER CHROMATOGRAPHY

(QHPTLC).

The QHPTLC method was developed to replace the polar component test because it is faster, consumes less reagents, however, it can be used by trained personnel only.

#### 3.1.1.6.1 SEPARATION OF POLAR AND NON-POLAR COMPOUNDS.

Heated oil samples were separated into polar and non-polar fractions. The polarity% obtained by (QHPTLC) was expected to represent the polarity% obtained by the open column chromatographic method of Paquot (1982). Appropriate solvent systems were tested in order to identify a mixture that provided optimum separation.

In order to reduce variability, moisture in the adsorbent, chamber saturation, temperature, chamber solvent quantity, sample volume, sprayed sample width and spot position on the plate were kept constant throughout the study.

#### 3.1.1.6.2 SOLVENT SYSTEM OPTIMIZATION.

Nine types of solvents were tested with and without acetic acid in fifty one combinations in order to examine their effectiveness in separating the heated oil into two fractions, polar and non-polar, in ratios identical to that

obtained with the polar component test (open column chromatography).

These solvents are Fisher Scientific brands and they are: Acetone, HPLC grade (Cat. A949-1<sup>\*</sup>), Acetonitrile, HPLC grade (Cat. A998-1<sup>\*</sup>), Benzene, 99 Mol% pure (Cat. B414-1), Chloroform, HPLC grade (Cat. C606-1), Diethyl ether, HPLC grade (E198-4), Ethyl Alcohol (Cat. A407-1<sup>\*</sup>), Methanol, HPLC grade (Cat. 452-1<sup>\*</sup>), Petroleum Ether, Optima (Cat. 120-4), Xylenes (Cat. X5-4<sup>\*</sup>) and Acetic Acid, Glacial (Cat. A38-500<sup>\*</sup>).

The solvent combinations are shown in Tables 2 and 3.



Table 2. Solvent systems evaluated for the QHPTLC analysis.

| Solvent type  | solvent ratios |     |    |    |    |    |    |    |
|---------------|----------------|-----|----|----|----|----|----|----|
|               | 1              | 2   | 3  | 4  | 5  | 6  | 7  | 8  |
| Pet. Ether    | 50             | 50  | 50 | 50 | 70 | 70 | 90 | 90 |
| Methanol      | --             | 50  | -- | 50 | -- | 30 | -- | 10 |
| Diethyl Ether | 50             | --  | 50 | -- | 30 | -- | 10 | -- |
| Acetic Acid   | 0.2            | 0.2 | 4  | 4  | 2  | 2  | 2  | 2  |

| Solvent type  | solvent ratios |    |    |    |    |    |    |    |
|---------------|----------------|----|----|----|----|----|----|----|
|               | 9              | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
| Pet. Ether    | 95             | 95 | 95 | 95 | 99 | 99 | 99 | 99 |
| Methanol      | --             | -- | 5  | 5  | -- | -- | 1  | 1  |
| Diethyl Ether | 5              | 5  | -- | -- | 1  | 1  | -- | -- |
| Acetic Acid   | 2              | -- | 2  | -- | 2  | -- | 2  | -- |

| Solvent type  | solvent ratios |     |     |     |     |     |
|---------------|----------------|-----|-----|-----|-----|-----|
|               | 17             | 18  | 19  | 20  | 21  | 22  |
| Pet. Ether    | --             | --  | --  | --  | 100 | 100 |
| Methanol      | --             | --  | 100 | 100 | --  | --  |
| Diethyl Ether | 100            | 100 | --  | --  | --  | --  |
| Acetic Acid   | 2              | --  | 2   | --  | 2   | --  |

Table 3. Solvent systems evaluated for QHPTLC analysis.

| Solvent type  | solvent ratios |     |     |     |     |     |     |     |     |
|---------------|----------------|-----|-----|-----|-----|-----|-----|-----|-----|
|               | 1              | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9   |
| Acetone       | 100            | 100 | --  | --  | --  | --  | --  | --  | --  |
| Chloroform    | --             | --  | 100 | 100 | --  | --  | --  | --  | --  |
| Benzene       | --             | --  | --  | --  | 100 | 100 | --  | --  | --  |
| Xylenes       | --             | --  | --  | --  | --  | --  | 100 | 100 | --  |
| Acetonitrile  | --             | --  | --  | --  | --  | --  | --  | --  | 100 |
| Ethanol       | --             | --  | --  | --  | --  | --  | --  | --  | --  |
| Diethyl Ether | --             | --  | --  | --  | --  | --  | --  | --  | --  |
| Pet. Ether    | --             | --  | --  | --  | --  | --  | --  | --  | --  |
| Acetic Acid   | 2              | --  | 2   | --  | 2   | --  | 2   | --  | 2   |

| Solvent type  | solvent ratios |     |     |    |    |    |    |    |    |
|---------------|----------------|-----|-----|----|----|----|----|----|----|
|               | 10             | 11  | 12  | 13 | 14 | 15 | 16 | 17 | 18 |
| Acetone       | --             | --  | --  | -- | -- | -- | -- | -- | -- |
| Chloroform    | --             | --  | --  | 95 | 95 | 99 | 99 | -- | -- |
| Benzene       | --             | --  | --  | -- | -- | -- | -- | 50 | 50 |
| Xylenes       | --             | --  | --  | -- | -- | -- | -- | -- | -- |
| Acetonitrile  | 100            | --  | --  | -- | -- | -- | -- | -- | -- |
| Ethanol       | --             | 100 | 100 | -- | -- | -- | -- | -- | -- |
| Diethyl Ether | --             | --  | --  | 5  | 5  | 1  | 1  | 50 | 50 |
| Pet. Ether    | --             | --  | --  | -- | -- | -- | -- | -- | -- |
| Acetic Acid   | --             | 2   | --  | 2  | -- | 2  | -- | 2  | -- |

Table 3. cont.

| Solvent type  | solvent ratios |    |    |    |    |    |    |    |    |
|---------------|----------------|----|----|----|----|----|----|----|----|
|               | 19             | 20 | 21 | 22 | 23 | 24 | 25 | 26 | 27 |
| Acetone       | --             | -- | -- | -- | -- | -- | -- | -- | -- |
| Chloroform    | --             | -- | -- | -- | -- | -- | -- | -- | -- |
| Benzene       | 50             | 50 | -- | -- | -- | -- | -- | -- | -- |
| Xylenes       | --             | -- | 50 | 50 | 50 | 50 | 60 | 40 | 80 |
| Acetonitrile  | --             | -- | -- | -- | -- | -- | -- | -- | -- |
| Ethanol       | --             | -- | -- | -- | -- | -- | -- | -- | -- |
| Diethyl Ether | --             | -- | 50 | 50 | -- | -- | -- | -- | -- |
| Pet. Ether    | 50             | 50 | -- | -- | 50 | 50 | 40 | 60 | 20 |
| Acetic Acid   | 2              | -- | 2  | -- | 2  | -- | -- | -- | -- |

| Solvent type  | solvent ratios |    |
|---------------|----------------|----|
|               | 28             | 29 |
| Acetone       | --             | -- |
| Chloroform    | --             | -- |
| Benzene       | --             | -- |
| Xylenes       | 90             | 85 |
| Acetonitrile  | --             | -- |
| Ethanol       | --             | -- |
| Diethyl Ether | --             | -- |
| Pet. Ether    | 10             | 15 |
| Acetic Acid   | --             | -- |

### 3.1.1.6.3 LINOMAT III CALIBRATION.

The jet part of the Linomat head and the syringe were cleaned with a sonifier in a heated methanol bath for one hour before use. The jet assembly was also calibrated to the correct height and two gaskets were placed before the jet head to avoid any gas leak. The nitrogen flow rate was adjusted at 28 psi and the Linomat gas valve was maintained at +1.4 turns. The other knobs were adjusted at 6 sec/ $\mu$ l and 30 mm/ $\mu$ l. Accordingly, the sample could be sprayed in a fixed fine line, approximately 0.8 mm in width, to maintain best reproducibility.

### 3.1.1.6.4 HPTLC PLATES PREPARATION.

HPTLC precoated silica gel plates (MERCK 60, 20 x 10 cm) were marked to half height with a pencil and washed with methanol (HPLC grade, Fisher, Cat. No. A 452-4) passing 50% of the plate height (to the half height mark) in a closed chamber. The methanol was then volatilized under the hood with a hair dryer (1200 Watts) followed by activation of the plates in a drying oven at 105°C for one hour. The plates were immediately wrapped in aluminum foil and stored in a desiccator until used.

### 3.1.1.6.5 SAMPLE APPLICATION.

The heated oil samples were dissolved in 4% chloroform (Fisher, Cat. No. C 298-1) and sprayed on the HPTLC plates

in 2, 3, 4 and 5  $\mu$ l volumes in 10 mm bands, 5 mm apart using the sample applicator, Linomat III, CAMAG Scientific Inc., Wrightsville Beach, NC). The 3  $\mu$ l volume was selected since it provided optimum separation with minimum variation. Each sample was spread in a fine band 0.8 mm, under  $N_2$  pressure at 26 psi, in triplicate on two plates. The plates were developed to their half heights immediately after being sprayed in a closed chamber containing 3 ml of an 85:15 xylene: petroleum ether mixture. The solvent was removed by the hair dryer to avoid any band broadening.

#### 3.1.1.6.6 BAND VISUALIZATION.

The separated bands were visualized using four different methods:

1. Dipping the developed plates in 0.1% 2',7'-dichlorofluorescein (Eastman Kodak Co. Rochester NY, Cat. 136 9362) in 95% ethanol.
2. Dipping the plates in 6-P-toluidino-2-naphthalene-sulf-onic acid (TNS) (Jones et al., 1982). Methods one and two are known as fluorescence quenching methods.
3. Exposing the developed plates to iodine (Fisher, Cat. I 37) vapor in a closed chamber.
4. Spraying the plates with 10% phosphomolibdic acid (Sigma, St. Louis, MO. Cat P-1518) in 95% ethanol with subsequent heating in a drying oven at 105°C for 10 min.

This procedure is known as charring (Touchstone et al., 1980).

The first method was selected because it was faster, provided sharper and more defined lipid bands, and provided improved detection of the separated bands at 365 nm. Also, 2', 7'-dichlorofluorescein is a stable reagent which can be used in a rapid, sensitive and nondestructive procedure for evaluating lipid quality and quantity on HPTLC. Immediately after the plates were dipped in the visualization solution, the ethanol was evaporated with a hair dryer, and the plates were rewrapped with the aluminum foil and stored in a desiccator until scanned.

#### 3.1.1.6.7 SCANNER II (DENSITOMETER) CALIBRATION.

A CAMAG Scanner II was used with the following adjustments: Hg lamp 365 nm, Fluorescence, a 0.2 mm slit width adjusted to 3 and macro, a 4.0 mm slit length adjusted to 3 and macro, and a scan speed of 1.0 mm/sec.

#### 3.1.1.6.8 INTEGRATOR CALIBRATION.

CAMAG integrator parameters were used and adjusted as follows: Peak width=2, Peak threshold=12, Chart speed=4 in/min. and Attenuation=64.

#### 3.1.1.6.9 SCANNING QUANTITATION.

Under UV light at 365 nm, two fluorescing bands could be observed. The first band (polar fraction) from the plate's bottom was narrow and sharp with an  $R_f$  value of approximately zero. The upper band (nonpolar fraction) was wide with an  $R_f$  value of 0.20 (Fig. 14). The separated bands were scanned at 365 nm using a CAMAG, Scanner II, and two sharp peaks were plotted and integrated polar component% was determined.

### 3.1.1.7 CONTACT ANGLE.

The objective of this experiment is to find a surface with high selectivity (capable of distinguishing between fresh and previously used frying shortening based on differences in their free energy) which will favor differences in their contact angles (Fig. 2).

#### 3.1.1.7.1 SURFACE SELECTION.

In a preliminary investigation, solids of varying ranges of surface free energy (SFE) were tested for their contact angle selectivity with unheated and abused frying oils. The solids tested included: microslide cover glass (Fisher Scientific Cat.# 12-541A); GOLD SEAL BEVELED microslides (Fisher Scientific Cat.# 12-518-110B); Corning microslides (Fisher Scientific Cat.# 12-518-101), heated at 525°F in a glass annealing oven in order to remove the

surface coating; Fisher finest microslides (Fisher Scientific Cat.# 12-544-4); Fisher precleaned microslides (Fisher Scientific Cat.# 12-550A); stainless steel (kitchen spatula); Teflon (Dupont), epoxy slide (Chemistry Department, Virginia Tech, Blacksburg, VA), Parafilm (American Can Company, Greenwich, CT), Mylar polyester film composed of polyethylene terephthalate (DuPont, Wilmington, DE), and four polymers obtained from the research laboratory of Aristech Chemical Corporation (Monroeville, PA). These latter polymers were produced from TI-4040-G, which is an impact copolymer of polypropylene with propylene and ethylene side chains; D-028-S, a homopolymer of polypropylene mixed with a specific acid scavenger; F-120-F, a homopolymer of polypropylene without mono and distearate (anti-static agent); FT-120-W; a homopolymer of polypropylene with 0.75% anti-static agent, and 30 ppm of calcium stearate (HCl scavenger); and finally FT-120-WW which is similar in composition to FT-120-W but contains 1.25% anti-static agent. These polymers were molded in a press at 300°C to form the step pellet (10 x 12.5 cm, (width x length) and 0.3 and 0.15 cm, (thickness). The pellets were allowed to cool at room temperature and stored until used.

The surfaces, with the exception of the polypropylene, were cleaned with Tide (TM) and distilled water, followed by thorough rinsing in distilled water, drying in a vacuum



oven at room temperature and storing in a desiccator (Dann, 1970). Cleaning the FT-120-WW with Tide (TM) and water or with pure methanol eliminates surface selectivity and substantially increases surface free energy. The transfer of organic materials from fingers and hands to these solids was avoided during the handling process since it could affect the contact angle of any solid (Newmann and Good, 1979).

The FT-120-WW pellets manifested a higher oil selectivity (more differences between fresh and heated oil) than any other tested surface and was chosen as the proposed surface.

The treatment was performed as follows: a 0.35 cm diameter hole was drilled in the thicker corner of each of ten pellets which were then hung in a vacuum oven by a thick wire (0.3 cm) inserted through each hole. Two heat sink pellets were placed on the sides to reduce heat radiation from the oven walls. The pellets were annealed by heating in vacuum (-100 kpa) at 140°C for 2 hours and overnight cooling in the oven. The annealing process was considered complete when the temperature inside the oven reached room temperature (four and half hours after the heat was turned off). The pellets were then removed from the oven and tested, in duplicate, after 0, 1, 2, 3, 4, 5, 6 and 7 days of the treatment in order to study the effect of time on the selectivity and behavior of the surface

after annealing. Ten microliters of unheated and 14 day heated corn oil were gently placed on each surface with an Eppendorf micropipet using Fisher virgin polypropylene (Cat 21-381-10A) reference tips. Spreading of the sessile drops with time on FT-120-WW (Fig. 3) was observed visually without magnification.

Contact angles were measured by a horizontal microscope with a protractor eyepiece equipped with cross hairs (Gaertner Scientific Company, Chicago, IL). Contact angle measurements on FT-120-WW surface after 4 days of annealing were taken from both sides of each oil droplet at 1, 5 and 10 min intervals after the oil droplets were placed on the surface. However, the 5 minute period was chosen for providing better selectivity. All measurements were made at  $28.5^{\circ}\text{C} \pm 0.5$  using the "sessile drop" method.

In order to understand why surface selectivity changed with time after annealing, two surface analysis techniques were conducted, Electron Spectroscopy for Chemical Analysis (ESCA) and High Resolution Scanning Electron Microscopy (HSEM). Five samples of the FT-120-WW polymer were given five treatments as follows:

1. untreated control;
2. methanol wash;
3. six days storage after annealing;
4. four days storage after annealing; and
5. directly after annealing.

### 3.1.1.7.2 SURFACE STUDY BY ESCA AND HSEM OF THE FT-120-WW.

#### 3.1.1.7.2.1 ESCA OF THE FT-120-WW SURFACE.

After the pellets were treated, they were prepared for sampling as follow: each pellet was placed on a Kleenex tissue in an inverted position to maintain the surface cleanliness and a 0.6 mm deep hole was made with a 5/8" hole saw (The Real McCoy-True Value, Chicago, IL). The discs were removed with a paper cutter and fixed inside a small box by double sided scotch tape with the clean surface up. ESCA spectra were obtained on a Perkin Elmer 5300 electron spectrometer using Mg  $K_{\alpha}$  (1253.6 eV) radiation. Samples were mounted as discs (5/8" in diameter, 3.5 mm in thickness) onto the sample probe tip using a piece of double-sided tape.

#### 3.1.1.7.2.2 SEM OF THE FT-120-WW:

After the pellets were heat treated (annealed), samples were prepared by reducing the thickness of the pellet to 1 mm using a single point fly cutter, and a 3 x 9 mm rectangular piece was cut from each treated pellet using a knife with a flat sharp tip and a hammer.

Scanning electron microscopy photographs were obtained with a Phillips EM420 electron microscope using two magnifications, 50,000 and 12,500.

### 3.1.1.8 HIGH TEMPERATURE GAS CHROMATOGRAPHIC ANALYSIS OF TRIGLYCERIDES.

Frying oils and liquid shortenings are complex mixtures of triglycerides which determine the physical and chemical nature of the frying media.

Triglyceride alterations during the heating and frying processes were studied using a two step analytical procedure. The first step was the separation of the non-polar fraction from the open column in the polar component test (Paquot, 1982) followed (second step) by injection of the fraction into the triglyceride analysis phase (TAP) capillary column using a movable cold on-column injection device (Geeraert et al., 1984).

#### 3.1.1.8.1 OPERATION CONDITIONS:

|             |   |
|-------------|---|
| Technique   | :GC-Chrompack capillary column (Cat. 7483).   |
| Column      | :25 m x 0.25 mm WCOT fused silica coated with TAP (OV-17 type), 0.10 $\mu\text{m}$ (Cat 7483)   |
| Temperature | :340 $^{\circ}\text{C}$ (1 min) ==> 352 $^{\circ}\text{C}$ (1 $^{\circ}\text{C}/\text{min}$ )<br>340 $^{\circ}\text{C}$ (1 min) ==> 360 $^{\circ}\text{C}$ (1 $^{\circ}\text{C}/\text{min}$ ) for peanut oil only |
| Carrier gas | :H <sub>2</sub> , 15 psi, 50 cm/sec<br>13 psi, 50 cm/sec (for soybean oil)  |

liquid shorting)

Injector :J & W movable cold on-column (Cat.  
20010200)  
Injection :0.2  $\mu$ l of 0.05% non-polar fraction of  
shortening in hexane  
Detector :FID

The triglycerides were separated on the basis of carbon number and degree of unsaturation. Peak identification was obtained from identical chromatograms provided as application notes by CHROMPACK (RARITAN, NJ). The triglyceride profiles of heated oils were compared with those obtained from the unheated oils.

### 3.1.1.9 FLAVOR EVALUATION OF HEATED SHORTENINGS.

#### 3.1.1.9.1 EFFECT OF STATIC HEATING TIME ON CHICKEN NUGGETS FRIED FOR FLAVOR.

Chicken nuggets were cut from chicken breasts, lightly salted and fried, in each of the five shortenings for each one sensory session at the end of a 0, 2, 5, 8, 11 and 14 day heating period. Each shortening was statically heated in duplicate. Fifteen panelists were presented with 6 chicken nugget samples, under a red light, and instructed

to rate these against the control (chicken nuggets cooked in fresh oil) according to the following rating scale (Stone and Sidel, 1985): 10 bland; 9 and 8 slightly rancid; 7 and 6 moderately rancid; 5, 4 and 3 strongly rancid; and 2 and 1 extremely rancid.

#### 3.1.1.9.2 EFFECT OF SHORTENING TYPE AND HEATING TIME ON CHICKEN NUGGETS FRIED FOR FLAVOR.

The five types of oils used in section 3.1.1 were again used in this study. Each of the five oils were placed in a fryer and heated at 360°F for 5 days in duplicate. The procedure was repeated with heating for an 11 day period. At the end of each heating period, chicken nuggets were fried in the heated shortenings and prepared for sensory evaluation as reported in section 3.1.1.9.1. Differences between shortening flavor due to differences in shortening type were determined by a sensory analysis at both heating periods, 5 and 11 days.

#### 3.1.1.10 ODOR EVALUATION OF HEATED SHORTENING.

##### 3.1.1.10.1 EFFECT OF STATIC HEATING TIME ON SHORTENING ODOR.

Five milliliters of each shortening sample heated for 0, 2, 5, 8, 11 and 14 days was evaluated by a sensory panel for rancidity by evaluating the odor from a small beaker at room temperature. The samples were rated on the basis of rancid odor intensity as explained in section 3.1.1.9.

#### 3.1.1.10.2 EFFECT OF SHORTENING TYPE AND HEATING TIME ON ODOR.

The five types of oils used in section 3.1.1 were again used in this study. Each of the five oils were placed in a fryer and heated at 360°F for 5 days in duplicate. One sensory session was performed on each replicate. The procedure was repeated with heating for an 11 day period. At the end of each heating period, 5 ml of each of the oil samples was placed in 50 ml beaker under the red light and 15 panelists were asked to compare rancid odor intensity of heated oil samples with that of fresh oil by sniffing, as reported in section 3.1.1.9.1. Differences between shortening odor and due to differences in shortening type were determined by a sensory analysis at both heating periods, 5 and 11 days.

#### 3.1.1.11 STATISTICAL ANALYSES.

Under static heating conditions, heating time, shortening type and tools used for analysis were the only independent variables which affected changes in the dependent variables.

Two replicates and two or more samples were tested and the data were statistically analyzed using the SAS package as follows:

1. Intercepts: identify and compare the degree of freshness in unheated oils and liquid shortenings using Duncan's multiple range tests. This procedure was applied to all analyses except the triglycerides.

2. Slopes: define the rate of increase or decrease in subjective and objective data (decomposition) with time. Slopes were calculated from the general linear regression equation,  $Y = \text{intercepts} + \text{slopes } X$ . Significant differences between slopes were determined using the following equation:

$$t = \frac{\beta_1 - \beta_2}{\sqrt{(2\text{MSE} / \sum (\text{HT} - \text{average HT})^2)}}$$

$\beta$ : slope, HT: heating time in days and MSE: mean square error.

$t_{df, \alpha}$ , where df is the degrees of freedom of the error term and  $\alpha = 0.01$  (two sided). If  $t > t_{df, \alpha}$  indicates significance. This procedure was applied to all analyses except the triglycerides.



3. Effect of heating time, shortening type or tools used for analysis and interactions: The Analysis of Variance (ANOVA) was applied to all analyses.

4. Linearity and curvature: tested using orthogonal polynomial equations.

$$t = \sqrt{(\sum c_n y_n)^2 / (1/N(\sum (c_n)^2) \text{MSE})}$$

$t_{df, \alpha}$ , where df is the degrees of freedom of the error term;

$\alpha = 0.1$  (one sided) for linear regression, however,  $\alpha = 0.05$  (two sided) for quadratic regression.

$c_n = -5, -3, -1, +1, +3$  and  $+5$  for linear regression, however,  $c_n = +5, -1, -4, -4, -1$  and  $+5$  for quadratic regression.

$y$  = mean of one treatment, and  $N$  = number of observations from each treatment. If  $t > t_{df, \alpha}$  indicates significance. Significant linearity and insignificant curvature are preferred for predicting heat abuse of used shortening. Applied to all analyses but not to analysis of triglycerides.

5. Standard deviations: applied to all analyses.

6. Regression: Quadratic and linear regressions at the 95% confidence level were performed, however, only the linear regression was reported. Correlation coefficients between experimental values and heating time were applied to investigate the possibility of predicting heat abuse from the regression plots. Correlation coefficients among

all objective and subjective tests would provide information on the possible substitution of time consuming complex quality tests with simple rapid procedures. The analysis was applied to all analyses except triglycerides.

7. Orthogonal contrast: this test was used to compare the differences between shortening samples heated to different periods and tested for flavor or odor only.

### 3.1.2 EFFECT OF COMMERCIAL FRYING CONDITIONS ON SHORTENING QUALITY.

#### 3.1.2.1 EFFECT OF CHICKEN FILET (PRESSURIZED) AND NUGGET (NON-PRESSURIZED) FRYING CONDITIONS ON THE QUALITY OF THREE TYPES OF SHORTENINGS.

##### 3.1.2.1.1 FILET AND NUGGET PREPARATION.

Marinated chicken filets and nuggets were battered with a milk and egg mixture (2 gallons milk: 1 egg). After draining, a thin flour coating mix (seasoned coating, Chick-fil-A, Inc., Atlanta, GA) was added by dipping the chicken parts in the flour mixture. Chicken filets were fried under pressure at 325°F for 3.5 minutes, while chicken nuggets were fried in an open fryer at 325°F for 2.5 minutes.

### 3.1.2.1.2 SAMPLING OF SHORTENINGS.

Three types of shortenings were collected from a fast food restaurant. Cooking oil (peanut oil) currently used at the restaurant, containing TBHQ and dimethylpolysiloxane; soybean and cottonseed oil liquid shortenings containing TBHQ, dimethylpolysiloxane, citric acid and propyl galate (LouAna Foods, Inc.). Filtered shortening was cooled at room temperature over night, and shortening samples were collected in the morning after 2, 5 and 7 days of frying. The samples were placed in 1.3 pint polypropylene containers (Rubbermaid Inc. Wooster, OH) and transported to the Virginia Tech campus. On arrival, a blanket of nitrogen was placed over the samples which were stored at 0°C if the analyses were conducted within two weeks, and at -20°C when the tests were conducted within a longer time period.

### 3.1.2.1.3 FILTRATION AND CLEANING.

The shortenings were filtered at least once every 2-5 hr depending on the chicken volume cooked. The last filtration was performed at the end of each working day. Refilling the frying vessels with filtered shortening was done following the vessel cleaning. At the end of the last sampling, the fryers were emptied from the shortening and washed using the following procedure.

Commercial grade sodium hydroxide was used to remove the shortening residues and remaining food particles remaining after filtration. After an initial rinsing, commercial acetic acid was added to neutralize the sodium hydroxide and eliminate the catalytic metals. Clean water was used for rinsing and a paper towel was used to dry the frying vessels.

#### 3.1.2.1.4 INFORMATION PROVIDED BY THE RESTAURANT.

1. Weight (lb) per day of cooked chicken filets and nuggets.
2. Weight (lb) per day of each shortening added.
3. Operating conditions:

Frying conditions:

Fryer type : Henny Penny pressure fryer model  
500 (Henny Penny, Inc. Eaton,  
OH).

Chicken nugget: nonpressurized and frying time  
2.5 min/batch.

Chicken filet : pressurized and frying time  
3.5 min/batch.

Operating temperature: 325<sup>o</sup>F for both nuggets  
and filets.

Total heating time: 9-11.3 hr/day.

4. Consumer acceptability, worker and operator opinions about the performance of each of the three shortenings were considered.

#### 3.1.2.1.5 SHORTENING YIELD AND MOISTURE DETERMINATION.

The effect of shortening types on its yield during the cooking of chicken nuggets and chicken filets was studied. Shortening yield was estimated from the weight (lb) of raw chicken cooked divided by the weight (lb) of shortening used (absorbed) to cook the chicken products.

In order to study the effect of moisture removal from the chicken products on shortening yield, moisture percentages in chicken nuggets and chicken filets before and after frying were estimated in 2 gram of samples in quadruplicate, at 100-102<sup>o</sup>C for 16-18hr, in air oven, using the procedure of AOAC (1984).

#### 3.1.2.2 ANALYTICAL PROCEDURE.

The same analyses which were applied on the statically heated shortenings were repeated on the commercially used frying shortenings without modification. However, the Volatile Profile Analysis (VPA) was not conducted because of time limitations.

Furthermore, it was expected that food ingredients which were introduced from the chicken, batter and breading would have noticeable effects on the objective and subjective analytical data. Consequently, another objective was added to the gas chromatographic analysis of triglycerides which was the effect of lipids, introduced through the chicken or chicken coating, on triglyceride modification in the frying shortening. The results from this study were compared with the results obtained from the heating experiment in order to understand the effect of chicken fillet/nugget frying on the stability of the frying media.

### 3.1.2.3 STATISTICAL ANALYSES.

An evaluation of differences due to total frying time, chicken volume, time of use, shortening replenishment and shortening type were considered as an independent variable in order to investigate their significance toward the rate of deterioration, hence the final results.

Two replicates and two or more samples depending on the analysis, were tested and the data statistically analyzed using the SAS package as follows:

1. Effect of heating time (days), total heating time (hr), total frying time (hr), shortening type, chicken

weight, oil weight, and product form (chicken nuggets/filets) were tested using the General Linear Models (GLM) procedures.

2. Intercepts: The degree of freshness in unheated oils and liquid shortenings was identified and compared using Duncan's multiple range test was applied to all analyses, except the triglyceride.

3. Standard deviation: applied to all analyses.

4. Orthogonal contrast: this test was used to compare the differences between shortening samples evaluated for flavor and odor.

5. Regression: The Principle Component Regression (PCR) was used to develop predicting equations for oil quality. A copy of this program is presented in appendix B.

## CHAPTER IV

### RESULTS AND DISCUSSION

#### 4.1 EFFECT OF STATIC HEATING CONDITIONS ON SHORTENING QUALITY.

The stability of corn oil (C/O), cottonseed oil (CS/O), peanut oil (P/O), cottonseed (CS/OLS) and soybean (SB/OLS) oil liquid shortenings was investigated using ten chemical, physical and sensory analyses. All five shortenings were in good conditions at the time of purchase because they had light color, bland flavor and odor and their free fatty acid content were less than 0.08%.

##### 4.1.1 FREE FATTY ACID - % OLEIC ACID.

Unheated samples of the oils and liquid shortenings had initial FFA contents between 0.01 and 0.04% (Table 4). Generally, low free fatty acid content is considered a good indicator of shortening quality. A significant ( $P < 0.0001$ ) effect of static heating time on increases in FFA% in all five shortening samples was found. Also, significant ( $P < 0.0001$ ) differences were found among all shortening



types. The final FFA content in all samples was in the range of 0.148 to 0.335%. However, all these values were much below the suggested (Weiss, 1983) FFA cut-off point (2%). This situation indicated a low production of FFA from both hydrolysis and oxidation as a result of static heating since moisture and oxygen are limited when compared with simulated or commercial frying.

The data points which represent the increases in FFA with heating time indicated a significant ( $\alpha=0.1$ ) linearity with a slight ( $\alpha=0.05$ ) curvature for all five shortenings (Fig. 3). High positive correlation coefficients between FFA% and heating time were found: 1.00, 0.98, 0.97, 0.99 and 0.96, for C/O, CS/O, CS/OLS, P/O and SB/OLS, respectively. Moreover, good correlation coefficients among different shortenings and tests are reported in Tables 31-35.

The slope (rate of FFA accumulation) of SB/OLS was significantly lower (Fig. 3) than the slopes of all other shortenings ( $\alpha=0.01$ ), and no significant differences between the remaining slopes were found. Moreover, there was no significant ( $\alpha=0.05$ ) difference among the intercepts (initial FFA%). It was concluded from these results that the linear increase in FFA content in the frying shortenings could be used as an indicator of heat abuse under static heating conditions. While there were no significant differences in the deterioration rate between

PLOT OF FREE FATTY ACIDS% VS HEATING TIME

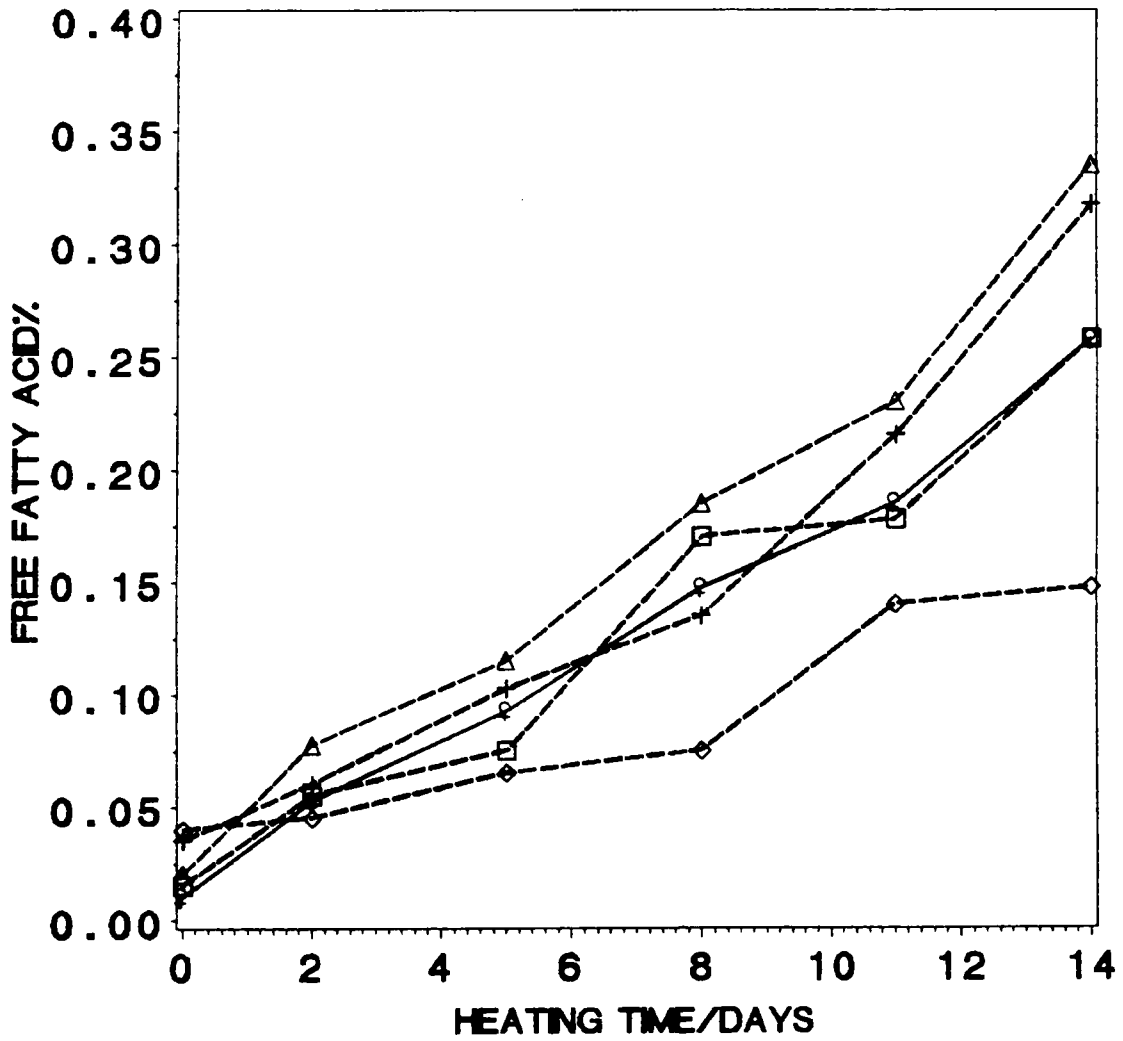


Fig. 3. Effect of static heating time (Days) on Fatty Acid-(%Oleate) of corn (♀), cottonseed (□), peanut oils (Δ), cottonseed (+) and soybean (◇) oil liquid shortenings.

Table 4. Effect of static heating time on free fatty acid (as %oleic acid) in corn, cottonseed, and peanut oils and cottonseed and soybean oil liquid shortening.<sup>1</sup>

| HEATING TIME<br>(DAYS) | FREE FATTY ACID (%OLEIC ACID) <sup>5</sup> |                   |                     |                  |                     |
|------------------------|--|-------------------|---------------------|------------------|---------------------|
|                        | SHORTENING TYPE <sup>3</sup>               |                   |                     |                  |                     |
|                        | C/O <sup>4</sup>                           | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0                      | 0.010<br>(0.000)                           | 0.015<br>(0.007)  | 0.035<br>(0.007)    | 0.020<br>(0.000) | 0.040<br>(0.000)    |
| 2                      | 0.053<br>(0.005)                           | 0.055<br>(0.017)  | 0.060<br>(0.000)    | 0.078<br>(0.015) | 0.045<br>(0.006)    |
| 5                      | 0.093<br>(0.005)                           | 0.075<br>(0.006)  | 0.103<br>(0.009)    | 0.115<br>(0.006) | 0.065<br>(0.006)    |
| 8                      | 0.148<br>(0.009)                           | 0.170<br>(0.023)  | 0.135<br>(0.006)    | 0.185<br>(0.006) | 0.075<br>(0.006)    |
| 11                     | 0.185<br>(0.006)                           | 0.178<br>(0.005)  | 0.215<br>(0.010)    | 0.230<br>(0.000) | 0.140<br>(0.023)    |
| 14                     | 0.258<br>(0.009)                           | 0.258<br>(0.009)  | 0.318<br>(0.013)    | 0.335<br>(0.017) | 0.148<br>(0.005)    |
| $r^2$                  | 1.00                                       | 0.98              | 0.97                | 0.99             | 0.96                |

<sup>1</sup>: Average values of two replicates and two samples.

<sup>2</sup>: Correlation coefficient of FFA with heating time.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.

<sup>5</sup>: Standard deviations are the values in brackets.

C/O, CS/O, CS/OLS and P/O, the thermal stability of soybean oil liquid shortening was significantly higher under the static heating conditions, as determined by the linear accumulation of free fatty acids. Nevertheless, it is uncertain if these shortenings will maintain identical stability under commercial frying conditions.

#### 4.1.2 POLAR COMPONENT% (OPEN COLUMN CHROMATOGRAPHIC METHOD).

A wide range of polarity was detected in the unheated shortening samples. The initial values were in the range of 4.03 to 9.33% (Table 5). Fresh shortening samples with higher initial polar component% also had higher final polar component% at the end of the static heating period. Although CS/OLS had a significantly ( $\alpha=0.05$ ) lower initial polar component% than CS/O, (7.06) vs (9.33%), the final polar component% of the CS/OLS was higher than the CS/O (41.8 vs 37.8 %), which indicated that the initial polar component% was not always indicative of the increased deterioration rate. However, this increase could be related to the triglyceride composition of the shortening used since triglycerides represent the major nonpolar portion of any shortening.

Table 5. Effect of static heating on polar component% in corn, cottonseed and peanut oils, and cottonseed and soybean oil liquid shortening.<sup>1</sup>

| HEATING TIME<br>(DAYS) | POLAR COMPONENT% <sup>5</sup> |                   |                     |                  |                     |
|------------------------|-------------------------------|-------------------|---------------------|------------------|---------------------|
|                        | SHORTENING TYPE <sup>3</sup>  |                   |                     |                  |                     |
|                        | C/O <sup>4</sup>              | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0                      | 4.29<br>(0.82)                | 9.33<br>(0.46)    | 7.06<br>(0.23)      | 5.56<br>(0.41)   | 4.03<br>(1.08)      |
| 2                      | 12.20<br>(0.44)               | 15.70<br>(1.06)   | 13.69<br>(0.86)     | 12.45<br>(0.72)  | 9.93<br>(0.14)      |
| 5                      | 18.29<br>(0.55)               | 23.10<br>(0.39)   | 19.61<br>(0.77)     | 18.89<br>(1.82)  | 16.03<br>(0.75)     |
| 8                      | 23.12<br>(1.22)               | 31.70<br>(0.81)   | 26.96<br>(0.04)     | 24.94<br>(0.21)  | 22.32<br>(1.89)     |
| 11                     | 27.41<br>(0.97)               | 32.33<br>(0.29)   | 31.65<br>(1.98)     | 28.03<br>(0.76)  | 25.15<br>(1.37)     |
| 14                     | 32.74<br>(0.57)               | 37.84<br>(0.65)   | 41.80<br>(2.59)     | 36.35<br>(2.02)  | 29.51<br>(1.00)     |
| $r^2$                  | 0.99                          | 0.98              | 1.00                | 0.99             | 0.99                |

<sup>1</sup>: Average values of two replicates and two samples.

<sup>2</sup>: Correlation coefficient of polar components% with heating time.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.

<sup>5</sup>: Standard deviations are the values in brackets.

From the Analysis of Variance, significant ( $P < 0.0001$ ) differences between the five shortenings were found in their polar component content. Also, the increase in static heating time from 0 to 14 days caused a significant ( $P < 0.0001$ ) increase in the polar component% in all shortenings. Moreover, a significant ( $P < 0.0047$ ) interaction between heating time and shortening indicated that different shortenings manifested different polar component% at different heating times.

At the end of the heating period (14 days), all shortenings had final polar component% higher than the cut-off level of 27%; therefore, they were considered abused and over heated. However, free fatty acid% represented a minute quantity of this ratio, as reported earlier in section 4.1.1 of this chapter. The data points of all curves (Fig. 4) indicated that polar component% increased significantly ( $\alpha = 0.1$ ) in a linear fashion; however C/O, CS/O and SB/OLS had a slight ( $\alpha = 0.05$ ) curvature. High positive correlation coefficients ( $r$ ) between polar component% and heating times (0.99, 0.98, 1.00, 0.99 and 0.99 for C/O, CS/O, CS/OLS, P/O and SB/OLS, respectively) were obtained. Furthermore, high correlation coefficients between polar component% and other tests are reported in Tables 31-35. No significant ( $\alpha = 0.05$ ) differences between slopes (rate of polar component% increase) and intercepts (initial polar component%) were detected among the five

## PLOT OF POLAR COMPONENT% VS HEATING TIME

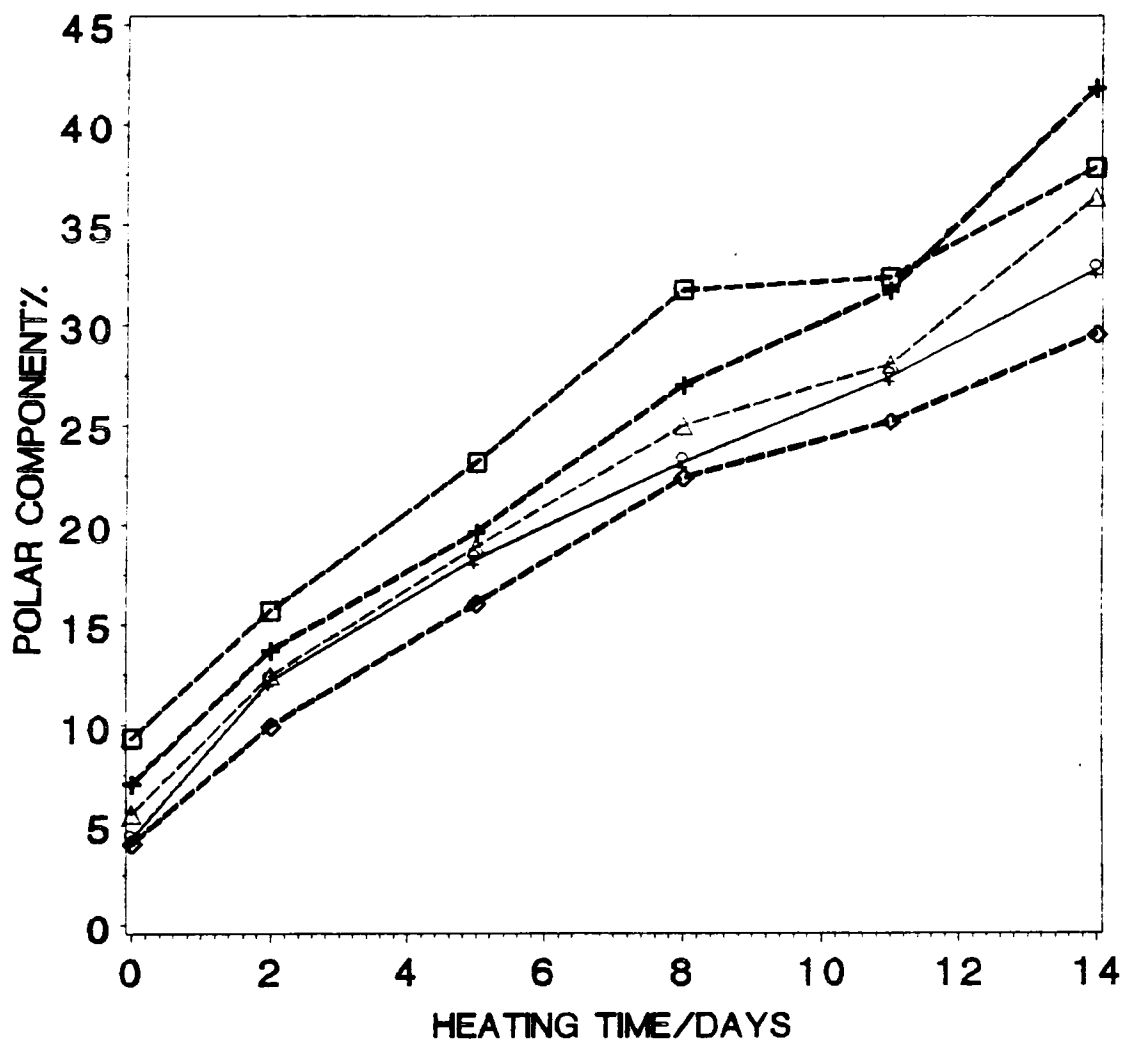


Fig. 4. Effect of static heating time on polar component% of corn (○), cottonseed (□), peanut oils (Δ), cottonseed (+) and soybean (◇) oil liquid shortenings.

shortenings. Under ideal conditions of static heating, it was proven that the polar component test is an excellent method to evaluate the deterioration rate of all the shortenings studied under the experimental conditions.

The polar component test was used as a standard test since it is known to be the most accurate and precise method to evaluate polar component% in frying shortening. Therefore, it was used to evaluate the accuracy and precision of other tests which are less time-consuming and/or less expensive, such as dielectric constant, free fatty acid%, contact angle measurement, and quantitative thin layer chromatography techniques. These tests are expected to replace the polar component% test, which is more complex, requires highly skilled personnel, is time intensive and more suited for use by regulatory agencies where precision is important.

The recommended 27% cut-off (Paradis and Nawar, 1981a,b) was reached after eight days of static heating for CS/O and CS/OLS, at or before eleven days for C/O and P/O, and after eleven days for SB/OLS. These results indicated that SB/OLS, C/O, P/O, CS/OLS and CS/O are less stable in increasing order.

By comparing both slopes, FFA from the previous section and polar component% from this section, it can be concluded that the rate of FFA% production represents a minor component if compared to the rate of production of



other polar components in the investigated shortenings. Therefore, these results indicated that thermal and oxidative deterioration of the five shortenings was promoted to a higher extent, as compared to hydrolytic deterioration, under the static heating conditions.

#### 4.1.3 VOLATILE PROFILE ANALYSIS- GAS CHROMATOGRAPHIC METHOD (GC-VPA).

Tables 6-11 present the effects of static heating time (days) on specific volatiles (such as pentane, pentanal, hexanal, t-2-heptenal, t,c- and t,t-2,4-decadienal) and on total volatiles in addition to the correlation coefficients between these volatiles and heating times. Volatiles produced from corn oil C/O (Table 6) indicated that t,t-2,4-decadienal, pentane, followed by t,c-2,4-decadienal, were major decomposition products. The production of pentane from corn oil due to the high concentration of linoleate was previously reported (Snyder et al., 1985).

A high positive correlation coefficient with heating time was obtained with pentane ( $r=0.87$ ) and t,c-2,4-decadienal ( $r=0.83$ ); however a low correlation ( $r=0.70$ ) was given by total volatiles. The situation, however, was slightly different for cottonseed oil (CS/O)

Table 6. Effect of static heating time (days) on volatiles of corn oil.

| VOLATILES <sup>4</sup> | PEAK AREA <sup>2,3</sup> |               |               |               |               |               | r <sup>1</sup> |
|------------------------|--------------------------|---------------|---------------|---------------|---------------|---------------|----------------|
|                        | HEATING TIME (DAYS)      |               |               |               |               |               |                |
|                        | 0                        | 2             | 5             | 8             | 11            | 14            |                |
| Pentane                | 110<br>(17)              | 435<br>(73)   | 560<br>(31)   | 790<br>(67)   | 835<br>(54)   | 740<br>(59)   | 0.87           |
| Pentanal               | ND <sup>5</sup>          | 350<br>(28)   | 65<br>(16)    | 90<br>(7)     | 90<br>(19)    | 95<br>(4)     | 0.64           |
| Hexanal                | 70<br>(8)                | 285<br>(17)   | 240<br>(29)   | 270<br>(60)   | 300<br>(16)   | 285<br>(9)    | 0.67           |
| t-2-Heptenal           | 50<br>(10)               | 445<br>(25)   | 425<br>(68)   | 415<br>(31)   | 330<br>(46)   | 435<br>(54)   | 0.50           |
| t,c-2,4-Decadienal     | ND                       | 590<br>(98)   | 560<br>(74)   | 590<br>(90)   | 620<br>(40)   | 650<br>(37)   | 0.83           |
| t,t-2,4-Decadienal     | 67<br>(9)                | 1975<br>(128) | 1875<br>(130) | 1845<br>(169) | 1875<br>(142) | 2100<br>(167) | 0.64           |
| Total                  | 1072<br>(340)            | 5827<br>(228) | 5670<br>(50)  | 5873<br>(468) | 6015<br>(309) | 6565<br>(143) | 0.70           |

<sup>1</sup>: Correlation coefficient of specific volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: Retention times of volatiles from top to bottom are 8.1, 15.4, 21.2, 31.8, 53.8 and 55.2 minutes.

<sup>5</sup>: Not detected.

(Table 7). Pentanal, hexanal, t,c-2,4-decadienal, t,t-2,4-decadienal, and total volatiles had high positive correlation coefficients with heating time ( $r=0.95, 0.95, 0.89, 0.83,$  and  $0.86,$  respectively). Moreover, high correlation coefficients between total volatiles and other tests are reported in Tables 31-35. Cottonseed oil (CS/O) was similar to corn oil in producing greater quantities of t,t-2,4-decadienal, t,c-2,4-decadienal, followed by pentane after 14 days of heating. Cottonseed oil liquid shortening (Table 8) also produced t,t-2,4- and t,c-2,4-decadienals, followed by pentane and hexane in greater quantities after 14 days of heating. High positive correlation coefficients of  $0.83, 0.97, 0.97, 0.85, 0.93,$  and  $0.99$  were obtained with pentane, pentanal, hexanal, t,c-2,4- and t,c-2,4-decadienal and total volatiles, respectively. The production of pentane and/or hexanal in corn oil and cottonseed oil liquid shortening results from the presence of linoleate, as reported earlier by Snyder et al. (1985). Among the primary decomposition product of peanut oil were t,t-2,4-decadienal, pentane, hexanal, followed by t,c-2,4-decadienal produced peak areas of 1710, 885, 711 and 600, respectively (Table 9). Moreover, pentane, pentanal, hexanal and total volatiles gave high positive correlation coefficients of  $0.90, 0.85, 0.92, 0.82,$  and  $0.96,$  respectively. In soybean oil liquid shortening (Table 10), hexanal was the major product in the unheated sample;

Table 7. Effect of static heating time (days) on volatiles of cottonseed oil.

| VOLATILES <sup>4</sup> | PEAK AREA <sup>2,3</sup> |               |                     |               |               |               | r <sup>1</sup> |
|------------------------|--------------------------|---------------|---------------------|---------------|---------------|---------------|----------------|
|                        | 0                        | 2             | HEATING TIME (DAYS) |               |               |               |                |
|                        |                          |               | 5                   | 8             | 11            | 14            |                |
| Pentane                | 102<br>(19)              | 1245<br>(93)  | 990<br>(31)         | 1107<br>(167) | 880<br>(74)   | 750<br>(89)   | 0.25           |
| Pentanal               | ND <sup>5</sup>          | 68<br>(8)     | 65<br>(19)          | 110<br>(17)   | 135<br>(29)   | 140<br>(14)   | 0.95           |
| Hexanal                | ND                       | 290<br>(57)   | 305<br>(69)         | 355<br>(80)   | 460<br>(96)   | 440<br>(19)   | 0.93           |
| t-2-Heptenal           | 30<br>(7)                | 530<br>(55)   | 450<br>(69)         | 420<br>(131)  | 565<br>(148)  | 485<br>(124)  | 0.60           |
| t,c-2,4-Decadienal     | ND                       | 660<br>(98)   | 646<br>(104)        | 750<br>(98)   | 915<br>(70)   | 860<br>(87)   | 0.89           |
| t,t-2,4-Decadienal     | 40<br>(8)                | 1980<br>(158) | 2055<br>(170)       | 2375<br>(189) | 2660<br>(152) | 2800<br>(187) | 0.83           |
| Total                  | 663<br>(43)              | 6332<br>(151) | 6010<br>(69)        | 7333<br>(439) | 8971<br>(188) | 9003<br>(565) | 0.86           |

<sup>1</sup>: Correlation coefficient of specific volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: Retention times of volatiles from top to bottom are 8.1, 15.4, 21.2, 31.8, 53.8 and 55.2 minutes.

<sup>5</sup>: Not detected.

Table 8. Effect of static heating time (days) on volatiles of cottonseed oil liquid shortening.

| VOLATILES <sup>4</sup> | PEAK AREA <sup>2,3</sup> |               |                     |               |               |                | r <sup>1</sup> |
|------------------------|--------------------------|---------------|---------------------|---------------|---------------|----------------|----------------|
|                        | 0                        | 2             | HEATING TIME (DAYS) |               |               |                |                |
|                        |                          |               | 5                   | 8             | 11            | 14             |                |
| Pentane                | 90<br>(11)               | 485<br>(63)   | 680<br>(51)         | 560<br>(178)  | 825<br>(144)  | 750<br>(166)   | 0.83           |
| Pentanal               | ND <sup>5</sup>          | 60<br>(9)     | 70<br>(16)          | 105<br>(19)   | 120<br>(27)   | 125<br>(24)    | 0.97           |
| Hexanal                | 48<br>(10)               | 240<br>(37)   | 270<br>(66)         | 450<br>(110)  | 480<br>(126)  | 575<br>(119)   | 0.97           |
| t-2-Heptenal           | 36<br>(5)                | 400<br>(125)  | 360<br>(79)         | 460<br>(111)  | 521<br>(48)   | 440<br>(24)    | 0.74           |
| t,c-2,4-Decadienal     | 42<br>(7)                | 670<br>(99)   | 815<br>(14)         | 780<br>(138)  | 920<br>(150)  | 1065<br>(87)   | 0.85           |
| t,t-2,4-Decadienal     | ND                       | 2425<br>(180) | 2935<br>(190)       | 2780<br>(177) | 3300<br>(123) | 4020<br>(87)   | 0.93           |
| Total                  | 3902<br>(91)             | 5556<br>(398) | 7007<br>(359)       | 7416<br>(63)  | 9135<br>(879) | 11267<br>(555) | 0.99           |

<sup>1</sup>: Correlation coefficient of specific volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: Retention times of volatiles from top to bottom are 8.1, 15.4, 21.2, 31.8, 53.8 and 55.2 minutes.

<sup>5</sup>: Not detected.

Table 9. Effect of static heating time (days) on volatiles of peanut oil.

| VOLATILES <sup>4</sup> | PEAK AREA <sup>2,3</sup> |               |               |               |               |               | r <sup>1</sup> |
|------------------------|--------------------------|---------------|---------------|---------------|---------------|---------------|----------------|
|                        | HEATING TIME (DAYS)      |               |               |               |               |               |                |
|                        | 0                        | 2             | 5             | 8             | 11            | 14            |                |
| Pentane                | 138<br>(7)               | 345<br>(53)   | 525<br>(71)   | 760<br>(78)   | 770<br>(164)  | 885<br>(126)  | 0.97           |
| Pentanal               | ND <sup>5</sup>          | 100<br>(4)    | 125<br>(14)   | 195<br>(22)   | 190<br>(32)   | 310<br>(12)   | 0.94           |
| Hexanal                | 60<br>(11)               | 320<br>(44)   | 350<br>(56)   | 455<br>(89)   | 455<br>(76)   | 711<br>(19)   | 0.93           |
| t-2-Heptenal           | 65<br>(7)                | 48<br>(5)     | 440<br>(39)   | 468<br>(13)   | 445<br>(28)   | 265<br>(34)   | 0.60           |
| t,c-2,4-Decadienal     | --                       | 330<br>(48)   | 285<br>(24)   | 360<br>(38)   | 320<br>(10)   | 600<br>(77)   | 0.72           |
| t,t-2,4-Decadienal     | 40<br>(6)                | 1048<br>(110) | 810<br>(150)  | 990<br>(187)  | 860<br>(17)   | 1710<br>(55)  | 0.73           |
| Total                  | 1001<br>(68)             | 4703<br>(164) | 4610<br>(369) | 6137<br>(304) | 7247<br>(900) | 9856<br>(614) | 0.95           |

<sup>1</sup>: Correlation coefficient of specific volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: Retention times of volatiles from top to bottom are 8.1, 15.4, 21.2, 31.8, 53.8 and 55.2 minutes.

<sup>5</sup>: Not detected.

Table 10. Effect of static heating time (days) on volatiles of soybean oil liquid shortenings.

| VOLATILES <sup>4</sup> | PEAK AREA <sup>2,3</sup> |               |               |               |               |               | r <sup>1</sup> |
|------------------------|--------------------------|---------------|---------------|---------------|---------------|---------------|----------------|
|                        | HEATING TIME (DAYS)      |               |               |               |               |               |                |
|                        | 0                        | 2             | 5             | 8             | 11            | 14            |                |
| Pentane                | 64<br>(11)               | 230<br>(55)   | 290<br>(56)   | 360<br>(87)   | 400<br>(64)   | 385<br>(26)   | 0.90           |
| Pentanal               | ND <sup>5</sup>          | 37<br>(5)     | 100<br>(13)   | 95<br>(21)    | 100<br>(12)   | 125<br>(22)   | 0.85           |
| Hexanal                | 2689<br>(221)            | 160<br>(41)   | 320<br>(16)   | 345<br>(59)   | 270<br>(86)   | 405<br>(49)   | 0.56           |
| t-2-Heptenal           | ND                       | 330<br>(15)   | 370<br>(29)   | 425<br>(43)   | 405<br>(26)   | 390<br>(44)   | 0.68           |
| t,c-2,4-Decadienal     | ND                       | 320<br>(18)   | 350<br>(25)   | 360<br>(48)   | 380<br>(22)   | 470<br>(45)   | 0.92           |
| t,t-2,4-Decadienal     | ND                       | 1130<br>(100) | 1190<br>(153) | 1210<br>(137) | 1220<br>(117) | 1580<br>(155) | 0.82           |
| Total                  | 2854<br>(53)             | 4204<br>(79)  | 4651<br>(37)  | 5320<br>(492) | 5465<br>(20)  | 7028<br>(842) | 0.96           |

<sup>1</sup>: Correlation coefficient of specific volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: Retention times of volatiles from top to bottom are 8.1, 15.4, 21.2, 31.8, 53.8 and 55.2 minutes.

<sup>5</sup>: Not detected.

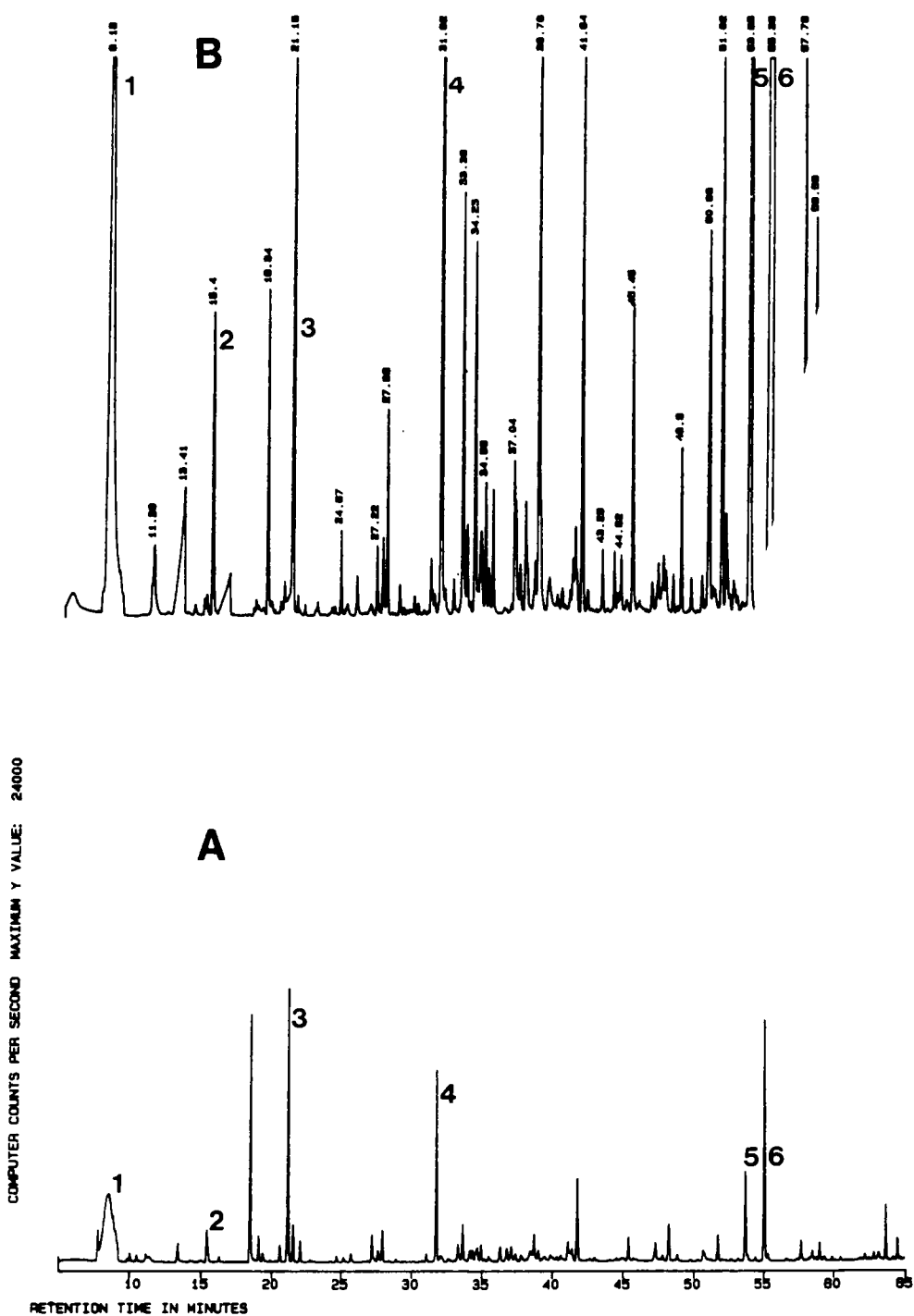


Fig. 5. Effect of static heating time (Days) on Total Volatile Profile in corn oil. A-unheated; B-heated to 14 days. Volatiles: 1=Pentane, 2=Pentanal, 3=Hexanal, 4=t-2-Heptenal, 5=t,c-2,4-Decadienal and 6=t,t-2,4-Decadienal.



Table 11. Effect of static heating time (days) on total volatiles of corn, cottonseed and peanut oils, and cottonseed and soybean oil liquid shortenings.

| SHORTENING<br>TYPE | PEAK AREA <sup>2,5</sup> |                |                |                |                 |                 | r <sup>1</sup> |
|--------------------|--------------------------|----------------|----------------|----------------|-----------------|-----------------|----------------|
|                    | HEATING TIME (DAYS)      |                |                |                |                 |                 |                |
|                    | 0 <sup>4</sup>           | 2 <sup>4</sup> | 5 <sup>4</sup> | 8 <sup>4</sup> | 11 <sup>4</sup> | 14 <sup>4</sup> |                |
| C/O <sup>3</sup>   | 1072<br>(340)            | 5827<br>(228)  | 5670<br>(50)   | 5873<br>(468)  | 6015<br>(309)   | 6565<br>(143)   | 0.70           |
| CS/O               | 663<br>(43)              | 6332<br>(151)  | 6010<br>(69)   | 7333<br>(439)  | 8971<br>(188)   | 9003<br>(565)   | 0.86           |
| CS/OLS             | 3902<br>(91)             | 5556<br>(398)  | 7007<br>(359)  | 7416<br>(63)   | 9135<br>(879)   | 11268<br>(555)  | 0.99           |
| P/O                | 1001<br>(68)             | 4703<br>(164)  | 4610<br>(369)  | 6137<br>(304)  | 7247<br>(900)   | 9856<br>(614)   | 0.95           |
| SB/OLS             | 2854<br>(53)             | 4204<br>(79)   | 4651<br>(37)   | 5320<br>(492)  | 5465<br>(20)    | 7028<br>(842)   | 0.96           |

<sup>1</sup>: Correlation coefficient of total volatiles with heating time.

<sup>2</sup>: Average peak areas of two replicates.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.

<sup>5</sup>: Standard deviations are the values in brackets.

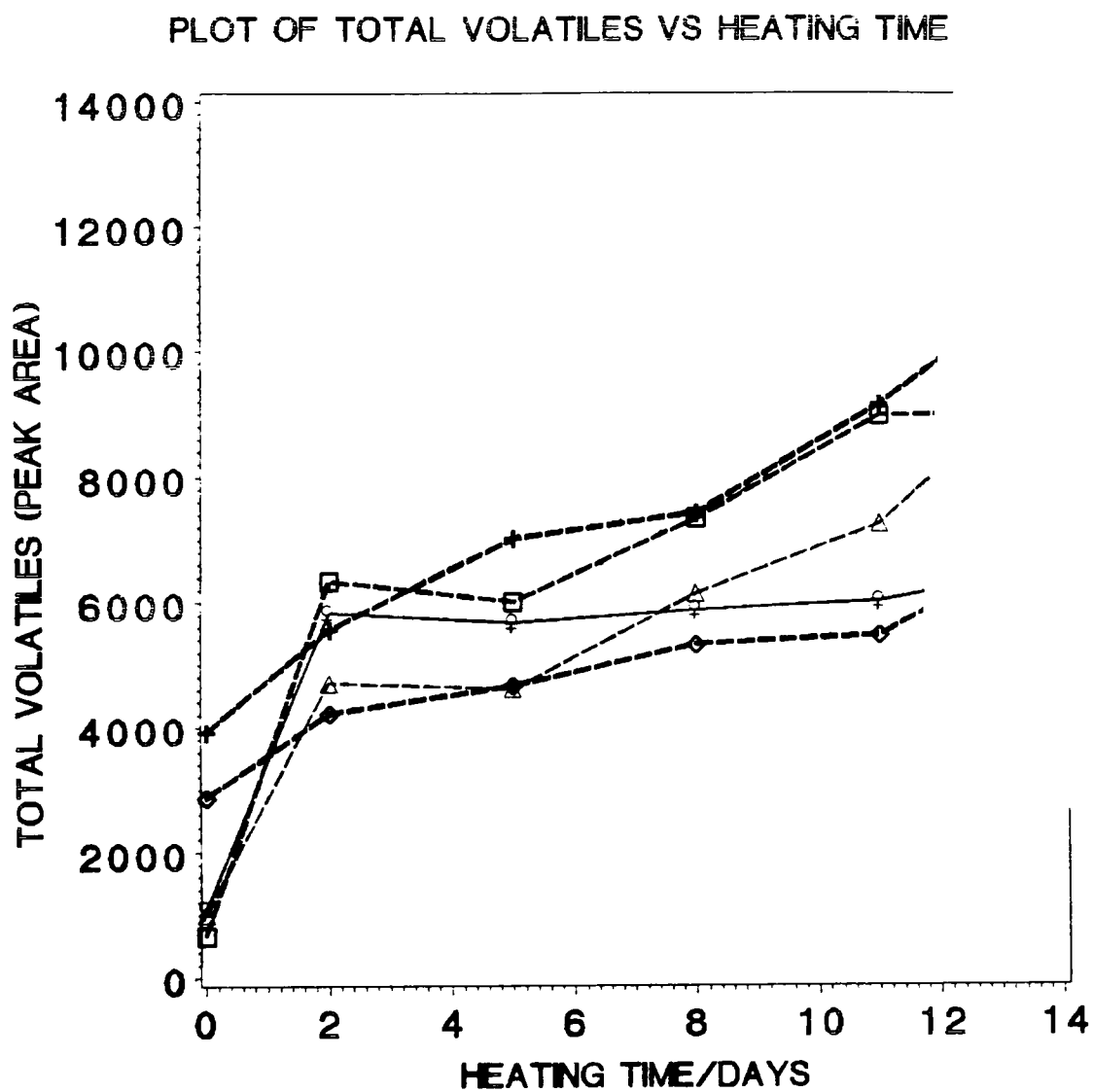


Fig. 6. Effect of static heating time on total volatiles (peak area) of corn (♀), cottonseed (□), peanut oils (△), cottonseed (+) and soybean (◇) oil liquid shortenings.

however, after two days of heating its peak area was reduced from 2689 to 160. Furthermore, t,t-2,4-decadienal was the major decomposition product. High positive correlation coefficients of 0.90, 0.85, 0.92 and 0.82 for pentane, pentanal, t,c-2,4- and t,t-2,4-decadienal, respectively, with heating time were obtained (Table 10).

The effect of static heating time on total volatiles in all five shortenings is summarized in Table 11 and Fig. 6. The total volatiles (peak area) of all fresh shortenings were significantly ( $\alpha=0.05$ ) different from each other. Moreover, a significant ( $P<0.0001$ ) effect of shortening type on changes in total volatiles was found. Heating time had a significant ( $P<0.0001$ ) effect not only on C/O, CS/O, CS/OLS, P/O, but also on SB/OLS ( $P<0.0006$ ). A typical chromatogram of unheated corn oil samples were compared with samples heated to 14 days in Fig. 5. Similar chromatograms for different shortenings are presented in Appendix D, Fig. 1-4. Moreover, typical volatiles produced from vegetable oils are presented in Appendix C, Table 2. The distribution of the data points along the regression line for CS/O, CS/OLS, P/O and SB/OLS were significantly ( $\alpha=0.1$ ) linear with a slight ( $\alpha=0.05$ ) curvature (Fig. 6). There were no significant ( $\alpha=0.05$ ) differences found among the following slopes: CS/O & CS/OLS, CS/O & P/O, and P/O and SB/OLS. However, significant ( $\alpha=0.05$ ) differences

among the intercepts of all five shortenings were detected (Fig. 6).

In conclusion, it was not possible to use either of the marker volatiles or total volatiles to predict heat abuse for all types of shortenings. However, pentane can be used successfully in C/O, CS/OLS, P/O and SB/OLS; pentanal can be used in CS/O, CS/OLS and P/O; both t,t-,t,c-2,4-decadienals can be used for CS/O, CS/OLS and SB/OLS; on the other hand, only t,t-2,4-decadienal can be used in corn oil. Finally, total volatiles are best used to predict shortening abuse for CS/O, CS/OLS, P/O and SB/OLS, but not C/O. Snyder et al. (1985) also reported that 2,4,-heptadienal is produced from linoleanate rich oils, while octanal and nonanal are produced from oleate rich oils; however, neither of these products was a major component in any of the shortenings.

The gas chromatographic analyses of specific volatiles and total volatiles produced from statically heated C/O, CS/O, CS/OLS, P/O and SB/OLS were very useful in evaluating and predicting frying shortening stability. However, it is unclear how accurate these linear relations will remain under commercial frying conditions. During frying processes, the shortening is maintained in a continuous dynamic status. Consequently major portions of the volatiles are removed with the steam generated from cooked

chicken portions; hence less volatiles will be detected with the gas chromatograph detectors.

#### 4.1.4 DIELECTRIC CONSTANT (FOODOIL-SENSOR READINGS-FOS).

The dielectric constants of fresh shortenings were in the range of 1.20 to 2.46 for oils and liquid shortenings (Table 12). From the Analysis of Variance, it was confirmed that heating time had a significant ( $P < 0.0001$ ) effect on increases in the dielectric constant measurements of all five shortenings (Fig. 7). Moreover, these measurements varied significantly ( $P < 0.0001$ ) among shortening types. After 14 days of static heating, the final measurements reached a range of 4.58 to 6.48. Soybean shortening had the lowest final value (4.58), while cottonseed shortening had the highest (6.48). All data points along the regression line were distributed significantly ( $\alpha = 0.05$ ) in the linear regression. High positive correlation coefficients ( $r$ ) between dielectric constants and heating times were obtained (0.99, 0.98, 0.99, 0.99 and 0.99 for C/O, CS/O, CS/OLS, P/O and SB/OLS, respectively). No significant ( $\alpha = 0.01$ ) differences among the slopes were observed. However, there were significant ( $\alpha = 0.05$ ) differences among the intercepts of C/O, CS/O, CS/OLS and P/O on SB/OLS but not between P/O and SB/OLS. Therefore,

Table 12. Effect of static heating time on dielectric constant of corn, cottonseed and peanut oils, and cottonseed and soybean oil liquid shortening.<sup>1</sup>

| HEATING TIME<br>(DAYS) | DIELECTRIC CONSTANT          |                   |                     |                  |                     |
|------------------------|------------------------------|-------------------|---------------------|------------------|---------------------|
|                        | SHORTENING TYPE <sup>3</sup> |                   |                     |                  |                     |
|                        | C/O <sup>4</sup>             | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0                      | 1.93<br>(0.03)               | 2.46<br>(0.01)    | 2.23<br>(0.01)      | 1.20<br>(0.01)   | 1.23<br>(0.03)      |
| 2                      | 2.80<br>(0.03)               | 3.31<br>(0.01)    | 3.00<br>(0.04)      | 2.22<br>(0.06)   | 2.03<br>(0.08)      |
| 5                      | 3.51<br>(0.03)               | 4.18<br>(0.01)    | 3.88<br>(0.07)      | 2.88<br>(0.08)   | 2.70<br>(0.06)      |
| 8                      | 3.91<br>(0.04)               | 5.05<br>(0.13)    | 4.49<br>(0.03)      | 3.76<br>(0.06)   | 3.44<br>(0.16)      |
| 11                     | 4.55<br>(0.07)               | 5.35<br>(0.06)    | 5.19<br>(0.14)      | 4.19<br>(0.04)   | 4.04<br>(0.13)      |
| 14                     | 5.26<br>(0.15)               | 6.01<br>(0.04)    | 6.48<br>(0.42)      | 5.29<br>(0.25)   | 4.58<br>(0.07)      |
| $r^2$                  | 0.99                         | 0.98              | 0.99                | 0.99             | 0.99                |

<sup>1</sup>: Average values of two replicates and two samples, and their standard deviations are the values in brackets.

<sup>2</sup>: Correlation coefficient of dielectric constant with heating time.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.

PLOT OF DIELECTRIC CONSTANTS VS HEATING TIME

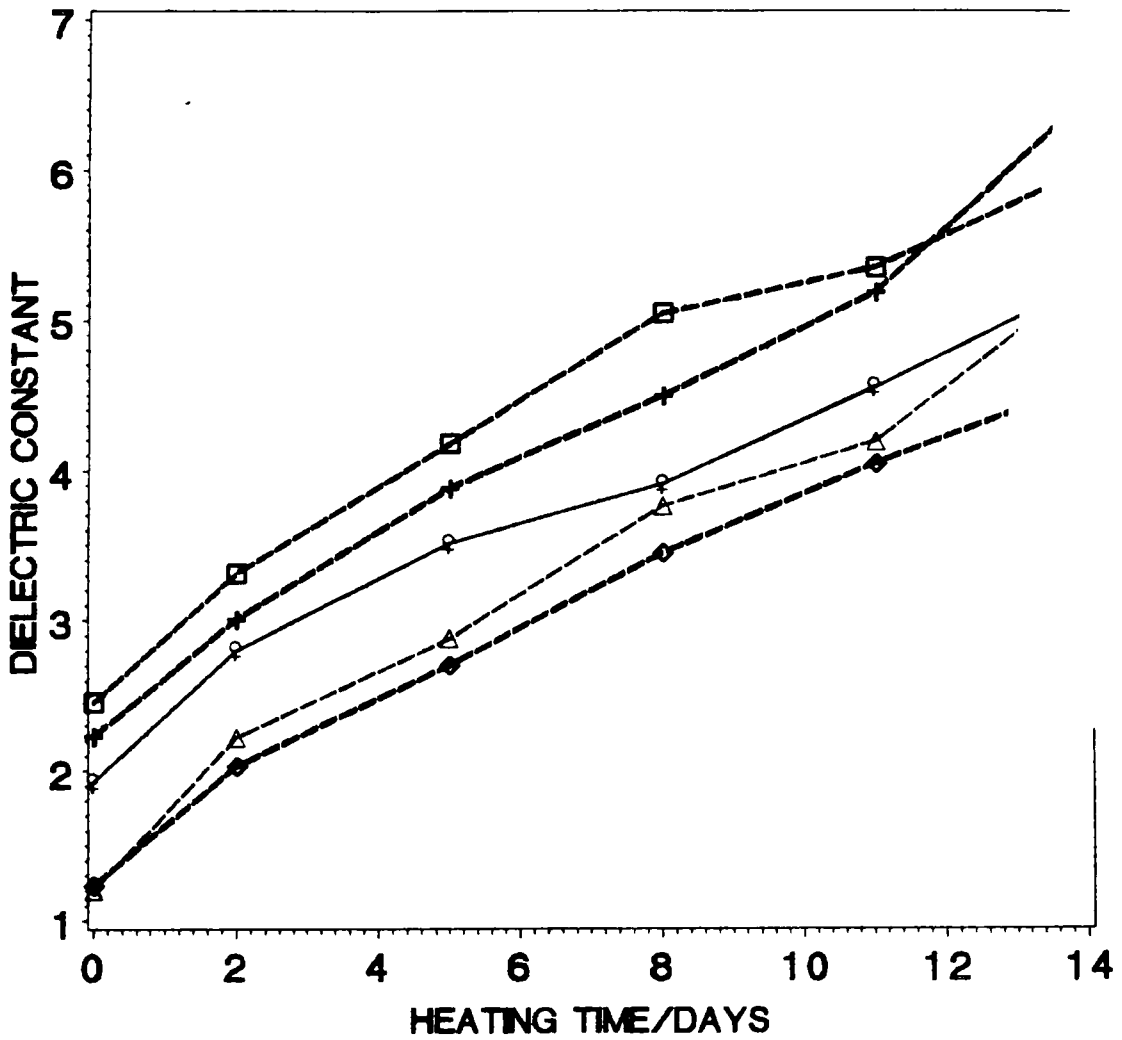


Fig. 7. Effect of static heating time on dielectric constants of corn (○), cottonseed (□), peanut oils (△), cottonseed (+) and soybean (◇) oil liquid shortenings.

the linear increase in the dielectric constants of all five shortenings can be considered a good indicator of heat abuse under static heating conditions (Fritsch et al., 1979).

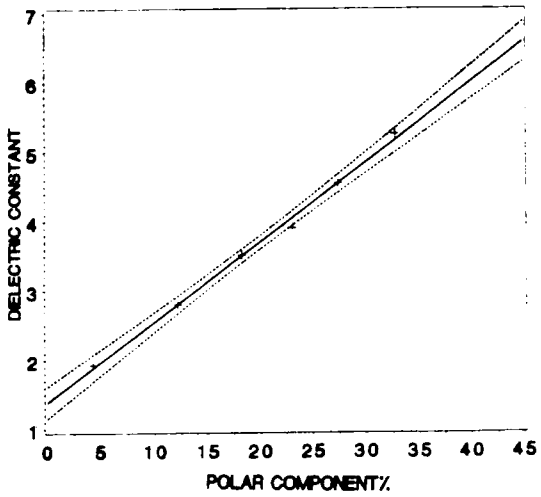
By comparing the curves of the polar component test (Fig. 4) with those of the dielectric constants (Fig. 7), it was found that the FOS readings of corn oil samples were higher than polar component% values for the same samples when compared to other shortenings. The FOS instrument may have a lower detection threshold for specific compounds produced in corn oil than those produced in peanut oil.

High correlation coefficients were obtained between the modified procedure of dielectric constant and the polar component test, and these values are  $r=1.00$  for the five tested oils and liquid shortenings (Fig. 8-A and 8-B), Paradis and Nawar, 1986; and Griffith et al., 1986).

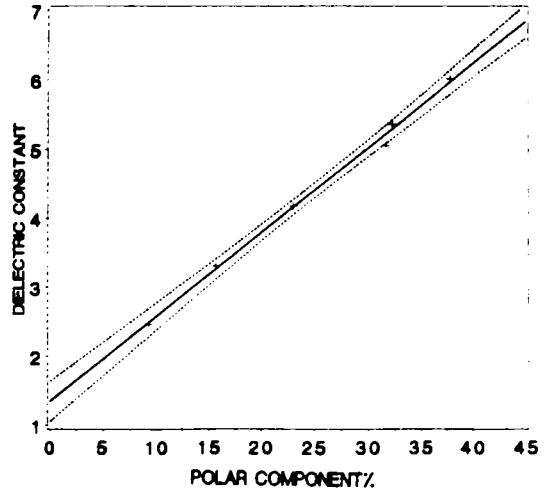
Different oils and liquid shortenings statically heated up to eleven days showed increase in the dielectric constant values (Table 12) in the following increasing order: SB/OLS, P/O, C/O, CS/OLS and CS/O. However, at fourteen days of heating, FOS values of P/O became identical to that of C/O while CS/OLS's, exceeded CS/O's. The increase in FOS readings for P/O and CS/OLS after fourteen days of heating was observed with the polar component test as well.



PLOT OF DIELECTRIC CONSTANTS VS POLAR COMPONENT%  
CORN OIL



PLOT OF DIELECTRIC CONSTANTS VS POLAR COMPONENT%  
COTTONSEED OIL



PLOT OF DIELECTRIC CONSTANTS VS POLAR COMPONENT%  
COTTONSEED OIL LIQUID SHORTENING

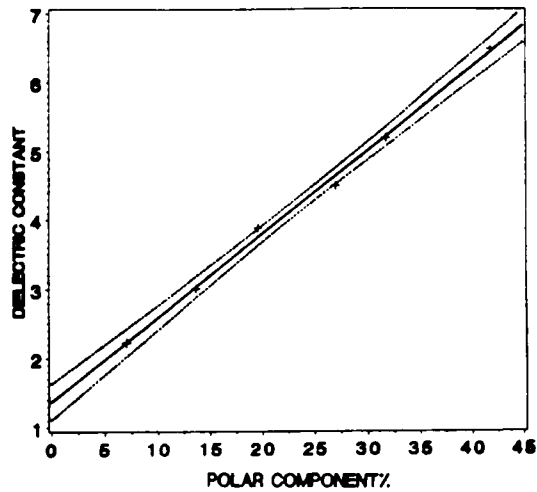


Fig. 8.A. Plots of Linear Regressions Relating Dielectric Constants with Polar Component% of Corn Oil, Cottonseed Oil and Cottonseed Oil Liquid Shortening Heated Under Static Conditions.

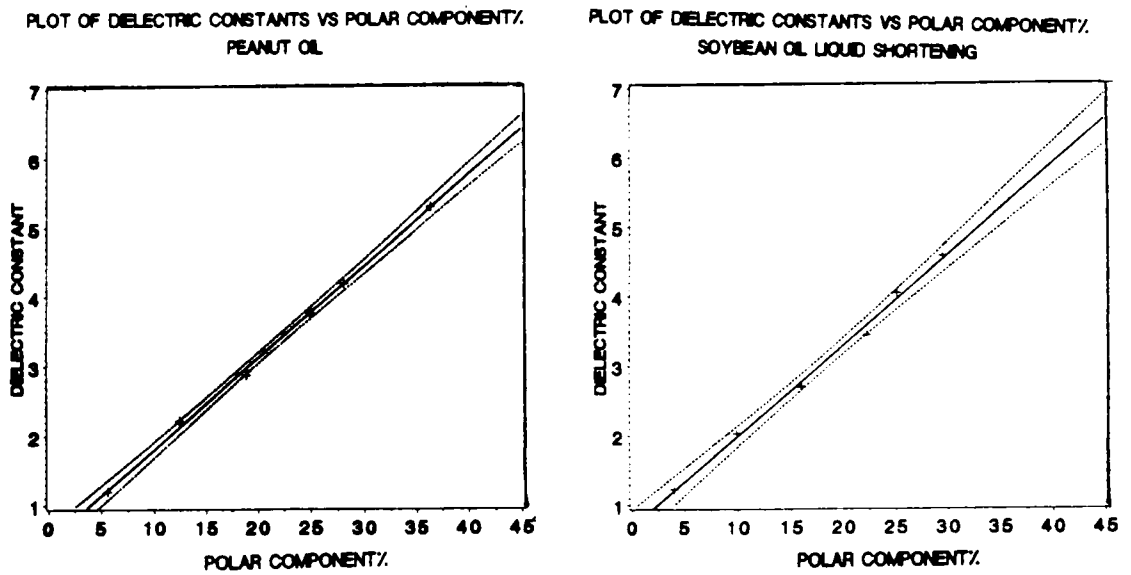


Fig. 8.B. Plots of Linear Regressions Relating Dielectric Constants with Polar Component% of Peanut Oil and Soybean Oil Liquid Shortening Heated Under Static Conditions.

From this study, it is concluded that the modified FOS technique is as accurate and precise as the conventional FOS method; however, additional advantage is obtained with the modification, a greater correlation and representation with the actual polar component% in the used and unheated shortenings.

The test is fast and simple and requires no reagents and only limited skills. However, three conditions must be met to obtain accuracy and precision:

1. A constant shortening volume (0.4ml) must be used to reduce FOS reading variation;

2. Thirty seconds must elapse after the test light (green) is lit to take measurements.

3. The use of fresh shortening must be replaced with the use of 0.00 test oil which is provided with the instrument.

Therefore, the dielectric constants of unheated shortenings will provide information about their quality.

#### 4.1.5 VISCOSITY.

The viscosity in centipoise (CP) was measured in unheated oils and liquid shortenings at 40°C and the range was from 35.8 to 60.73 CP (Table 13). Heating times up to 14 days at 360°C had a significant ( $P < 0.0001$ ) effect on the

Table 13. Effect of static heating time on viscosity measurement of corn, cottonseed and peanut oils, and cottonseed and soybean oil liquid shortening (centipoise) at 40°C.

| HEATING TIME<br>(DAYS) | VISCOSITY (CP) <sup>1</sup>  |                   |                     |                  |                     |
|------------------------|------------------------------|-------------------|---------------------|------------------|---------------------|
|                        | SHORTENING TYPE <sup>3</sup> |                   |                     |                  |                     |
|                        | C/O <sup>4</sup>             | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0                      | 35.80<br>(0.06)              | 40.52<br>(1.73)   | 60.73<br>(0.05)     | 42.85<br>(0.47)  | 50.30<br>(0.00)     |
| 2                      | 37.71<br>(0.11)              | 44.57<br>(1.34)   | 67.11<br>(1.64)     | 45.30<br>(1.22)  | 77.68<br>(2.28)     |
| 5                      | 43.75<br>(0.86)              | 48.76<br>(1.49)   | 87.29<br>(2.61)     | 55.75<br>(1.88)  | 79.05<br>(2.23)     |
| 8                      | 53.27<br>(0.61)              | 59.56<br>(2.26)   | 160.09<br>(2.06)    | 61.84<br>(0.50)  | 113.05<br>(1.41)    |
| 11                     | 56.44<br>(0.72)              | 64.23<br>(3.42)   | 174.57<br>(3.21)    | 73.85<br>(0.93)  | 124.49<br>(2.08)    |
| 14                     | 65.99<br>(1.81)              | 77.02<br>(2.06)   | 556.28<br>(8.83)    | 82.27<br>(2.03)  | 136.56<br>(1.42)    |
| r <sup>2</sup>         | 0.99                         | 0.99              | 0.83                | 1.00             | 0.98                |

<sup>1</sup>: Average values of two replicates and two samples, and their standard deviations are the values in brackets.

<sup>2</sup>: Correlation coefficient of viscosity with heating time.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.

increase in viscosity for all five shortenings (Fig. 9). Also, these measurements significantly ( $P < 0.0001$ ) varied with shortening type. At the end of the heating periods, the viscosity measurements ranged from 65.99 to 556.28 CP. Corn oil had the lowest value, while cottonseed oil liquid shortening had the highest. All the final viscosity measurements on day 14 of heating time were related to the initial values before heating. This outcome might indicate that the initial concentration of polymers in the fresh shortening samples is an important factor in enhancing the rate of polymerization from triglycerides, particularly under static heating conditions.

The data points of viscosity measurements as related to heating time were distributed significantly ( $\alpha = 0.1$ ) in a linear fashion; however, CS/OLS and SB/OLS had some curvature ( $\alpha = 0.05$ ). No significant ( $\alpha = 0.01$ ) differences were observed among the slopes (rate of viscosity increase) of C/O, CS/O and P/O. There were, however, significant ( $\alpha = 0.01$ ) differences existed among C/O & CS/OLS, C/O & SB/OLS, CS/O & CS/OLS, CS/O & SB/OLS, P/O & CS/OLS, SB/OLS & CS/OLS and P/O & SB/OLS, where the first slope was smaller than the second in the series. Furthermore, significant differences among the intercepts of all five shortenings were observed. High positive correlation coefficients, (0.99, 0.99, 0.83, 0.99 and 0.99) among viscosity and heating time were observed for C/O, CS/O,

CS/OLS, P/O, and SB/OLS, respectively. Furthermore, high correlation coefficients between viscosity and other tests are reported in Tables 31-35.

The major conclusions that can be drawn from these results are as follows: the increase in viscosity with heating time can be considered a good indication of heat abuse of commercial frying fats under static heating conditions; and the polymerization rate (rate of increase in viscosity) was higher in CS/OLS and SB/OLS when compared with C/O, CS/O and P/O.

The increase in CS/OLS viscosity after 11 days of heating was out of proportion when compared to all other shortenings (Fig. 9). This increase indicated a high rate of polymerization.

#### 4.1.6 POLAR COMPONENT% BY QUANTITATIVE HIGH PERFORMANCE THIN LAYER CHROMATOGRAPHY (QHPTLC).

##### 4.1.6.1 Solvent System Optimization.

The objective of this preliminary study was the selection of an appropriate solvent system to separate nonpolar lipids from the polar fraction into two defined bands representative of the actual polarity in the shortening.

## PLOT OF VISCOSITY VS HEATING TIME

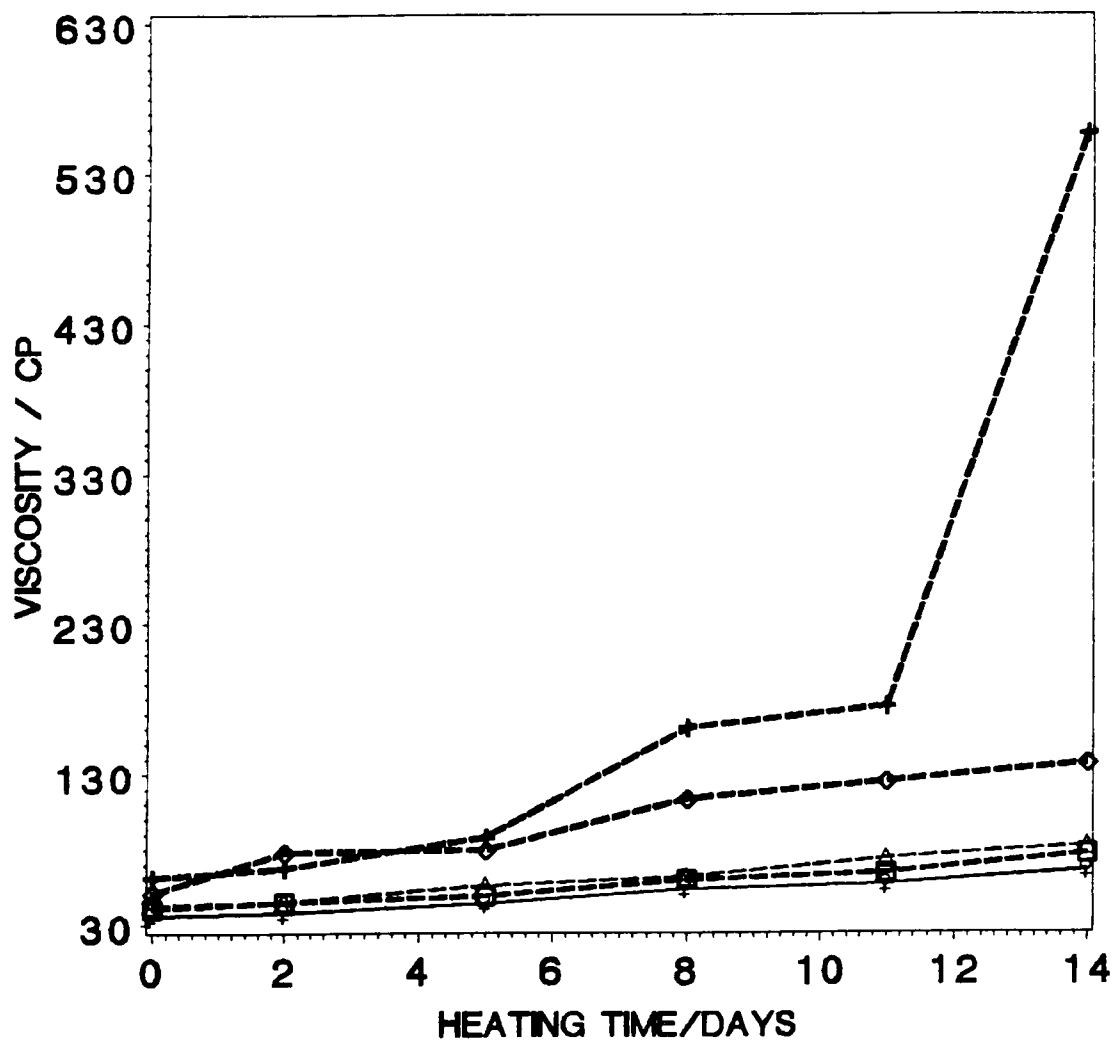


Fig. 9. Effect of static heating time on viscosity (CP) of corn (⊗), cottonseed (□), peanut oils (Δ), cottonseed (+) and soybean (◇) oil liquid shortenings.

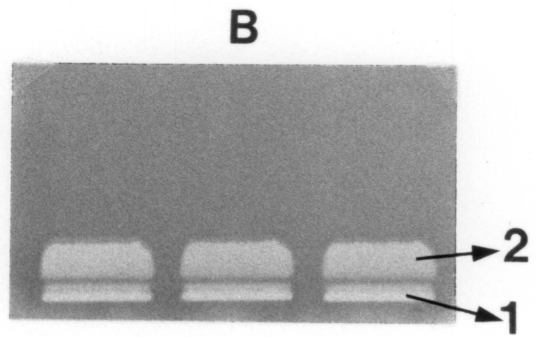
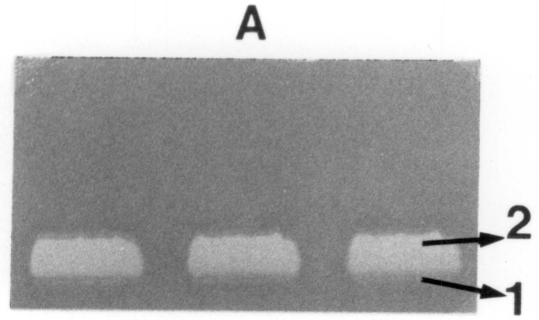
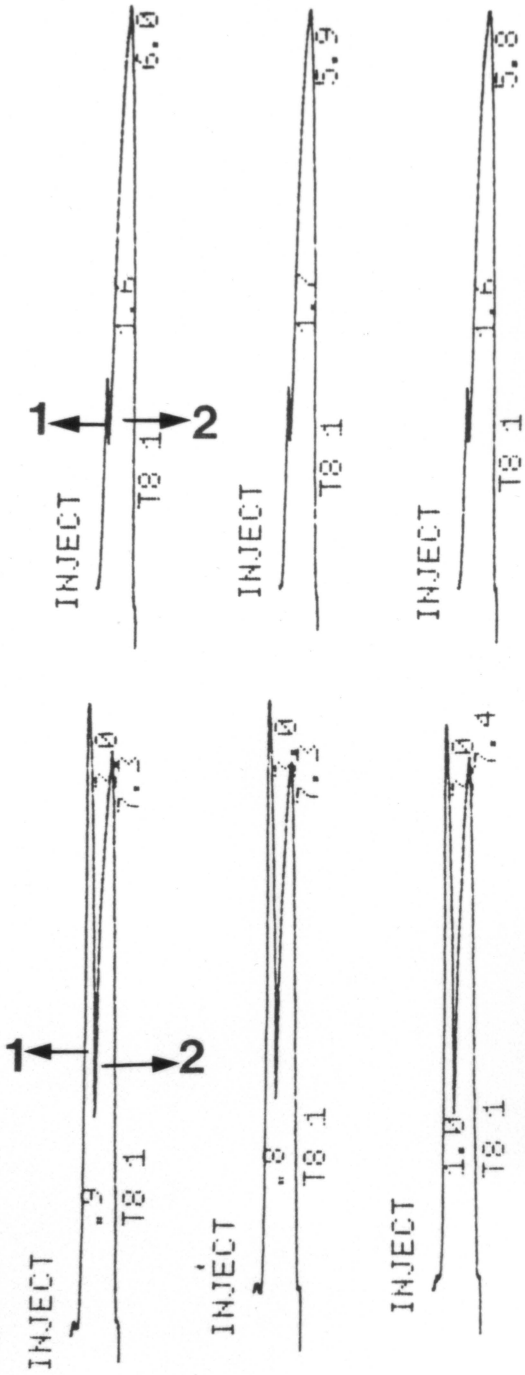
Two trails were attempted in order to optimize band separations. The first attempt was with three solvents tested in twenty-two combinations for this purpose, with and without acetic acid, were petroleum ether, methanol, and diethyl ether (Table 2) as presented in chapter III. The second trail was with eight solvents in twenty-nine combinations (Table 3) as presented in chapter III. These solvents were acetone, chloroform, benzene, xylenes, acetonitrile, ethanol, diethyl ether, petroleum ether, used with or without the addition of acetic acid. From this experiment, solvent system #29 85:15 xylenes; pet. ether was selected with no further change in its polarity (Fig. 10). From the results obtained, it was observed that petroleum ether was very nonpolar and did not have sufficient affinity for the nonpolar fraction of the shortening and a separation did not occur. When xylenes (mixture of o-, m- and p-xylenes) were added, up to 85%, the polarity of the system was increased enough to produce the best resolution. The  $R_f$  value of the polar band was almost zero using this solvent environment.

#### 4.1.6.2 Sample application, plate development, dye application and scanning quantitation.

This section was previously explained in detail in chapter III, sections 3.1.1.6.5 to 3.1.1.6.9.



Fig. 10. Polar Component% as determined by Quantitative High Performance Thin Layer Chromatography (QHPTLC). A-unheated corn oil; B-14 day statically heated corn oil. 1-polar band or peak; 2-nonpolar band or peak.



#### 4.1.6:3 Polar component% estimation by QHPTLC.

The polar component% of the five types of shortening samples was determined before and after static heating on QHPTLC plates (Table 14). In general, unheated cottonseed (CS/O) and peanut (P/O) oils had a high initial polar component%, 29.65 and 21.38%, respectively, while corn oil (C/O), cottonseed (CS/OLS) and soybean (SB/OLS) oil liquid shortenings had lower polar component%, 10.49, 7.48 and 9.66%, respectively. Analysis of variance indicated a significant ( $P < 0.0001$ ) effect of heating time in the increases of polar component% by QHPTLC in all five shortenings (Fig. 11). Also, significant ( $P < 0.0001$ ) differences on the increase in polar component% were found among all shortenings. Different plates had significant ( $P < 0.0001$ ) effect on changes in the polar component% of frying shortenings. Moreover, a significant ( $P < 0.0001$ ) interaction between heating time and shortening type was found, indicating that different shortenings had different polar component% at different heating times.

The final polar component% ranged from 43.23 to 49.83% in all shortenings. Plotted data were significantly ( $\alpha = 0.1$ ) linear for all five shortenings. However, C/O and CS/O had a slight ( $\alpha = 0.05$ ) curvature. When the slopes (rate of polar component% increase) were compared, no significant ( $\alpha = 0.01$ ) differences in shortenings were

Table 14. Effect of static heating time on polar component percent as determined by quantitative high performance thin layer chromatography (QHPTLC).

| POLAR COMPONENT% BY QHPTLC <sup>1,5</sup> |                              |                   |                     |                  |                     |
|---|------------------------------|-------------------|---------------------|------------------|---------------------|
| HEATING TIME<br>(DAYS)                    | SHORTENING TYPE <sup>3</sup> |                   |                     |                  |                     |
|   | C/O <sup>4</sup>             | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0   | 10.49<br>(3.62)              | 29.65<br>(0.67)   | 7.48<br>(0.75)      | 21.38<br>(3.24)  | 9.66<br>(0.63)      |
| 2   | 23.42<br>(1.32)              | 36.40<br>(1.70)   | 18.02<br>(0.76)     | 28.92<br>(4.82)  | 19.18<br>(1.28)     |
| 5   | 29.73<br>(4.76)              | 38.61<br>(3.29)   | 25.81<br>(2.11)     | 32.37<br>(3.69)  | 26.95<br>(3.18)     |
| 8   | 35.62<br>(2.32)              | 43.91<br>(2.58)   | 31.18<br>(2.13)     | 39.25<br>(2.01)  | 37.27<br>(2.35)     |
| 11  | 40.28<br>(3.34)              | 41.85<br>(4.93)   | 29.81<br>(0.59)     | 40.38<br>(2.13)  | 43.64<br>(3.39)     |
| 14  | 44.30<br>(1.39)              | 46.33<br>(4.97)   | 43.23<br>(4.96)     | 45.83<br>(1.61)  | 49.83<br>(2.89)     |
| $r^2$                                     | 0.96                         | 0.92              | 0.95                | 0.98             | 0.99                |

<sup>1</sup>: Average of (average of 12 measurements) two replicates, three samples on two plates.

<sup>2</sup>: Correlation coefficient of polar component% with heating time.

<sup>3</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

Table 14. cont.

- 
- <sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.
- <sup>5</sup>: Standard deviations are the values in brackets.

PLOT OF POLAR COMPONENT%. BY QHPTLC VS HEATING TIME

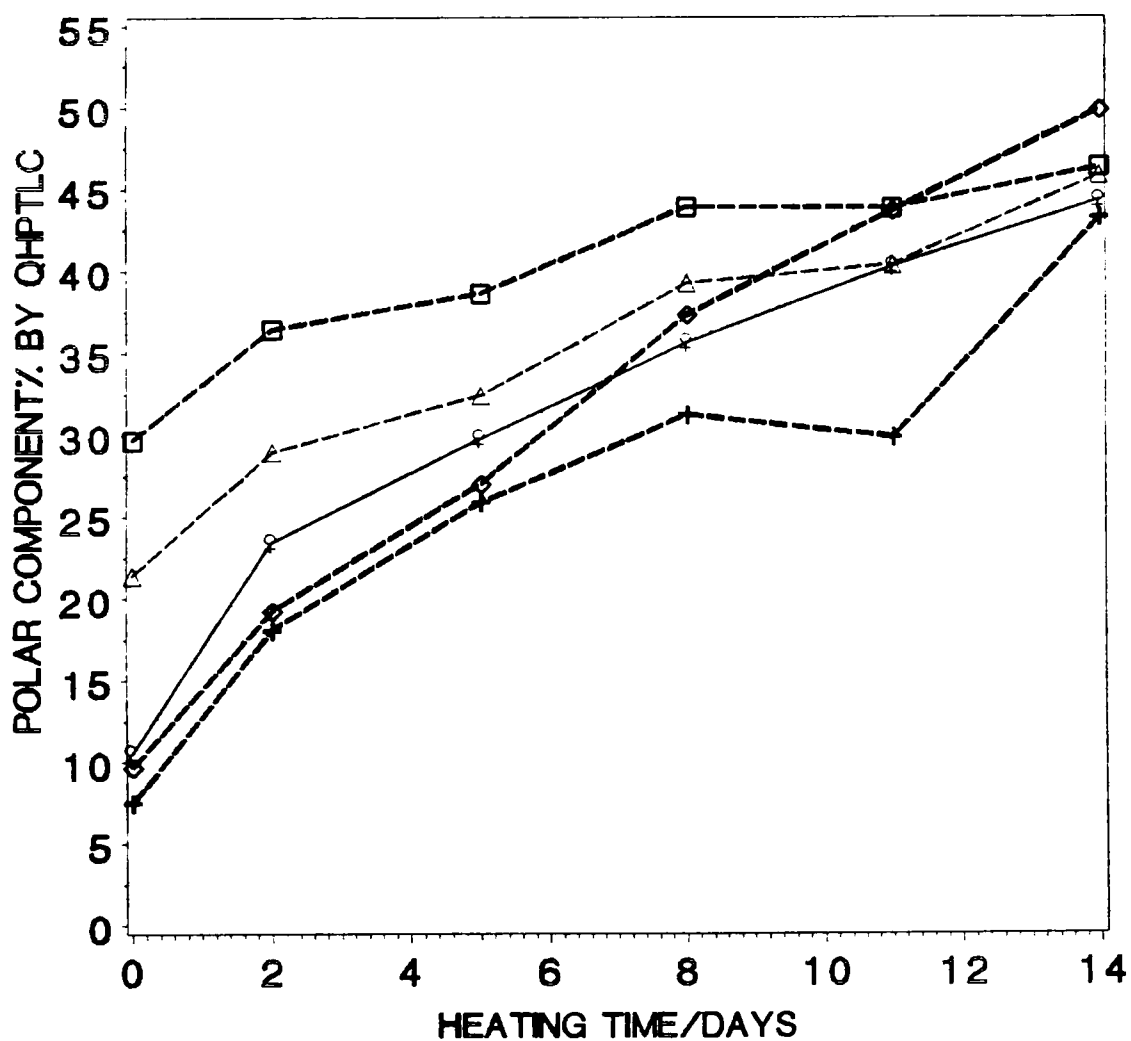


Fig. 11. Effect of static heating time on polar component% by QHPTLC of corn (♀), cottonseed (□), peanut oils (Δ), cottonseed (+) and soybean (◇) oil liquid shortenings.

noticed, with the exception of CS/O and SB/OLS; the SB/OLS slope was significantly greater than CS/O's, which is contradicting to the results obtained from the polar component test (section 4.1.2.). This observation indicated QHPTLC procedures are more sensitive in detecting polar components in fresh samples than the standard method. There were significant ( $\alpha=0.05$ ) differences among the intercepts of all five shortenings. However, no significant ( $\alpha=0.05$ ) differences were found among intercepts (unheated samples) of polar component% (section 4.1.2). In general, high initial polar component% may lead to higher final polar component% (Stevenson et al., 1984b). Although SB/OLS had a relatively low initial polar component%, a high rate of deterioration (high slope) resulted in the highest final polar component%. This indicated that there were other factors that affected the deterioration rate, such as the quality and the quantity of triglycerides, which are the major chemical components of any shortening.

It was difficult to obtain the actual polar component percentage in the shortenings from the QHPTLC technique. This method magnified the initial polar component percent particularly for peanut oil and cottonseed oil. The QHPTLC values were 21.38 and 29.65%, respectively, while readings of 5.56 and 9.33% were determined by open column chromatography, respectively. However, the high

correlation obtained (Tables 25-29) between the QHPTLC and open column methods indicated that the QHPTLC technique is precise and can be used to replace the open column technique particularly after obtaining a standard curve for each shortening under specific heating conditions.

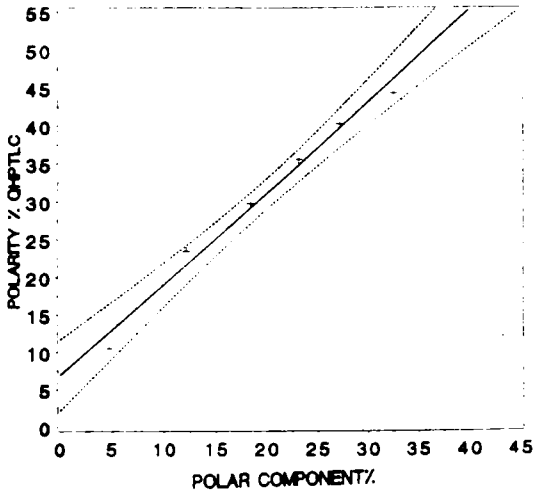
High correlation coefficients were obtained between the polar component% by QHPTLC and the polar component test, and these values were  $r=0.99$ ,  $0.98$ ,  $0.97$ ,  $0.99$  and  $0.96$  for C/O, CS/O, CS/OLS, P/O and SB/OLS, respectively (Fig. 12-A and 12-B).

#### 4.1.6.4 Advantages and limitations of QHPTLC in evaluating polar component%.

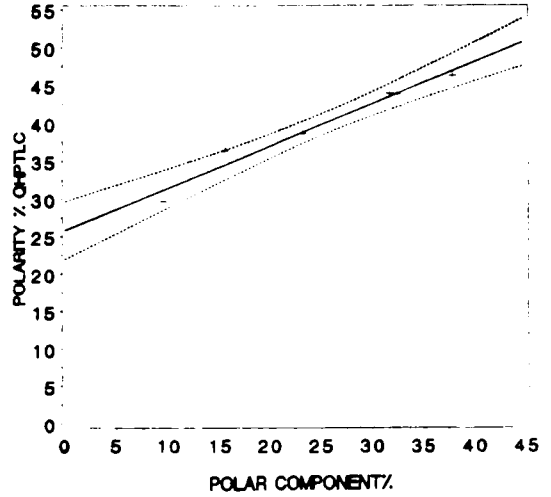
The advantage of using this QHPTLC technique can be explained as follows: polar component% quantitation of 12 shortening samples in duplicate required 6 plates, 18 ml of solvent and one working day, while an open column chromatography method for 12 shortening samples in duplicate required 24 prepared columns, 4.8 liters of solvent and six working days. Nevertheless, there are three disadvantages of using QHPTLC: the high cost of instrumentation; personnel training time and different HPTLC plates have significant effects on polar component% which may influence reproducibility.



PLOT OF POLAR COMPONENT% BY QHPTLC VS POLAR COMPONENT%  
CORN OIL



PLOT OF POLAR COMPONENT% BY QHPTLC VS POLAR COMPONENT%  
COTTONSEED OIL



PLOT OF POLAR COMPONENT% BY QHPTLC VS POLAR COMPONENT%  
COTTONSEED OIL LIQUID SHORTENING

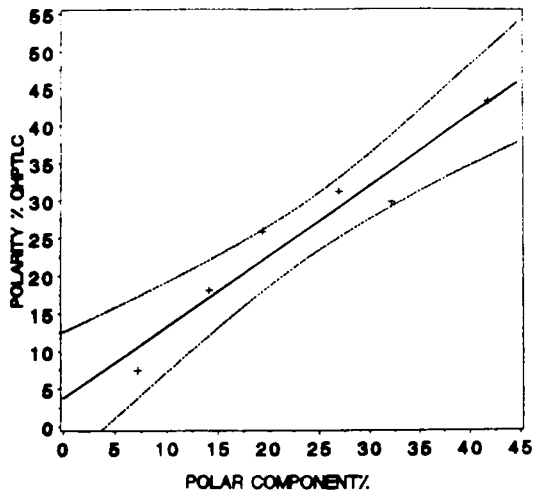


Fig. 12.A. Plots of Linear Regressions Relating Polar Component% by (QHPTLC) with Polar Component% of Corn Oil, Cottonseed Oil and Cottonseed Oil Liquid Shortening Heated Under Static Conditions.

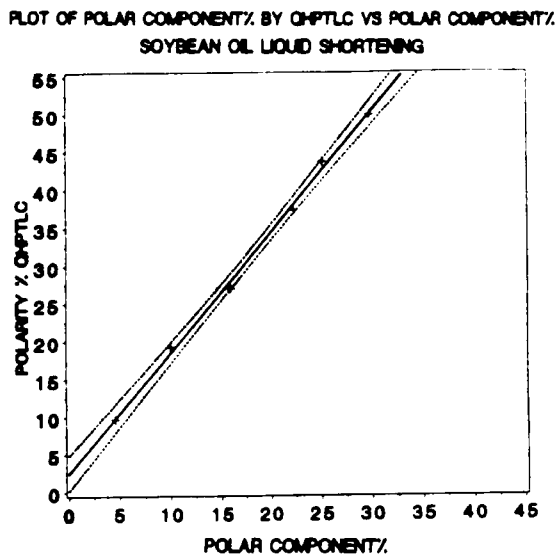
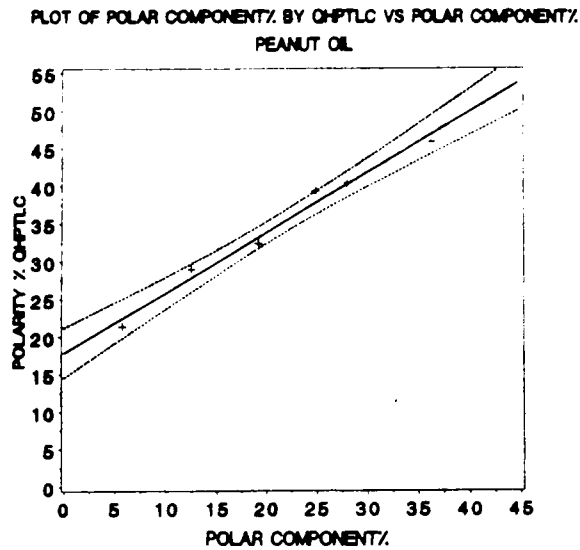


Fig. 12.B. Plots of Linear Regressions Relating Polar Component% by (QHPTLC) with Polar Component% of Peanut Oil and Soybean Oil Liquid Shortening Heated Under Static Conditions.

#### 4.1.7 CONTACT ANGLE.

Fifteen types of surfaces (as described in chapter III) were tested for their effectiveness to differentiate between unheated and heated frying shortenings. From these surfaces, FT-120-WW was chosen because it showed better selectivity toward oils heated for different time periods. Figure 13 gives an example of two contact angles, one for unheated oil and other for oil heated statically up to 14 days, and Figure 14 includes a scheme of the FT-120-WW surface in detail. It was further found that this surface exhibited improved selectivity four days after annealing, as reported earlier in chapter III.

In order to understand the unusual behavior of this surface, two surface analyses were conducted using Electron Spectroscopy For Chemical Analysis (ESCA) and High Resolution Scanning Electron Microscopy (HSEM).

##### 4.1.7.1. Electron Spectroscopy For Chemical Analysis (ESCA) of the FT-120-WW surface.

The objective of this experiment was to investigate the modification of the FT-120-WW pellet surface after annealing. Such understanding might help in designing a new mixture of FT-120-WW components which would provide a

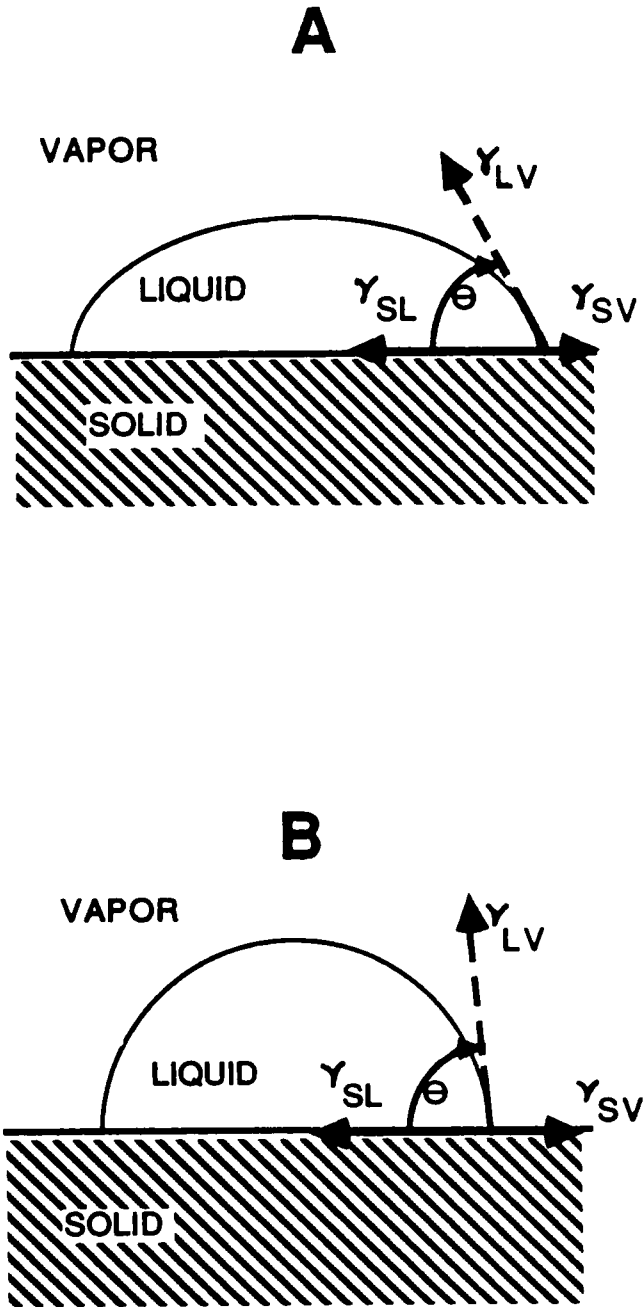


Fig. 13. Scheme of two sessile drops of frying oils and their contact angles with a FT-120-WW solid surface. A-unheated corn oil sample; B-14 day statically heated oil sample.

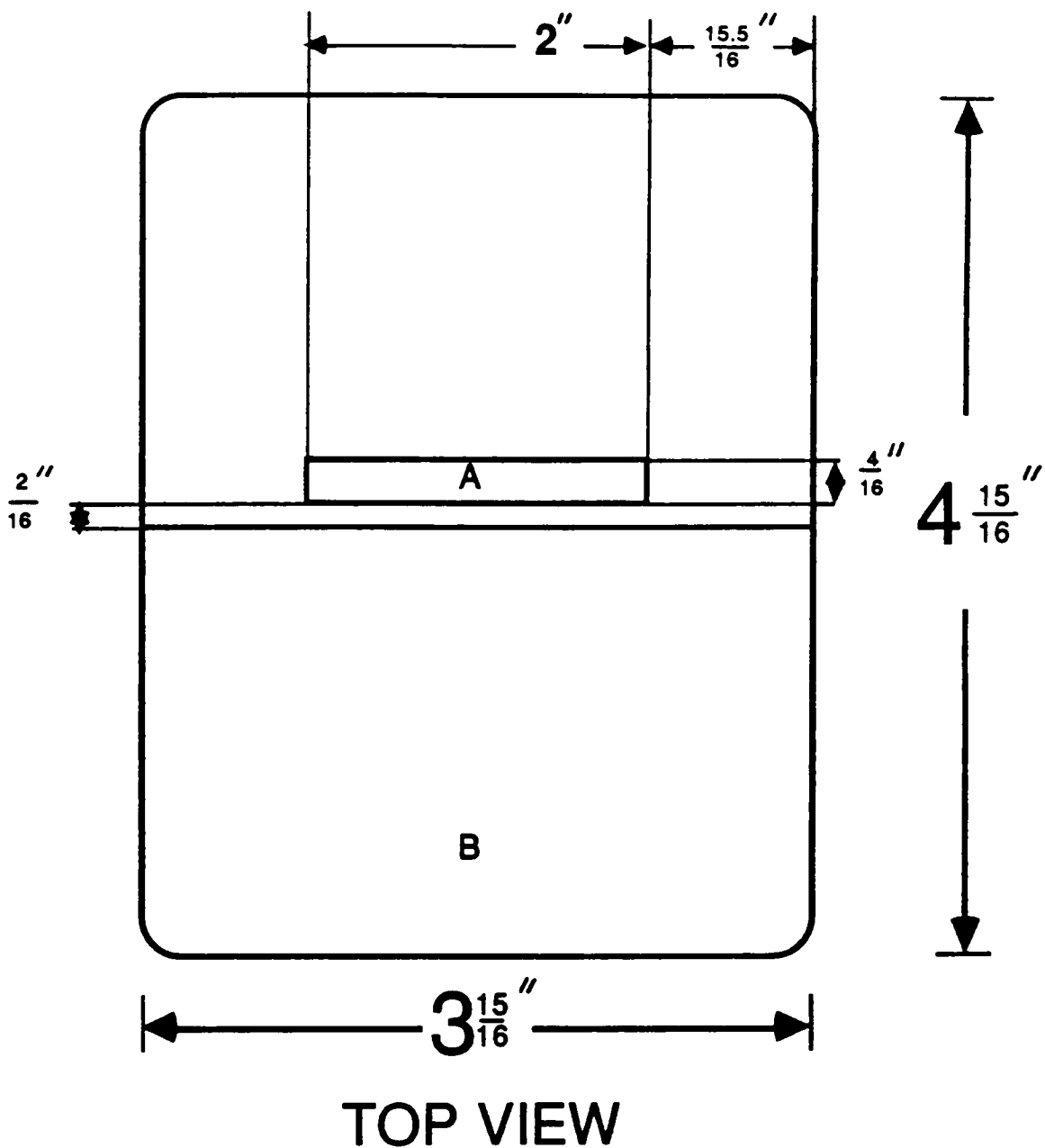


Fig. 14. Scheme of FT-120-WW surface used for contact angle measurement of oils heated to varying periods of time. A-contact angle measuring area; B-handling area.

highly selective and efficient pellet for contact angle measurement of both fresh and heated frying oils or fats.

Step pellets of FT-120-WW were molded from polypropylene mixed with an antistatic agent (mono and distearin at 1.25%) and 30 ppm calcium stearate, which served as a hydrochloric acid scavenger. Immediately after annealing (c) (Tables 15 and 16), the surface-free energy increased toward fresh corn oil and heat abused-corn oil, resulting in lower contact angles. However, after four days of annealing the surface exhibited a selectivity between fresh corn oil and the heat-abused corn oil. This selectivity was associated with a decrease in surface-free energy resulting in a contact angle increase of both the fresh and abused corn oil. After six days of storage (E), the surface lost its selectivity while maintaining a high surface-free energy.

Five types of treated surfaces were analyzed by ESCA, and the results from this study are summarized in Tables 15 and 16. The atomic percents for carbon, oxygen and calcium are presented in Table 15. Also, the atomic ratios for carbon to oxygen (C/O), carbon to calcium (C/Ca) and oxygen to calcium (O/Ca) are presented in Table 15. After washing the FT-120-WW surface with methanol (B), complete elimination of calcium from the surface was observed (Fig. 15-b). Changes in the surface-free energy of FT-120-WW (B) was predicted by measuring changes in the contact angles of

Table 15. The ESCA (XPS) obtained atomic concentration% and binding energy of selected surface elements of the FT-120-WW.

| ATOM <sup>4</sup> |                 | SURFACE TREATMENTS <sup>1</sup> |       |                 |       |       |
|-------------------|-----------------|---------------------------------|-------|-----------------|-------|-------|
|                   |                 | A                               | B     | C               | D     | E     |
| C                 | BE <sup>2</sup> | 285.0                           | 285.0 | 284.8           | 284.9 | 284.8 |
|                   | BE <sub>3</sub> | 289.1                           | 289.0 | ND <sup>5</sup> | ND    | ND    |
|                   | AP <sup>3</sup> | 88.26                           | 95.50 | 96.49           | 95.19 | 95.95 |
| O                 | BE              | 532.8                           | 532.6 | 531.7           | 532.1 | 532.0 |
|                   | AP              | 11.69                           | 4.50  | 3.04            | 4.36  | 3.69  |
| Ca                | BE              | ND                              | ND    | 347.4           | 347.5 | 351.0 |
|                   | BE              | ND                              | 0.00  | 351.0           | 351.0 | 347.5 |
|                   | AP              | 0.04                            | 0.00  | 0.47            | 0.45  | 0.36  |

<sup>1</sup>: A (Original), B (Methanol washed), C (Annealed), D (Annealed + 4 day storage) and E (Annealed + 6 day storage).

<sup>2</sup>: Binding energy.

<sup>3</sup>: Atomic percentage.

<sup>4</sup>: C (Carbon), O (Oxygen) and Ca (Calcium).

<sup>5</sup>: Not detected.

Table 16. ESCA atomic ratios of the FT-120-WW replicates.

| ATOMIC<br>RATIOS              | SURFACE TREATMENTS <sup>1</sup> |      |       |       |       |
|-------------------------------|---------------------------------|------|-------|-------|-------|
|                               | A                               | B    | C     | D     | E     |
| C/O                           | 7.6                             | 21.2 | 31.7  | 21.8  | 26.0  |
| C/Ca                          | 2206.5                          | --   | 205.3 | 211.5 | 266.5 |
| O/Ca                          | 292.3                           | --   | 6.5   | 9.7   | 10.3  |
| CON <sub>2</sub> <sup>2</sup> | 50°                             | 25°  | 30°   | 37.5° | 45°   |
| CON <sub>3</sub> <sup>3</sup> | 50°                             | 25°  | 30°   | 42.5° | 45°   |

<sup>1</sup>: A (Original), B (Methanol washed), C (Annealed), D (Annealed + 4 day storage) and E (Annealed + 6 day storage).

<sup>2</sup>: Contact angles of fresh corn oil.

<sup>3</sup>: Contact angles of abused corn oil (heated to 14 days).



fresh corn oil and corn oil heated statically to 14 days at 360°F. Both contact angles were reduced from 50° to 25°. This behavior indicated that methanol removed the antistatic agents, mono- and distearin (MDS), and the acid scavenger, calcium stearate (CS), exposing the polypropylene surface which had a higher surface free energy (less surface tension) than the lipids on the surface.

From the changes in the atomic percents, atomic ratios, surface-free energy and selectivity (Tables 15 and 16), the following observations can be made (summarized in Fig. 15): as a result of annealing, the O/Ca and C/Ca ratios decreased from 2206.5 and 292.3 (A) to 205.3 and 6.5 (C), respectively. This decrease was accompanied by an increase in calcium atomic% from 0.04 to 0.47 (a 1175% increase). Moreover, the surface-free energy increased as indicated by a decrease in the contact angle from 50° to 30° without any selectivity. The ESCA analysis indicated that in the original sample (A), mono- and distearin, and calcium stearate were randomly positioned on the polypropylene surface because calcium was a minor constituent in the mix, with atomic% = 0.04 and a bonding energy of 285; also the hydrocarbon chain  $(-\text{CH}_2-\text{CH}_2-)_x$  of the stearic acid represented the major hydrocarbon form on the surface, while the hydrocarbon in samples B, C, D and E

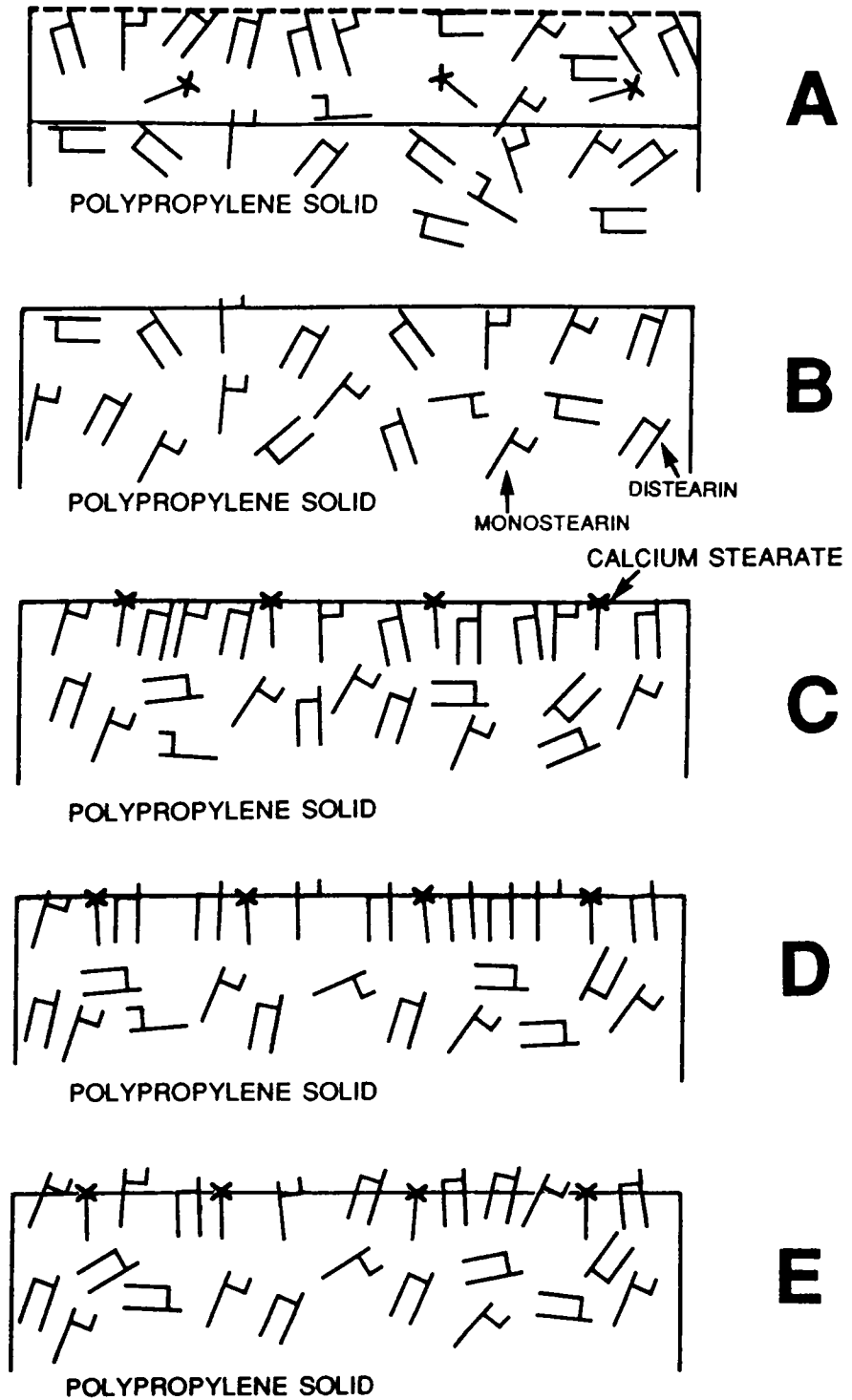


Fig. 15. Proposed scheme of FT-120-WW cross sections explains the dynamic behavior of mono- and distearate, and calcium stearate during different treatments. A-control; B-after methanol wash; C-directly after annealing; D-four day storage after annealing; E-six day storage after annealing.

were originated mostly from the polypropylene block. The contact angle of both fresh and heated oil was  $50^{\circ}$ .

In general, polypropylene is a hydrophobic polymer. Also, distearin, with two stearic acid chains in its molecule, is expected to be more hydrophobic than monostearin and calcium stearate. During the FT-120-WW annealing process at  $140^{\circ}\text{C}/2$  hr, the polypropylene became sufficiently soft and attracted the hydrophobic moities of the distearin, monostearin and calcium stearate, thereby exposing the hydrocarbon of polypropylene, calcium and the ester group. At this point, it is important to realize that the ester group carbon represents 5.56% of the stearic acid total carbon; hence it was not detected (Table 15). Changes in the ratios of different elements (Table 16), however, confirm its presence on the surface.

After four days (D) of annealing, the C/Ca ratio increased from 205.3 to 211.5, while the O/Ca ratio increased from 6.5 to 9.7. These changes indicated that mono and distearin return to the surface to partially mask the calcium and reduce its atomic% from 0.47 to 0.45. At this point, the surface manifests its best selectivity (possible differentiation among oils) toward the oils with contact angles of  $37.50^{\circ}$  and  $42.50^{\circ}$  for fresh and heated oils, respectively.

After 6 days of annealing sample (E), the C/Ca and O/Ca increased to 266.5 and 10.3, respectively; these

increases were associated with a further reduction in the calcium atomic% to 0.36, resulting in a loss of the surface selectivity. Probably after four days of annealing, a specific alignment and arrangement between calcium, the ester bond and the hydrocarbon chain resulted in the improved selectivity of the surface. Therefore, greater differences in the contact angle measurements were obtained between the unheated and heated corn oil.

Finally, the annealing process is necessary to modify the surface to the appropriate configuration. Therefore, further research is needed to improve the surface or find a new surface with improved selectivity.

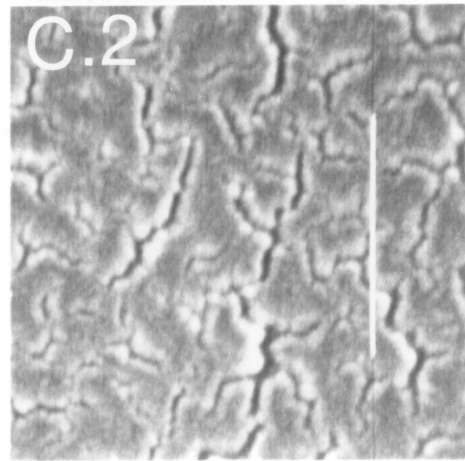
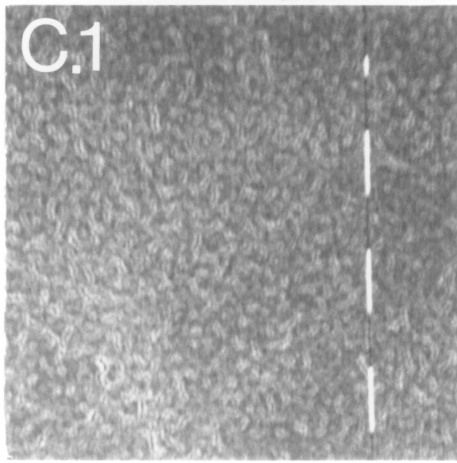
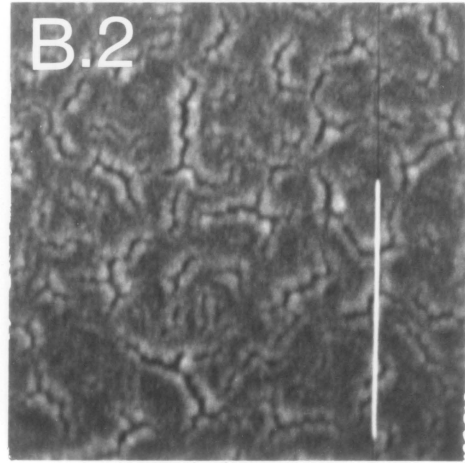
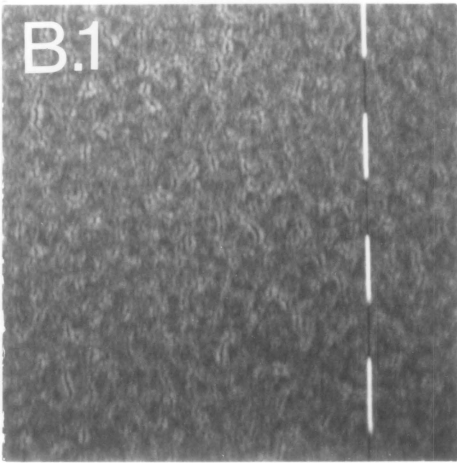
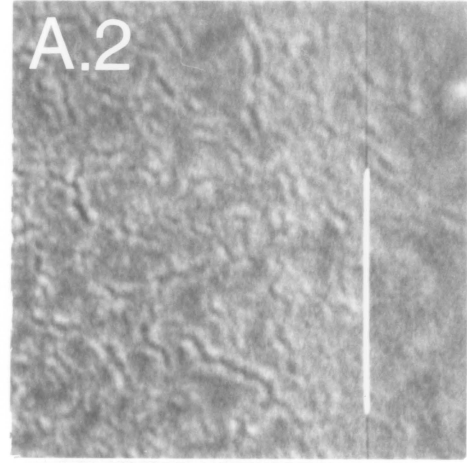
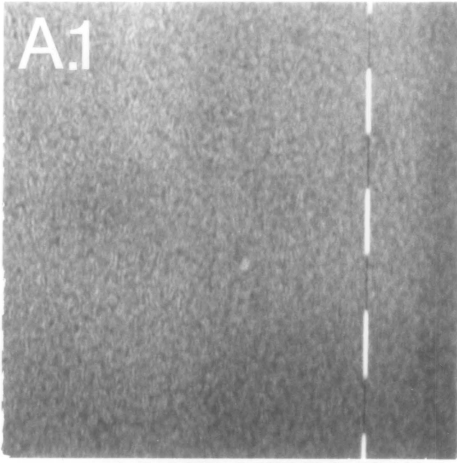
#### 4.1.7.2 High Resolution Scanning Electron Microscopy (HSEM) of the FT-120-WW surface.

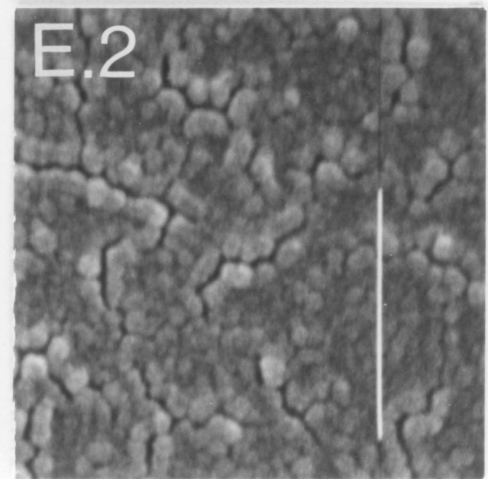
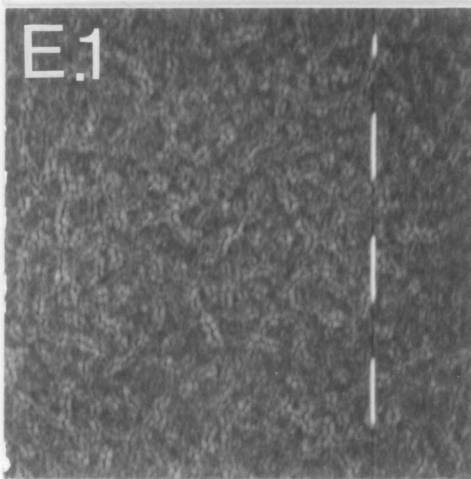
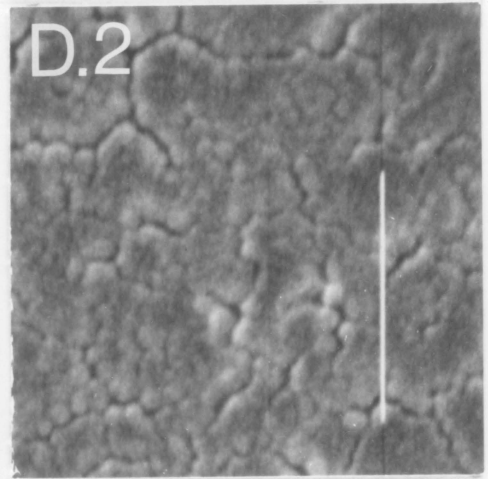
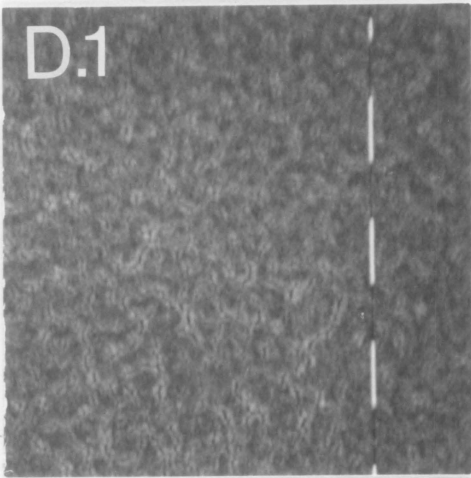
The resulting HSEM photomicrographs in Fig. 16 at 12,500X and 50,000X revealed that annealing, methanol wash and storage after annealing had no observable effect on the formation of surface crystallinity. This indicated that polypropylene crystallinity did not contribute to the surface selectivity of frying oils.

#### 4.1.7.3 Sessile drop application.

Fig. 16. HSEM photomicrograph of FT-120-WW.

- A. Untreated (control).
  - B. Methanol washed.
  - C. After annealing.
  - D. Four days after annealing.
  - E. Six days after annealing.
1. 12,500X. 2. 50,000X





Corn oil, heated for 14 days, exhibited less spreading over the FT-120-WW surface when compared with the unheated oil. When this experiment was repeated with a micro-syringe assembly through a needle (straight, 21 gauge, 0.02" internal diameter, Ramehart, Inc. Mountain Lake, NJ), the selectivity of the surface was reduced, thus indicating that the micropipet tip enhances selectivity while the microsyringe stainless steel needle retards it. The reason for this discrimination is probably due to the alignment of the nonpolar fraction on the inner surface of the polypropylene tips.

#### 4.1.7.4 Contact angle measurement.

The contact angles of the five shortenings were measured (Table 17) on the FT-120-WW step pellets, which were annealed four days prior to testing. Using the sessile drop method, the contact angles of the unheated shortenings were in the range of  $35.31^{\circ}$  to  $40.78^{\circ}$  (Table 17). In general CS/O and CS/OLS had higher initial contact angles when compared with C/O, P/O and SB/OLS. From the Analysis of Variance, no significant ( $P < 0.2474$ ) interaction was observed between contact angles and heating times, a fact which indicated that different shortenings had no different contact angles at different heating times. On the average, heating time had a significant ( $P < 0.0001$ )



## PLOT OF CONTACT ANGLE VS HEATING TIME

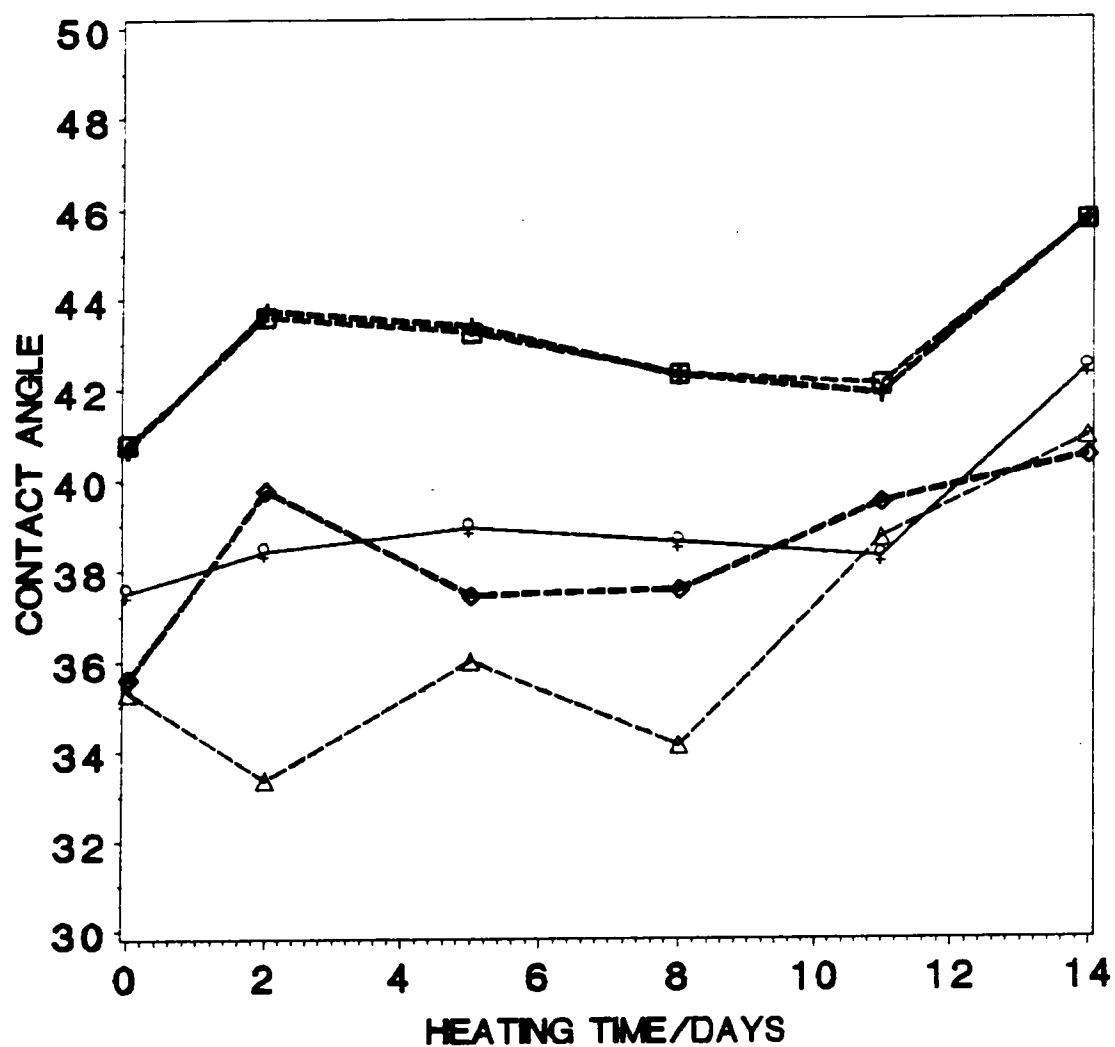


Fig. 17. Effect of static heating time on contact angles of corn (♀), cottonseed (□), peanut oils (Δ), cottonseed (+) and soybean (◇) oil liquid shortenings.

Table 17. Effect of static heating time of five shortening types on contact angle measurements as determined by the sessile drop method on the FT-120-WW step pellet surface.

| CONTACT ANGLE MEASUREMENTS <sup>1,5</sup> |                              |                   |                     |                  |                     |
|---|------------------------------|-------------------|---------------------|------------------|---------------------|
| HEATING TIME<br>(DAYS)                    | SHORTENING TYPE <sup>3</sup> |                   |                     |                  |                     |
|   | C/O <sup>4</sup>             | CS/O <sup>4</sup> | CS/OLS <sup>4</sup> | P/O <sup>4</sup> | SB/OLS <sup>4</sup> |
| 0   | 37.51<br>(0.69)              | 40.78<br>(1.09)   | 40.68<br>(0.84)     | 35.31<br>(2.58)  | 35.60<br>(0.56)     |
| 2   | 38.41<br>(0.49)              | 43.60<br>(0.50)   | 43.74<br>(0.56)     | 33.35<br>(1.43)  | 39.75<br>(0.66)     |
| 5   | 38.93<br>(0.73)              | 43.25<br>(0.46)   | 43.34<br>(0.41)     | 35.99<br>(1.57)  | 37.43<br>(2.98)     |
| 8   | 38.61<br>(0.99)              | 42.31<br>(1.39)   | 42.29<br>(1.20)     | 34.13<br>(2.48)  | 37.55<br>(1.63)     |
| 11  | 38.29<br>(0.64)              | 42.11<br>(1.90)   | 41.88<br>(1.96)     | 38.71<br>(2.04)  | 39.48<br>(2.57)     |
| 14  | 42.45<br>(1.67)              | 45.75<br>(1.51)   | 45.76<br>(1.54)     | 40.93<br>(2.03)  | 40.49<br>(0.29)     |
| $r^3$                                     | 0.74                         | 0.60              | 0.55                | 0.81             | 0.67                |

<sup>1</sup>: Average of two values from two plates.

<sup>2</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>3</sup>: Correlation coefficient of contact angles with heating time.

Table 17. cont.

- 
- <sup>4</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.
- <sup>5</sup>: Standard deviations are the values in brackets.

effect on changes in contact angle measurements. Moreover, changes in the contact angles were significantly ( $P < 0.0001$ ) different with different shortening types. After 14 days of static heating, the contact angles increased to a higher range of  $40.49^\circ$  and  $45.76^\circ$ . Probably, the accumulation of polymers or lipophylic molecules counteracted the polar or hydrophylic components in the shortening, causing a shifting in the hydrophylic lipophylic balance (HLB) of the media to a more lipophylic characteristic.

HLB probably is the single-most important factor controlling the surface free energy and has the greatest effect on contact angle measurement. The relationship between contact angle changes and heating time was not consistent for all shortenings. Peanut oil was the only shortening that had a high correlation coefficient ( $r = 0.81$ ) with heating time. The linear regression equation for peanut oil is  $y = 33.5 + x 0.44$ . Although the data points on the graph (Fig. 17) were significantly linear, with a slight curvature, the points varied erratically between 0 to 8 days of heating. Other shortenings, however, had shown increases to different extents in contact angles at early stages of heating, followed by decreases upon subsequent heating and finally, steady increases after 8 or 11 days of heating. The cause of this behavior is unclear. Also, how different lipid classes are aligned on the surface is also unknown.

From Fig. 17 and Table 17, the following can be concluded:

1. Contact angle changes between CS/O and CS/OLS are identical. This observation indicated that the liquid phase alone governs the behavior of cottonseed oil liquid shortening and probably any type of shortening. Moreover, the identical behavior of CS/O and CS/OLS might indicate that some important components that regulate the surface free energy might be found or produced in parallel concentrations in both shortenings. For example, both shortenings have similar triglyceride profiles at different heating periods.

2. Although significant ( $P < 0.0001$ ) increases in contact angles were observed in all five shortenings up to day 14; however, the significant linearity of peanut oil only can be used to predict its heat abuse.

3. The method probably can be used to explain some of the molecular changes that occur in a shortening during its chemical modification.

4. The method is very simple and requires five minutes to equilibrate the shortening with the FT-120-WW surface. Further improvements of the method might be necessary to allow its effective use in the food industry. The following factors affected the contact angle measurement:

a. Temperature of the shortening and the FT-120-WW pellets: the optimum temperature for measurement was  $28.5 \pm 0.5^{\circ}\text{C}$ . A higher or lower temperature might reduce the selectivity by changing the surface configuration.

b. Method of placing the shortening on the surface: virgin polypropylene pipetter tips showed improved selectivity when compared with a steel needle microsyringe.

c. Pellet maturation: after annealing, the surface configuration continued to change on a daily basis. This superficial change was accompanied by an improvement in selectivity time (defined as the time which the surface required to show a difference between fresh and used shortening after being placed on the surface). Days four and five had optimum surface maturity and shortest selectivity time, however, after day five the pellets lost their selectivity.

d. Selectivity time: 5 min. was the preferred time to read the contact angle measurement within a 10 minute interval.

e. Sessile drop volume:  $10 \mu\text{l}$  was the smallest volume tested with the Eppendorff pipetter. It is unknown whether a smaller volume would improve the selectivity.

f. Any physical abuse of the pellets may bring about surface damage and a loss in selectivity. Also, the surfaces should be protected from any source of

contamination from hands or air during storage, handling and contact angle testing.

g. Only a small and specific portions of the pellets (Fig. 14 in this chapter) were used in order to reduce the variation.

#### 4.1.8 HIGH TEMPERATURE GAS CHROMATOGRAPHIC ANALYSIS OF TRIGLYCERIDES.

An unmodified triglyceride sample was separated from the five oils and liquid shortenings as a non-polar fraction in the polar component test (Paquot, 1982).

##### 4.1.8.1 EFFECT OF STATIC HEATING TIME (DAYS) ON TRIGLYCERIDES (TGs) IN CORN OIL.

Fourteen types of triglycerides were separated from the nonpolar fraction of fresh and statically heated corn oil in less than 10 minutes (Table 18 and Fig. 18, see Appendix A for names). In general, the heating time had no significant ( $P < 0.999$ ) effect on changes in total triglyceride patterns under static heating conditions; however, some individual triglycerides did vary significantly. The triglycerides (TGs) found in the

Table 18. Effect of Static Heating Time (Days) on Triglyceride Concentration in Corn Oil.

| Triglyceride Type | Triglycerides (Peak Area) <sup>SD,A</sup> |                |                |                |                |                |                |        |       |
|-------------------|---|----------------|----------------|----------------|----------------|----------------|----------------|--------|-------|
|                   | t <sub>R</sub>                            | 0              | 2              | 5              | 8              | 11             | 14             | +D%    | S     |
| POP               | 4.47                                      | 3794<br>(74)   | 3700<br>(61)   | 4251<br>(65)   | 4268<br>(45)   | 5189<br>(3)    | 6314<br>(149)  | + 39.9 | 0.041 |
| PLP               | 4.68                                      | 6576<br>(105)  | 6355<br>(153)  | 6580<br>(62)   | 6799<br>(6)    | 7609<br>(34)   | 8134<br>(33)   | + 19.2 | 0.285 |
| POS               | 5.66                                      | 1017<br>(20)   | 1049<br>(93)   | 1394<br>(56)   | 1993<br>(15)   | 943<br>(65)    | 1759<br>(65)   | + 42.2 | 0.028 |
| POO               | 5.84                                      | 7352<br>(146)  | 10121<br>(408) | 11288<br>(245) | 11563<br>(71)  | 11949<br>(122) | 13367<br>(250) | + 45.0 | 0.299 |
| PLS               | 5.90                                      | 4219<br>(83)   | 1723<br>(98)   | ND             | ND             | ND             | ND             | -100.0 | -     |
| PLO               | 6.17                                      | 27815<br>(159) | 26443<br>(667) | 28339<br>(390) | 27352<br>(105) | 27221<br>(270) | 29453<br>(175) | + 5.6  | 0.963 |
| PLL               | 6.47                                      | 35712<br>(170) | 34102<br>(834) | 35151<br>(436) | 33445<br>(138) | 32087<br>(341) | 33567<br>(190) | - 6.0  | 0.954 |

cont.



Table 18. cont.

|       |      |                |                |                |                |                |                       |       |
|-------|------|----------------|----------------|----------------|----------------|----------------|-----------------------|-------|
| S00   | 7.26 | 1357<br>(27)   | 2773<br>(65)   | 2954<br>(74)   | 3631<br>(14)   | 2506<br>(74)   | 2497 + 45.7<br>(18)   | 0.086 |
| 000   | 7.60 | 8748<br>(79)   | 10814<br>(187) | 11738<br>(71)  | 12308<br>(62)  | 11017<br>(44)  | 12338 + 29.1<br>(73)  | 0.075 |
| SLO   | 7.73 | 5531<br>(49)   | 5518<br>(24)   | 5438<br>(16)   | 5461<br>(14)   | 5554<br>(21)   | 5279 - 4.6<br>(27)    | 0.528 |
| OLO   | 7.98 | 35026<br>(99)  | 34353<br>(705) | 31272<br>(668) | 34324<br>(164) | 34111<br>(111) | 35172 + 0.4<br>(103)  | 0.946 |
| OLL   | 8.42 | 52622<br>(105) | 50154<br>(919) | 51055<br>(749) | 46933<br>(206) | 47486<br>(267) | 47109 - 10.5<br>(193) | 0.913 |
| LLL   | 8.81 | 46363<br>(93)  | 44632<br>(995) | 43875<br>(480) | 41579<br>(184) | 38339<br>(161) | 37055 - 20.1<br>(307) | 0.538 |
| LLLn  | 9.22 | 4205<br>(15)   | 4477<br>(191)  | 3963<br>(87)   | 4834<br>(104)  | 4693<br>(59)   | 3044 - 27.6<br>(150)  | 0.579 |
| Total |      | 236132         | 237977         | 244463         | 244683         | 232042         | 236300                | 0.993 |

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
 L: linoleic acid (C<sub>18</sub>:2) and Ln: linolenic acid (C<sub>18</sub>:3).

ND: Not Detected, assumed = zero.

S: significance.

Table 18. cont.

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$t_R$ : Retention time in minutes.

SD: standard deviations are the values in brackets.

$\pm D\%$ : percent increase or decrease in peak area after 14 days of static heating.

A: average of two replicates.

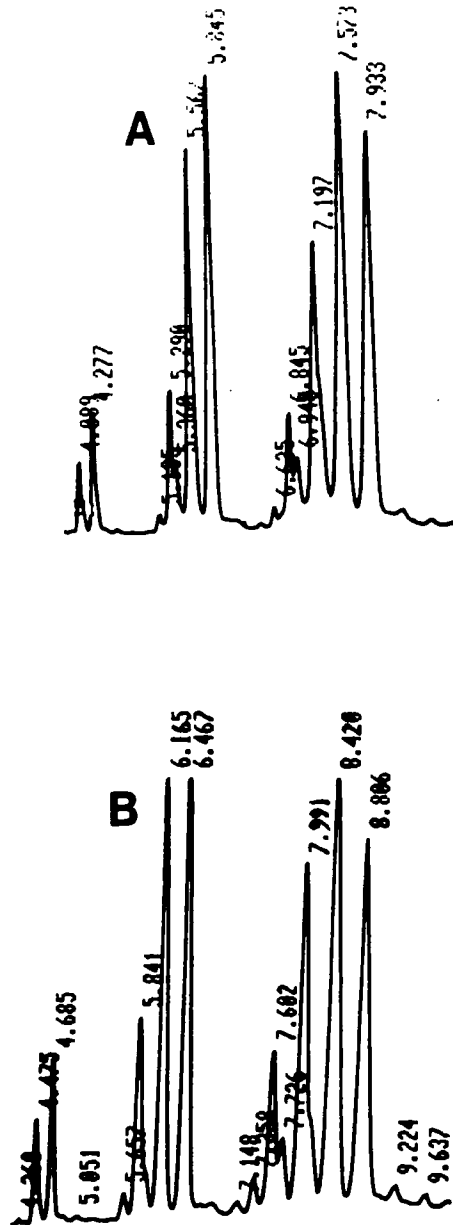


Fig. 18. Effect of static heating time (Days) on triglyceride profile of corn oil. A-unheated; B-heated to 14 days.

greatest concentrations were PLO, PLL, OLO, OLL and LLL (Table 18). The effect of heating time on changes in triglyceride (peak area) concentration varied with the type of triglyceride (Fig. 18). For example, POP, PLP, POS, POO, PLO, SOO and OOO had a relative tendency to accumulate until the end of the fourteen-day heating period in the range of 5.6 to 45.7%. Their accumulation indicated their relative resistance as a substrate for oxidation since they have only one to three double bonds. On the other hand, PLS, SLO, PLL, OLL, LLL and LLLn had a relative tendency to decrease in concentration by the end of the fourteen day heating period in the range 4.6 to 100%. Using the General Linear Models (GLM) procedure, POP and POS were found to be the only triglycerides that accumulated significantly ( $P < 0.041$  and  $P < 0.028$ ) with heating time. The peak areas of the total number of triglycerides did not change significantly ( $P < 0.993$ ) throughout the heating process.

In general, triglycerides with 1-3 double bonds tended to accumulate, while others with 2-7 double bonds tended to decrease in concentration with prolonged heating periods. Another unique observation obtained from this test was that the rate of accumulation of POP and PLP was higher than that of POS and PLS. This fact might indicate that in corn oil, shorter chain fatty acids such as palmitate, C16:0, may provide more stability to unsaturated fatty acids on

sn-2 of TG than longer chain fatty acids, such as stearate, C18:0.

#### 4.1.8.2 EFFECT OF STATIC HEATING TIME (DAYS) ON TRIGLYCERIDES (TGs) IN COTTONSEED OIL.

Twenty types of triglycerides were separated from the nonpolar fraction of fresh and heated cottonseed oil in less than 9 minutes (Table 19, and Fig. 10 in Appendix F). On the whole, heating time had no significant ( $P < 0.9999$ ) effect on changes in triglyceride profile under static heating conditions. Among the detected triglycerides, PLP, PLO, PLL, OLL and LLL represent triglycerides with high concentrations. The effect of heating time on changes in triglyceride (peak area) concentration varied with the specific triglyceride.

In general, the changes in triglyceride concentration were more dynamic with cottonseed oil than with corn oil. Some (TGs) such as POP, PLP, MLO+PLP<sub>o</sub>, POO, PLO, and PLL increased, in the range of 8.8 to 42.9%, by the end of the heating period; however, MOP, MLL, POS, PLS, SOS, SOO, SLO, OOO, SLL, OLL, and LLL were reduced in concentration in the range of 12.0 to 88.4%. The presence of OLL and LLL in high concentrations could be detrimental to shortening quality because of their low oxidative stabilities.

Table 19. Effect of Static Heating Time (Days) on Triglyceride Concentration in Cottonseed Oil.

| Triglyceride Type | Triglycerides (Peak Area) <sup>SD,A</sup> |                |                |                |                |                |                |             |
|-------------------|---|----------------|----------------|----------------|----------------|----------------|----------------|-------------|
|                   | t <sub>R</sub>                            | 0              | 2              | 5              | 8              | 11             | 14             | +D% S       |
| MOP               | 3.47                                      | 9023<br>(60)   | 5013<br>(56)   | 9783<br>(44)   | 1266<br>(12)   | 1095<br>(18)   | 1051<br>(21)   | -88.4 0.033 |
| MLP               | 3.63                                      | 2103<br>(34)   | 1743<br>(190)  | 1419<br>(25)   | 1702<br>(45)   | 2258<br>(20)   | 2085<br>(92)   | - 0.9 0.078 |
| POP               | 4.38                                      | 5521<br>(79)   | 5691<br>(24)   | 5812<br>(219)  | 6596<br>(57)   | 6626<br>(72)   | 7161<br>(45)   | +29.7 0.091 |
| PLP               | 4.61                                      | 14869<br>(203) | 14912<br>(512) | 15832<br>(185) | 17959<br>(992) | 19806<br>(830) | 19997<br>(161) | +34.5 0.011 |
| MLO+PLPo          | 4.75                                      | 3036<br>(70)   | 2921<br>(91)   | 2458<br>(38)   | 2415<br>(61)   | 3100<br>(49)   | 3307<br>(66)   | + 8.9 0.377 |
| MLL               | 4.95                                      | 3093<br>(50)   | 3068<br>(27)   | 1936<br>(57)   | 2199<br>(21)   | 3061<br>(69)   | 2117<br>(72)   | -31.6 0.155 |
| POS               | 5.54                                      | 2951<br>(12)   | 2565<br>(124)  | 2114<br>(100)  | 2092<br>(51)   | 2682<br>(108)  | 2077<br>(87)   | -29.6 0.644 |

cont.

Table 19. cont.

|      |      |                |                |                |                |                |                      |       |
|------|------|----------------|----------------|----------------|----------------|----------------|----------------------|-------|
| POO  | 5.71 | 6180<br>(32)   | 7432<br>(27)   | 6980<br>(15)   | 6296<br>(42)   | 6360<br>(28)   | 8837 +42.9<br>(76)   | 0.005 |
| PLS  | 5.81 | 5007<br>(29)   | 4428<br>(18)   | 4636<br>(125)  | 6096<br>(45)   | 6139<br>(73)   | 4362 -12.9<br>(49)   | 0.028 |
| PLO  | 6.04 | 27020<br>(224) | 27523<br>(73)  | 27240<br>(162) | 30573<br>(151) | 31784<br>(202) | 32389 +19.9<br>(149) | 0.028 |
| PLL  | 6.35 | 42894<br>(239) | 43106<br>(150) | 43114<br>(144) | 45115<br>(121) | 47355<br>(267) | 46656 + 8.8<br>(277) | 0.179 |
| PoLL | 6.51 | 3555<br>(72)   | 4399<br>(117)  | 4071<br>(95)   | 2880<br>(175)  | 2670<br>(177)  | 3537 - 0.5<br>(34)   | 0.842 |
| SOS  | 6.88 | 1440<br>(29)   | 2284<br>(68)   | ND<br>-        | ND<br>-        | ND<br>-        | ND -100<br>-         | 0.037 |
| S00  | 7.18 | 2424<br>(30)   | 2129<br>(78)   | 2146<br>(21)   | 1363<br>(52)   | 1081<br>(76)   | 1545 -36.3<br>(67)   | 0.037 |
| 000  | 7.39 | 4701<br>(21)   | 4418<br>(120)  | 4574<br>(31)   | 3394<br>(49)   | 3184<br>(58)   | 3755 -20.1<br>(79)   | 0.012 |
| SLO  | 7.52 | 4833<br>(20)   | 4233<br>(112)  | 4464<br>(45)   | 3667<br>(66)   | 3174<br>(56)   | 3871 -19.9<br>(86)   | 0.013 |
| OLO  | 7.77 | 9577<br>(22)   | 9687<br>(14)   | 9631<br>(34)   | 9611<br>(45)   | 9499<br>(59)   | 9573 0.0<br>(81)     | 0.625 |

cont.

Table 19. cont.

|       |      |                |                |                |                |                |                      |        |
|-------|------|----------------|----------------|----------------|----------------|----------------|----------------------|--------|
| SLL   | 7.89 | 6928<br>(20)   | 5595<br>(87)   | 5347<br>(46)   | 4770<br>(91)   | 4499<br>(10)   | 5195 -25.0<br>(67)   | 0.006  |
| OLL   | 8.17 | 25937<br>(22)  | 23233<br>(124) | 24717<br>(116) | 23267<br>(124) | 22118<br>(50)  | 22049 -14.9<br>(244) | 0.007  |
| LLL   | 8.56 | 29861<br>(161) | 26030<br>(104) | 28413<br>(132) | 23851<br>(147) | 22745<br>(112) | 21968 -26.4<br>(207) | 0.0001 |
| Total |      | 202931         | 197475         | 193836         | 202440         | 212048         | 201304               | 0.094  |

M: myristic acid (C<sub>14</sub>:0), P: palmitic acid (C<sub>16</sub>:0), Po: palmitoleic acid (C<sub>16</sub>:1), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1) and L: linoleic acid (C<sub>18</sub>:2).

ND: not detected, assumed = zero.

S: significance.

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 14 days of static heating.

A: average of two replicates.



Using the General Linear Model procedure (GLM), it was found that PLP, POO and PLO were the only TGs that accumulated significantly ( $P < 0.011$ ,  $0.005$  and  $0.028$ , respectively) under these heating conditions. However, MOP, PLS, SOS, SOO, OOO, SLO, SLL, OLL, and LLL were the TGs which were reduced significantly ( $P < 0.033$ ,  $0.028$ ,  $0.037$ ,  $0.037$ ,  $0.012$ ,  $0.013$ ,  $0.006$ ,  $0.007$  and  $0.0001$ , respectively). The peak areas of the total number of triglycerides did not change significantly ( $P < 0.094$ ) throughout the static heating period.

In general, no relationship was established between the number of double bonds and the rate of oxidation because MOP, MLP, MLL, POS, PLS, SOS, SOO, OOO, SLO, SLL, OLL and LLL decreased in concentration after fourteen days of heating even when the unsaturated fatty acids were in the second position, however, PLO and PLL increased, on the other hand. Therefore, triglycerides with 1 to 4 double bonds showed accumulation, while others with 1 to 6 double bonds showed reduction in their concentration. Hence, this result is contradictory to earlier published reports (Raughveer and Hammond, 1967; Yoshida and Alexander, 1984). Moreover, Thompson et al. (1967) indicated that the degree of shortening deterioration was independent of their degree of saturation but rather dependent upon how they were used. However, since corn oil and cottonseed oil were heated under the same conditions, the condition of use

was not necessarily a factor. The triglyceride pattern (quality and quantity) of each oil should be used as a tool to predict the rate of deterioration. Furthermore, the reduction in SLO and SLL which was associated with the increase in PLO and PLL indicated that palmitic acid on sn-1 or sn-3 might provide better protection to oleic and linoleic acids against oxidation than stearic acid in the same triglyceride molecule.

#### 4.1.8.3 EFFECT OF STATIC HEATING TIME ON TRIGLYCERIDES (TGs) IN COTTONSEED OIL LIQUID SHORTENING (CS/OLS).

Twenty three types of triglycerides were separated and detected from the nonpolar fraction of unheated and heated cottonseed oil liquid shortening in less than 9 minutes (Table 20, and Fig. 11 in Appendix F). In general, heating time had no significant ( $P < 0.9999$ ) effect on changes in the TG profile. PLP, PLO, PLL, OLL and LLL with peak areas of 26374, 31129, 54578, 24116, and 29249, respectively, represent the major TGs in CS/OLS, of which OLL and LLL are probably more detrimental to quality due to their low oxidation stability and their high concentrations. The effect of static heating time on the finger print of CS/OLS triglycerides depends on the carbon number and degree of

Table 20. Effect of Static Heating Time (Days) on Triglyceride Concentration in Cottonseed Oil Liquid Shortening.

| Triglyceride Type | Triglycerides (Peak Area) SD,A |                |                |                |                |                |               |       |       |
|-------------------|--------------------------------|----------------|----------------|----------------|----------------|----------------|---------------|-------|-------|
|                   | t <sub>R</sub>                 | 0              | 2              | 5              | 8              | 11             | 14            | +D%   | S     |
| PPP               | 3.31                           | 808<br>(14)    | 769<br>(10)    | 816<br>(55)    | 751<br>(5)     | 1014<br>(79)   | 976<br>(5)    | +20.8 | 0.037 |
| MOP               | 3.46                           | 1564<br>(34)   | 1206<br>(22)   | 1291<br>(33)   | 1093<br>(21)   | 1610<br>(22)   | 1442<br>(11)  | - 7.8 | 0.244 |
| MLP               | 3.63                           | 2114<br>(47)   | 1882<br>(10)   | 2268<br>(14)   | 2361<br>(47)   | 2191<br>(32)   | 2274<br>(55)  | + 7.6 | 0.386 |
| PPS               | 4.23                           | 1269<br>(31)   | 1438<br>(5)    | 1744<br>(24)   | 1704<br>(7)    | 2028<br>(52)   | 2030<br>(30)  | +59.9 | 0.200 |
| POP               | 4.38                           | 8817<br>(15)   | 8228<br>(70)   | 10270<br>(129) | 11530<br>(146) | 11738<br>(125) | 11989<br>(67) | +35.9 | 0.076 |
| PLP               | 4.61                           | 26374<br>(153) | 24271<br>(149) | 29439<br>(328) | 29144<br>(115) | 27576<br>(152) | 29566<br>(89) | +12.1 | 0.166 |
| MLO+PLPo          | 4.75                           | 2627<br>(73)   | 2505<br>(12)   | 3211<br>(11)   | 2970<br>(100)  | 2956<br>(33)   | 2935<br>(48)  | +11.7 | 0.266 |

cont.

Table 20. cont.

|          |      |                |                |                |                |                |                      |       |
|----------|------|----------------|----------------|----------------|----------------|----------------|----------------------|-------|
| MLL      | 4.95 | 1931<br>(18)   | 2064<br>(14)   | 2678<br>(17)   | 2649<br>(46)   | 2328<br>(30)   | 2097 + 8.6<br>(70)   | 0.417 |
| UNKNOWN1 | 5.24 | 1998<br>(64)   | 2113<br>(41)   | 2886<br>(28)   | 2835<br>(36)   | 3542<br>(49)   | 3569 +78.6<br>(71)   | 0.058 |
| POS      | 5.54 | 2048<br>(32)   | 2042<br>(18)   | 2576<br>(19)   | 2597<br>(51)   | 3089<br>(52)   | 2970 +45.0<br>(59)   | 0.131 |
| POO      | 5.71 | 6381<br>(95)   | 6455<br>(19)   | 8077<br>(32)   | 8999<br>(127)  | 9121<br>(14)   | 8946 +40.2<br>(84)   | 0.007 |
| PLS      | 5.81 | 5611<br>(63)   | 4984<br>(63)   | 5317<br>(105)  | 4311<br>(25)   | 4956<br>(15)   | 6625 +18.0<br>(58)   | 0.147 |
| PLO      | 6.04 | 31129<br>(416) | 28315<br>(212) | 33815<br>(320) | 33115<br>(143) | 31997<br>(151) | 33411 + 7.3<br>(79)  | 0.232 |
| PLL      | 6.35 | 54578<br>(568) | 47592<br>(340) | 52572<br>(247) | 50476<br>(91)  | 47107<br>(286) | 46611 -14.6<br>(381) | 0.168 |
| PoLL     | 6.52 | 2366<br>(25)   | 2275<br>(67)   | 2840<br>(82)   | 3190<br>(45)   | 2749<br>(79)   | 2441 + 3.1<br>(47)   | 0.647 |
| UNKNOWN2 | 6.98 | 2426<br>(57)   | 2716<br>(55)   | 3335<br>(36)   | 3353<br>(16)   | 3953<br>(58)   | 3948 +62.7<br>(72)   | 0.110 |
| SOO      | 7.18 | 829<br>(41)    | 1211<br>(65)   | 1307<br>(68)   | 1283<br>(30)   | 2039<br>(58)   | 1840 +122<br>(52)    | 0.037 |

cont.

Table 20. cont.

|       |      |                |               |                |               |                |                      |       |
|-------|------|----------------|---------------|----------------|---------------|----------------|----------------------|-------|
| 000   | 7.39 | 2564<br>(68)   | 31157<br>(85) | 3407<br>(48)   | 3583<br>(57)  | 4678<br>(58)   | 4504 +75.7<br>(73)   | 0.158 |
| SLO   | 7.52 | 3049<br>(17)   | 3444<br>(69)  | 3696<br>(37)   | 3651<br>(40)  | 4577<br>(79)   | 4409 +44.6<br>(57)   | 0.251 |
| OLO   | 7.77 | 8039<br>(94)   | 7985<br>(40)  | 9185<br>(48)   | 7246<br>(13)  | 9667<br>(51)   | 9521 +18.4<br>(34)   | 0.236 |
| SLL   | 7.89 | 5145<br>(95)   | 5165<br>(41)  | 5326<br>(29)   | 5181<br>(21)  | 6169<br>(4)    | 5798 +12.7<br>(12)   | 0.368 |
| OLL   | 8.17 | 24116<br>(182) | 22370<br>(32) | 23556<br>(99)  | 23086<br>(26) | 23329<br>(83)  | 22089 - 8.4<br>(254) | 0.866 |
| LLL   | 8.56 | 29249<br>192)  | 26463<br>(10) | 25729<br>(131) | 24617<br>(70) | 24480<br>(134) | 22038 -24.7<br>(232) | 0.260 |
| Total |      | 225004         | 208639        | 232429         | 233683        | 230838         | 234024               | 0.296 |

M: myristic acid (C<sub>14</sub>:0), P: palmitic acid (C<sub>16</sub>:0), Po: palmitoleic acid (C<sub>16</sub>:1), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1) and L: linoleic acid (C<sub>18</sub>:2).

S: significance.

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

Table 20. cont.

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$\pm$ D%: percent increase or decrease in peak area after 14 days of static heating.

A: average of two replicates.

unsaturation. Almost half the number of TGs followed a steady increase or decrease in their concentrations while the other half were more dynamic and followed a random order variation similar to the CS/O study. Some peaks such as PPP, MLP, PPS, POP, PLP, MLO+PLPo, MLL, UNKNOWN<sub>1</sub>, POS, POO, PLS, PLO, P<sub>o</sub>LL, UNKNOWN<sub>2</sub>, SOO, OOO, SLO, OLO, and SLL had higher concentrations by the end of the heating period and this increase was in the range of 3.7 to 122.0%.

From the General Linear Models procedure (GLM), PPP, POO and SOO were the only triglycerides that showed a significant ( $P < 0.037$ ,  $0.007$  and  $0.037$ , respectively) change (accumulation) with heating time. The total peak area of all triglycerides did not change significantly ( $P < 0.246$ ) throughout the heating period.

Other triglycerides such as MOP, PLL, OLL, and LLL appeared to decrease in their concentration, in the range of 7.8 to 24.7%, as a result of the heating process. However, their decrease in peak areas was not significant ( $P < 0.244$ ,  $0.168$ ,  $0.866$ , and  $0.260$ , respectively). Three of the TGs with four double bonds (MLL, OLO, and SLL) had increased peak areas from 1931, 8039 and 5145 to 2097, 9521 and 5798, respectively. Nevertheless, the PLL peak area decreased from 54578 to 46611 even though it had four double bonds, because it was reported that triglycerides with four double bonds have a relative tendency to resist oxidation (Yoshida and Alexander, 1984).

Therefore, some triglycerides with 0 to 4 double bonds manifest stability, while others with 1 and 4 to 6 double bonds showed low stability.

From the viscosity measurement study section 4.1.5, it was observed that CS/OLS had exceptionally high viscosity, although its triglyceride profile was relatively similar to that of CS/O's. The only difference was that CS/O did not include PPP, PPS, UNKNOWN<sub>1</sub> and UNKNOWN<sub>2</sub> in its triglyceride profile as compared to CS/OLS's. Otherwise, the reason for the tremendous increase in CS/OLS viscosity is yet unknown. Therefore, the question is "whether fat crystals participate in the polymerization process?"

#### 4.1.8.4 EFFECT OF STATIC HEATING TIME ON TRIGLYCERIDES (TGs) IN PEANUT OIL.

Twenty seven peaks were separated from fresh and heated peanut oil in less than 15 minutes (Table 21 and Fig. 19).

In general, the heating time had no significant ( $P < 0.9999$ ) effect on the changes in the triglyceride profile. Among the more concentrated TGs were POO, PLO, PLL, OOO + SLS, SLL + OLO, and OLL. Their peak areas were 19921, 27284, 10693, 32516, 45191, and 28615, respectively. Several peaks increased in area by the end of the fourteen



Table 21. Effect of Static Heating Time (Days) on Triglyceride Concentration in Peanut Oil.

| Triglyceride Type | Triglycerides (Peak Area) <sup>SD,A</sup> |                |                |                |                |                |                     |        |
|-------------------|---|----------------|----------------|----------------|----------------|----------------|---------------------|--------|
|                   | t <sub>R</sub>                            | 0              | 2              | 5              | 8              | 11             | 14 ±D%              | S      |
| POP               | 4.18                                      | 3734<br>(71)   | 4087<br>(35)   | 4200<br>(14)   | 4525<br>(21)   | 5250<br>(49)   | 4634 +24.1<br>(52)  | 0.020  |
| PLP               | 4.38                                      | 4888<br>(74)   | 5015<br>(49)   | 4741<br>(32)   | 5179<br>(49)   | 5392<br>(134)  | 4458 - 8.8<br>(36)  | 0.099  |
| POS               | 5.30                                      | 1540<br>(22)   | 1438<br>(74)   | 1845<br>(112)  | 1531<br>(40)   | 1928<br>(24)   | 2117 +37.5<br>(73)  | 0.027  |
| POO               | 5.51                                      | 19921<br>(250) | 22730<br>(587) | 24968<br>(97)  | 26931<br>(898) | 28153<br>(222) | 28274 +41.9<br>(72) | 0.0001 |
| PLO               | 5.79                                      | 27284<br>(861) | 27203<br>(140) | 27575<br>(247) | 27948<br>(115) | 28321<br>(345) | 26643 - 2.4<br>(51) | 0.384  |
| PLL               | 5.99                                      | 10693<br>(169) | 9298<br>(41)   | 9000<br>(82)   | 8073<br>(76)   | 8196<br>(36)   | 7805 -27.0<br>(104) | 0.045  |
| POA+SOS           | 6.71                                      | 1200<br>(9)    | 1166<br>(22)   | 1561<br>(68)   | 997<br>(62)    | 1010<br>(50)   | 1882 +56.8<br>(55)  | 0.274  |

cont.

Table 21. cont.

|           |       |               |               |               |               |              |                          |
|-----------|-------|---------------|---------------|---------------|---------------|--------------|--------------------------|
| OLGa      | 9.30  | 2813<br>(58)  | 2563<br>(299) | 2375<br>(77)  | 2359<br>(48)  | 2591<br>(18) | 2332 -17.1<br>(97) 0.137 |
| GalL      | 9.71  | 1648<br>(12)  | 2186<br>(173) | 1910<br>(75)  | 1605<br>(108) | 2315<br>(55) | 2073 +25.8<br>(93) 0.729 |
| POLg+SOBe | 10.48 | 1356<br>(65)  | 1238<br>(6)   | 1222<br>(6)   | 1340<br>(36)  | 1459<br>(91) | 1288 - 5.0<br>(46) 0.019 |
| BeOO      | 10.77 | 3980<br>(27)  | 4185<br>(119) | 2172<br>(124) | 4517<br>(31)  | 4642<br>(19) | 4625 +16.2<br>(64) 0.060 |
| PLLg      | 10.95 | 1289<br>(8)   | 1348<br>(249) | 1198<br>(11)  | 1363<br>(186) | 1426<br>(79) | 1327 + 2.9<br>(46) 0.721 |
| BeLO      | 11.25 | 5254<br>(39)  | 5163<br>(407) | 4756<br>(75)  | 4915<br>(303) | 4884<br>(28) | 4759 - 9.4<br>(65) 0.453 |
| BeLL      | 11.72 | 3182<br>(53)  | 2927<br>(300) | 2442<br>(32)  | 2482<br>(36)  | 2362<br>(28) | 1853 -41.8<br>(51) 0.001 |
| LgOO      | 13.24 | 3316<br>(59)  | 2388<br>(84)  | 2234<br>(79)  | 2364<br>(20)  | 1994<br>(88) | 2292 -30.9<br>(57) 0.178 |
| LgLO      | 13.79 | 5433<br>(132) | 3240<br>(189) | 3468<br>(39)  | 3223<br>(81)  | 1650<br>(93) | 4511 -16.9<br>(56) 0.023 |
| LgLL      | 14.37 | 4019<br>(139) | 2664<br>(24)  | 883<br>(48)   | 2279<br>(55)  | 759<br>(65)  | 3628 - 9.7<br>(33) 0.011 |

cont.

Table 21. cont.

|           |      |                |                |                |                |                |                     |        |
|-----------|------|----------------|----------------|----------------|----------------|----------------|---------------------|--------|
| S00       | 6.92 | 5092<br>(89)   | 5292<br>(14)   | 6426<br>(6)    | 5251<br>(119)  | 6201<br>(75)   | 7134 +40.1<br>(40)  | 0.108  |
| 000+SLS   | 7.23 | 32516<br>(256) | 35227<br>(106) | 38194<br>(84)  | 41570<br>(236) | 42516<br>(146) | 41940 +28.9<br>(91) | 0.0003 |
| SLO       | 7.40 | 6296<br>(83)   | 6731<br>(129)  | 6847<br>(4)    | 5893<br>(119)  | 6032<br>(34)   | 5906 - 6.2<br>(170) | 0.024  |
| SLL+OLO   | 7.58 | 45191<br>(259) | 46675<br>(197) | 45459<br>(115) | 45266<br>(196) | 45343<br>(230) | 41297 - 8.2<br>(58) | 0.079  |
| OLL       | 7.87 | 28615<br>(399) | 27994<br>(110) | 25100<br>(876) | 23374<br>(46)  | 22726<br>(133) | 19579 -31.6<br>(78) | 0.001  |
| LLL       | 8.15 | 5206<br>(33)   | 4876<br>(26)   | 3877<br>(142)  | 3378<br>(70)   | 3016<br>(160)  | 2715 -47.9<br>(17)  | 0.0001 |
| POBe      | 8.43 | 2005<br>(51)   | 2207<br>(5)    | 2176<br>(147)  | 2304<br>(50)   | 2320<br>(16)   | 2557 +27.5<br>(82)  | 0.034  |
| AOO       | 8.76 | 2069<br>(97)   | 2269<br>(42)   | 2507<br>(46)   | 2697<br>(86)   | 2810<br>(132)  | 2793 +34.9<br>(64)  | 0.003  |
| PLBe+Ga00 | 8.91 | 2019<br>(15)   | 2130<br>(70)   | 2020<br>(48)   | 2202<br>(124)  | 2073<br>(184)  | 2025 0.0<br>(65)    | 0.502  |
| ALO       | 9.10 | 2478<br>(92)   | 2330<br>(217)  | 2338<br>(57)   | 2222<br>(205)  | 2255<br>(75)   | 2093 -15.5<br>(57)  | 0.838  |

cont.

Table 21. cont.

|       |        |        |        |        |        |        |       |
|-------|--------|--------|--------|--------|--------|--------|-------|
| Total | 235002 | 234995 | 233230 | 236374 | 237932 | 235181 | 0.838 |
|-------|--------|--------|--------|--------|--------|--------|-------|

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
 L: linoleic acid (C<sub>18</sub>:2), A: arachidic acid (C<sub>20</sub>:0), Ga: gadoleic acid  
 (C<sub>20</sub>:1), Be: behenic acid (C<sub>22</sub>:0) and Lg: lignoceric acid (C<sub>24</sub>:0).

S: significance of day.

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 14 days of static heating.

A: average of two replicates.

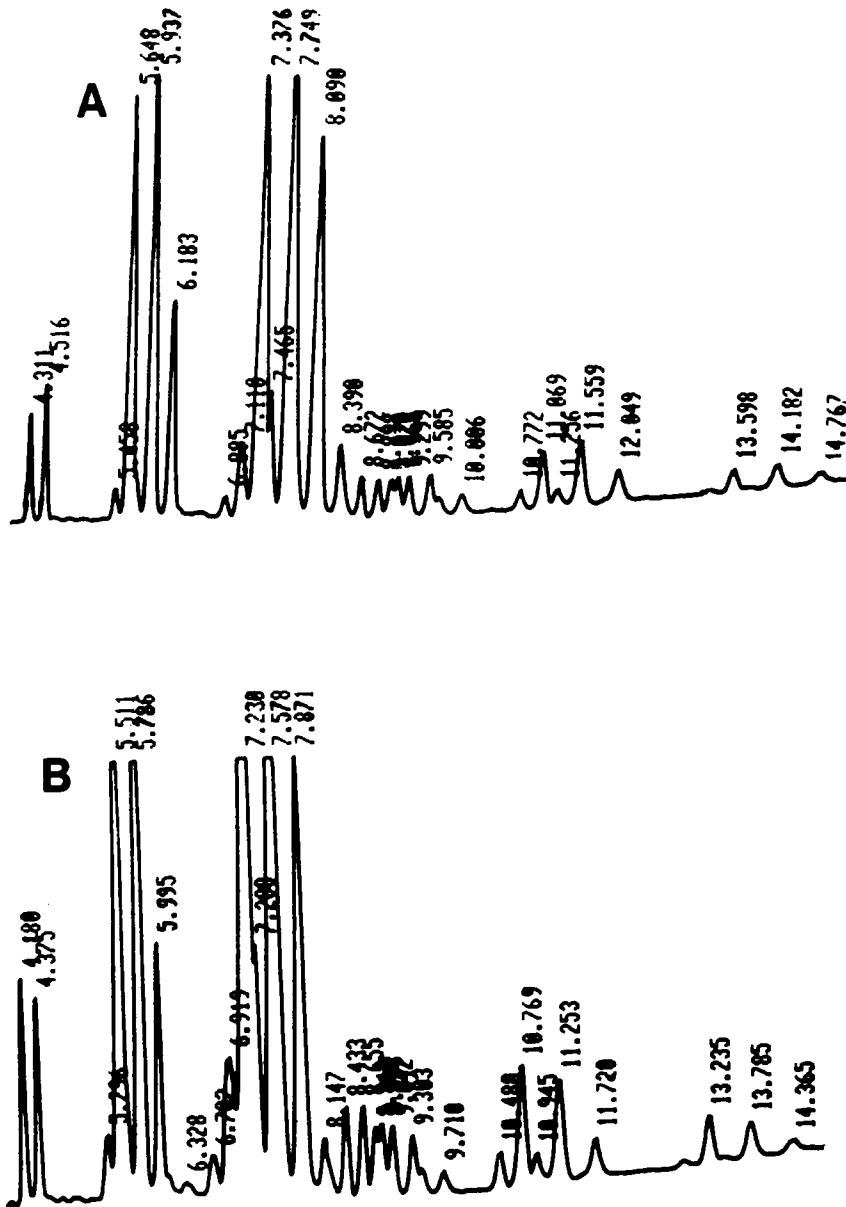


Fig. 19. Effect of static heating time (Days) on triglyceride profile of peanut oil. A-unheated; B-heated to 14 days.

day heating period. These peaks were POP, POS, POO, POA + SOS, SOO, OOO + SLS, POBe, AOO, GaLL, BeOO and PLLg. The relative increase in peak area was in the range of 2.9 to 56.8%. However, PLP, PLO, PLL, SLO, SLL + OLO, OLL, LLL, ALO, OLGa, POLg+SOBe, BeLO, BeLL, LgOO, LgLO, and LgLL had a reduction in peak area in the range of 2.4 to 47.9%. Of the major peaks, POO, and OOO + SLS increased in peak area by the end of the fourteen day heating period from 19921 and 32516 to 28274 and 41940, respectively.

From the General Linear Models (GLM) procedure, it was found that POP, POS, POO, OOO + SLS, POBe, AOO, increased significantly ( $P < 0.020$ ,  $0.027$ ,  $0.0001$ ,  $0.0003$ ,  $0.034$ , and  $0.003$ , respectively), while PLL, SLO, OLL, LLL, BeLL, LgLO, and LgLL were significantly reduced in peak areas ( $P < 0.045$ ,  $0.024$ ,  $0.001$ ,  $0.0001$ ,  $0.001$ ,  $0.023$ , and  $0.011$ , respectively).

In summary, it is observed that TGs with 1 to 5 double bonds showed accumulation, while other TG with 2 to 6 double bonds showed reduction. TGs with four double bonds PLL and SLL + OLO were prone to oxidation, even though they were expected to show relative resistance when compared to OLL, which has five double bonds (Yoshida and Alexander 1984). Only the LLL, triglycerides with 6 double bonds was unstable under the specified conditions. Also short chain saturated fatty acids provide better protection to unsaturated fatty acids in the triglycerides from the

saturated long chain fatty acid in peanut oil. Therefore, the oxidation rate of P/O was controlled by the interaction between all the triglycerides under static heating conditions.

#### 4.1.8.5 EFFECT OF STATIC HEATING TIME ON TRIGLYCERIDES (TGs) IN SOYBEAN OIL LIQUID SHORTENING (SB/OLS).

Seventeen peaks of triglycerides were separated from fresh and heated soybean oil liquid shortening in less than 10 minutes (Table 22, and Fig. 12 in Appendix F).

In general, the heating time had no significant ( $P < 0.9999$ ) effect on the changes in triglyceride profile under static heating conditions. The major peaks were POO, PLO, PLL, OOO, SLO, OOL, SLL, OLL, and LLL. At the end of a fourteen day heating period, several peaks increased in size, indicating their ability to accumulate and resist oxidation. These TGs were POP, PLP, UNKNOWN, POS, POO, PLS, PLLn, SOS, SOO, OOO, SLO, and SLL. Peak areas increased in the range of 9.4 to 54.4%. On the other hand, some TG peaks were decreased (PLO, PLL, OOL, OLL, and LLL). The reduced peak areas were in the range of 6.8 to 35.1%. With the General Linear Models (GLM) procedure, it was found that PLP, POS, POO, SOO, OOO, and SLL and their peak areas increased significantly ( $P < 0.040$ ,  $0.010$ ,  $0.001$ ,

Table 22. Effect of Static Heating Time (Days) on Triglyceride Concentration in Soybean Oil Liquid Shortening.

| Triglyceride Type | Triglycerides (Peak Area) SD; A |                |                |                |                |                |               |                |
|-------------------|---------------------------------|----------------|----------------|----------------|----------------|----------------|---------------|----------------|
|                   | t <sub>R</sub>                  | 0              | 2              | 5              | 8              | 11             | 14            | +D% S          |
| POP               | 4.67                            | 6599<br>(74)   | 5747<br>(32)   | 6509<br>(17)   | 7613<br>(26)   | 7206<br>(69)   | 7498<br>(72)  | +13.6<br>0.191 |
| PLP               | 4.88                            | 6018<br>(77)   | 5818<br>(69)   | 6331<br>(52)   | 6731<br>(45)   | 6149<br>(144)  | 6778<br>(35)  | +12.6<br>0.040 |
| UNKNOWN           | 5.72                            | 2672<br>(42)   | 3116<br>(54)   | 3003<br>(112)  | 3448<br>(40)   | 3699<br>(25)   | 3747<br>(76)  | +40.2<br>0.087 |
| POS               | 5.88                            | 3442<br>(41)   | 4378<br>(10)   | 3882<br>(47)   | 4535<br>(15)   | 5171<br>(45)   | 5314<br>(51)  | +54.4<br>0.010 |
| POO               | 6.09                            | 25871<br>(851) | 26043<br>(340) | 26175<br>(224) | 31443<br>(145) | 30407<br>(245) | 31157<br>(31) | +20.4<br>0.001 |
| PLS               | 6.10                            | 9394<br>(6)    | 10895<br>(229) | 12448<br>(31)  | 9355<br>(156)  | 11475<br>(49)  | 12323<br>(42) | +31.2<br>0.084 |
| PLO               | 6.38                            | 40011<br>(338) | 40209<br>(249) | 41395<br>(211) | 39888<br>(156) | 39730<br>(59)  | 37282<br>(66) | - 6.8<br>0.475 |

cont.



Table 22 cont.

|      |      |                |                |                |                |                |                      |       |
|------|------|----------------|----------------|----------------|----------------|----------------|----------------------|-------|
| PLL  | 6.63 | 18924<br>(59)  | 18381<br>(207) | 17895<br>(75)  | 16631<br>(333) | 15969<br>(128) | 15707 -17.0<br>(265) | 0.006 |
| PLLn | 6.91 | 6653<br>(29)   | 7107<br>(19)   | 6913<br>(12)   | 7780<br>(61)   | 7824<br>(29)   | 8236 +23.8<br>(74)   | 0.089 |
| SOS  | 7.30 | 2036<br>(75)   | 2227<br>(26)   | 2160<br>(16)   | 1974<br>(37)   | 2340<br>(61)   | 2342 +15.0<br>(86)   | 0.597 |
| S00  | 7.59 | 9403<br>(58)   | 9765<br>(35)   | 9863<br>(44)   | 9891<br>(23)   | 10979<br>(32)  | 11305 +20.2<br>(65)  | 0.005 |
| 000  | 7.89 | 32357<br>(158) | 33657<br>(299) | 33601<br>(277) | 37803<br>(148) | 37817<br>(218) | 38624 +19.4<br>(197) | 0.005 |
| SLO  | 8.00 | 22156<br>(215) | 22841<br>(270) | 23615<br>(148) | 21679<br>(124) | 23724<br>(184) | 24240 + 9.4<br>(265) | 0.458 |
| OOL  | 8.28 | 54486<br>(212) | 48440<br>(174) | 52402<br>(112) | 41127<br>(240) | 48175<br>(124) | 49220 - 9.7<br>(273) | 0.412 |
| SLL  | 8.38 | 16316<br>(256) | 24288<br>(106) | 20495<br>(384) | 19019<br>(236) | 20504<br>(246) | 20486 +25.6<br>(91)  | 0.016 |
| OLL  | 8.63 | 47631<br>(197) | 45097<br>(142) | 43427<br>(46)  | 34430<br>(86)  | 38101<br>(132) | 37435 -21.4<br>(264) | 0.043 |
| LLL  | 8.95 | 16819<br>(132) | 12389<br>(289) | 10283<br>(39)  | 12315<br>(82)  | 11480<br>(91)  | 10908 -35.1<br>(76)  | 0.378 |

cont.

Table 22. cont.

|       |        |        |        |        |        |        |       |
|-------|--------|--------|--------|--------|--------|--------|-------|
| Total | 321571 | 320981 | 321972 | 323641 | 321965 | 322756 | 0.108 |
|-------|--------|--------|--------|--------|--------|--------|-------|

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
 L: linoleic acid (C<sub>18</sub>:2) and Ln: linolenic acid (C<sub>18</sub>:3).

S: significance.

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 14 days of static heating.

A: average of two replicates.

0.005, 0.005 and 0.016, respectively). However, PLL and OLL were significantly reduced ( $P < 0.006$  and  $0.043$ , respectively). From this experiment it was found that PLO with three double bonds showed a reduction from 40011 to 37282 in the peak area, while the PLLn triglyceride with five double bonds tended to accumulate from 6653 to 8234 in the peak area. In general, triglycerides with 1 to 5 double bonds tended to accumulate during heating, while other triglycerides with 3 to 6 double bonds tended to decrease in concentration. These findings contradict previous studies (Raughveer and Hammond, 1967; Yoshida and Alexander, 1984), because TG with 4 and 5 double bonds manifested relative resistance to oxidation.

The following major conclusions were obtained from the static heating experiment:

1. In C/O, CS/O, CS/OLS, P/O and SB/OLS, triglycerides with 1-3, 1-4, 0-4, 1-5 and 1-5 double bonds tend to accumulate, while other triglycerides with 2-7, 1-6, 1 and 4-6, 2-6 and 3-6 double bonds tend to decrease with prolonged heating periods, respectively. This indicated that triglycerides with 4-7, 5-6, 5-6, 6 and 6 double bonds were unstable to oxidation, respectively. Therefore, it was not possible to specify the relation between the exact number of double bonds in triglycerides with their rate of deterioration because it varied among

different shortenings. Eventhough, LLL was always unstable in all shortenings.

2. In corn oil, the presence of palmitic acid (C16:0) in PLP and POP triglycerides provided better protection to unsaturated fatty acids on the sn-2 position when compared to stearic acid (C18:0) in PLS and POS. Furthermore, in cottonseed oil, the presence of C16:0 in POP, PLO and PLL triglycerides provided better protection to unsaturated fatty acids on the sn-2 position, as compared to C18:0 in POS, SLO and SLL triglycerides. This phenomenon was not observed in either cottonseed or soybean oil liquid shortenings. In peanut oil, however, C16:0 and C18:0 provided better protection to unsaturated fatty acids on the sn-2 position of triglycerides, as compared to arachidic acid C20:0, behenic acid C22:0 and lignoceric acid C24:0. Therefore, it is concluded that short chain saturated fatty acids could provide better protection to unsaturated fatty acids when compared with long chain saturated fatty acids. In other words the molecular weight of triglycerides may effect their stability toward oxidation with specific types of shortenings.

3. Triglycerides with five and six double bonds, such as OLL and LLL, decreased in all five shortenings. However, PLL triglyceride with four double bonds decreased in peak area after fourteen days of heating in corn oil, cottonseed oil liquid shortening, peanut oil, and soybean

oil liquid shortening; however, the peak area increased in cottonseed oil.

4. OLO was stable in corn oil and cottonseed oil, increased in cottonseed oil liquid shortening, but decreased in peanut oil and was not present in soybean oil liquid shortening. SLL increased in soybean and cottonseed oil liquid shortening and decreased in cottonseed and peanut oils. P11n, which has five double bonds, increased in soybean oil liquid shortening.

The triglyceride patterns of each shortening contributed differently to the deterioration rate of the particular triglyceride. Nevertheless, the stereochemistry of polyunsaturated fatty acids is not necessarily an important factor in determining the susceptibility of a particular triglyceride to oxidation. Each triglyceride has its own rate of deterioration which is controlled by the availability of other triglycerides with different carbon numbers, degree of unsaturation and stereochemistry.

#### 4.1.9 FLAVOR EVALUATION OF HEATED SHORTENINGS.

4.1.9.1 Effect of static heating time (days) on the sensory analysis of fried chicken nuggets evaluated for flavor in five types of shortenings.

Average ratings for chicken nuggets cooked in fresh oils and liquid shortenings ranged from 7.9 to 9.7 (Table 23 and Fig. 20). The average rating failed to provide acceptable correlation coefficients with heating times. After 14 days of heating, the average ratings ranged from 7.60 to 8.33. An Analysis of Variance (ANOVA) showed a significant ( $P < 0.0001$ ) effect of shortening types on average flavor ratings. However, heating time had a significant ( $P < 0.0001$ ) effect on the average flavor rating of CS/OLS only.

From the orthogonal contrast study of flavor rating of corn oil, cottonseed oil, peanut oil and soybean oil liquid shortening, it was found that there were neither significant ( $P < 0.1527$  to  $0.6820$ ,  $P < 0.1803$  to  $0.9475$ ,  $P < 0.0851$  to  $0.8981$  and  $P < 0.1219$  to  $1.000$ , respectively) differences between unheated and heated samples, nor among heated samples (2 to 14 days period). This finding indicated their flavor stabilities under static heating conditions. However, the flavor rating of cottonseed oil liquid shortening showed significant ( $P < 0.0001$ ) difference between unheated and heated samples, but not ( $P < 0.5849$  to  $0.9274$ ) among heated samples. This observation indicated that static heating conditions had an immediate effect on changes in CS/OLS flavor, however, this change in flavor was insignificant with prolonged heating period.

Table 23. Effect of static heating time on average rancid flavor intensity of chicken nuggets fried in shortenings heated for varying time intervals.

| HEATING TIME<br>(DAYS) | RANCID FLAVOR RATING <sup>1,2,6</sup> |                   |                     |                  |                     |
|------------------------|---------------------------------------|-------------------|---------------------|------------------|---------------------|
|                        | SHORTENING TYPES <sup>4</sup>         |                   |                     |                  |                     |
|                        | C/O <sup>5</sup>                      | CS/O <sup>5</sup> | CS/OLS <sup>5</sup> | P/O <sup>5</sup> | SB/OLS <sup>5</sup> |
| 0                      | 7.93<br>(1.77)                        | 8.33<br>(1.73)    | 9.73<br>(0.45)      | 8.67<br>(1.72)   | 8.07<br>(1.44)      |
| 2                      | 8.50<br>(1.85)                        | 8.30<br>(1.91)    | 8.57<br>(1.61)      | 8.56<br>(1.61)   | 8.43<br>(1.40)      |
| 5                      | 8.13<br>(1.67)                        | 7.67<br>(2.20)    | 8.70<br>(1.11)      | 8.50<br>(2.01)   | 8.10<br>(1.90)      |
| 8                      | 8.50<br>(1.59)                        | 7.70<br>(1.80)    | 8.50<br>(1.66)      | 7.67<br>(2.45)   | 7.40<br>(1.56)      |
| 11                     | 8.30<br>(1.70)                        | 7.50<br>(2.22)    | 8.36<br>(1.84)      | 8.40<br>(1.59)   | 7.40<br>(1.86)      |
| 14                     | 7.60<br>(2.55)                        | 7.87<br>(1.81)    | 8.33<br>(1.35)      | 7.50<br>(2.47)   | 7.97<br>(2.16)      |
| $r^3$                  | -0.32                                 | -0.71             | -0.76               | -0.77            | -0.57               |

<sup>1</sup>: Average of two replicates and 15 panelists.

<sup>2</sup>: Rating scale, 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.

<sup>3</sup>: Correlation of rancid flavor intensity with heating time.

<sup>4</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

Table 23. cont.

- 
- <sup>5</sup>: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.
- <sup>6</sup>: Standard deviations are the values in brackets.



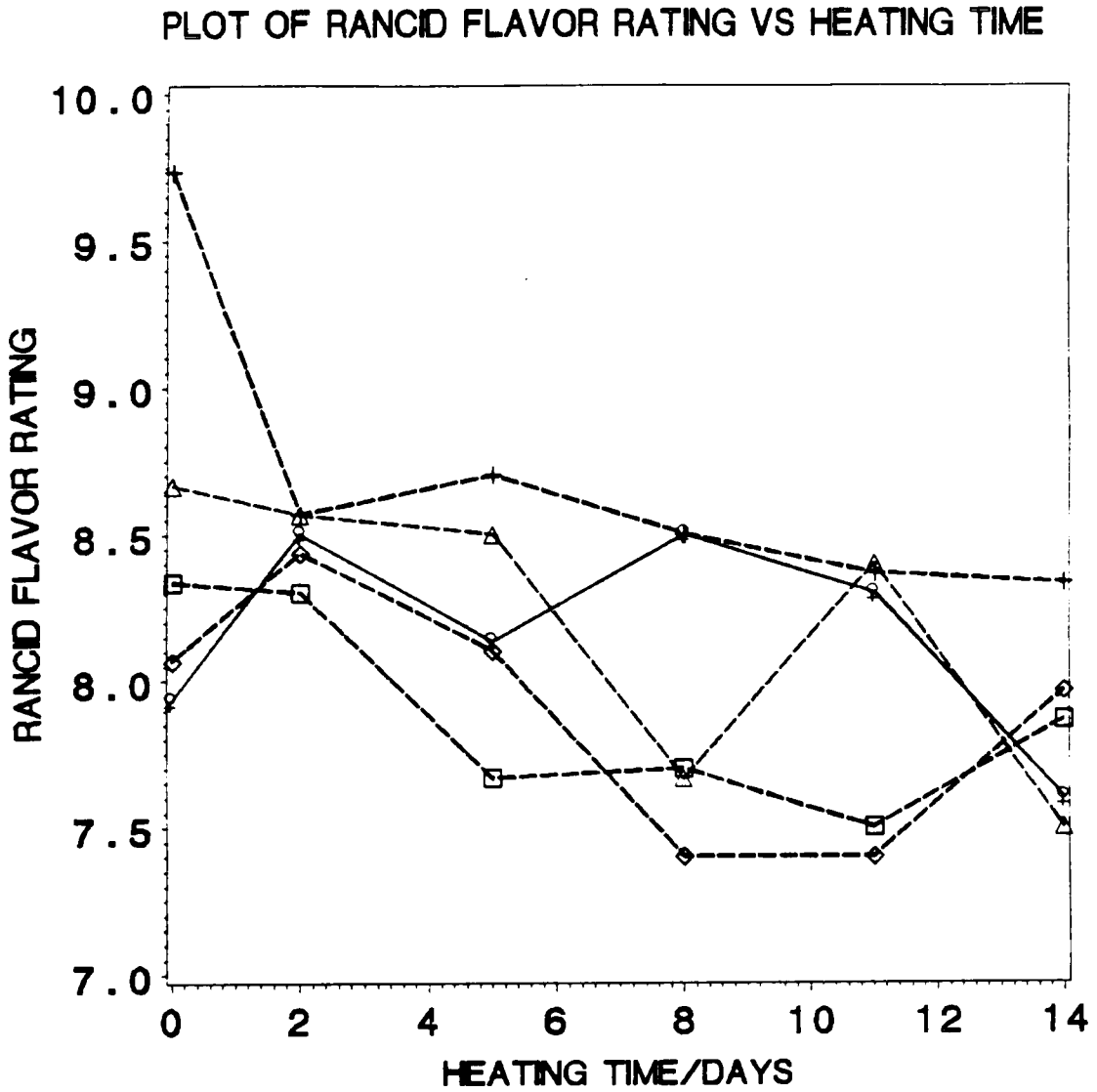


Fig. 20. Effect of static heating time on chicken nugget cooked for rancid flavor rating of Corn (○), Cottonseed (□), Peanut oils (△), Cottonseed (+) and Soybean (◇) Oil Liquid Shortenings. Lower rating indicates rancidity.

These results indicated that the 15 panelists could not differentiate significantly among nuggets fried in heated shortenings due to the shortenings relative stabilities. Because the intensity and the quality of the rancid flavor, developed under static heating conditions for all the shortenings, were not objectionable to the panelists. Therefore, the priority in rating frying oil flavor should be given to the interaction of food flavor with the frying shortening, which could be the most important factor that regulates or develops an undesirable flavor in the shortening. This interaction may vary between types of food and shortening used. No comment was received concerning flavor undesirability of any sample. Furthermore, a few panelists indicated that a slight rancidity in shortening tends to impart a nugget flavor identical to commercially fried nuggets.

Nonetheless, it was observed that frying the nuggets in shortenings statically heated to fourteen days, resulted in significant foaming as compared to other heating periods.

#### 4.1.9.2 EFFECT OF 5 AND 11 DAYS OF STATIC HEATING OF FIVE SHORTENINGS, ON FRIED CHICKEN NUGGETS FOR FLAVOR.

On the average, both heating time and shortening type had a significant ( $P < 0.01$  and  $0.0003$  respectively) effect

on the average rancid flavor rating during both heating periods. After five days of heating, the average rancid flavor rating was in the range of 7.37 to 8.43 (Table 24). Duncan's test produced no significant ( $\alpha=0.05$ ) differences among any of the shortenings. However, after eleven days of heating, the average rancid flavor rating was in the range of 5.93 to 7.93. Duncan's test showed no significant ( $\alpha=0.05$ ) difference between the first four shortenings; in contrast, SB/OLS was significantly ( $\alpha=0.05$ ) lower than the other four.

#### 4.1.10 ODOR EVALUATION OF HEATED SHORTENINGS.

##### 4.1.10.1 EFFECT OF STATIC HEATING TIME (DAYS) ON THE ODOR OF FIVE TYPES OF SHORTENINGS.

The rancid odor intensity of the five shortenings was determined by introducing to the fifteen panelists 5 ml samples of statically heated shortening in 50 ml beakers. Control fresh shortenings were given an average rating range of 9.07 to 9.67 (Table 25). Each point is an average of all the panelists in duplicate. After 14 days of heating, the average rancid odor intensity rating ranged from 6.37 to 6.50. On the average, both heating time and shortening type had a significant ( $P>0.0001$ ) effect on the

Table 24. Effect of two static heating times on average rancid flavor intensity of chicken nuggets fried in five different shortenings.

|                        |                               | RANCID FLAVOR RATING <sup>1,2,3</sup> |                   |                   |                                |
|------------------------|-------------------------------|---------------------------------------|-------------------|-------------------|--------------------------------|
| HEATING TIME<br>(DAYS) | SHORTENING TYPES <sup>4</sup> |                                       |                   |                   |                                |
|                        | C/O                           | CS/O                                  | CS/OLS            | P/O               | SB/OLS                         |
| 5                      | 8.23(A)<br>(1.50)             | 7.40(A)<br>(2.11)                     | 8.43(A)<br>(1.65) | 7.70(A)<br>(1.74) | 7.37(A) <sup>4</sup><br>(2.39) |
| 11                     | 7.77(A)<br>(2.16)             | 7.37(A)<br>(2.13)                     | 7.93(A)<br>(1.72) | 7.17(A)<br>(1.48) | 5.93(B)<br>(2.69)              |

<sup>1</sup>: Average of two replicates and 15 panelists.

<sup>2</sup>: Rating scale, 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>5</sup>: Duncan's multiple range test: means with the same letter are not significantly different for one heating period (rows).

Table 25. Effect of static heating time on average rancid odor intensity of five different shortenings heated up to 14 days.

| RANCID ODOR RATING <sup>1,2,6</sup> |                               |                   |                     |                  |                     |
|-------------------------------------|-------------------------------|-------------------|---------------------|------------------|---------------------|
| HEATING TIME<br>(DAYS)              | SHORTENING TYPES <sup>4</sup> |                   |                     |                  |                     |
|                                     | C/O <sup>5</sup>              | CS/O <sup>5</sup> | CS/OLS <sup>5</sup> | P/O <sup>5</sup> | SB/OLS <sup>5</sup> |
| 0                                   | 9.57<br>(0.82)                | 9.67<br>(0.47)    | 9.67<br>(1.12)      | 9.07<br>(1.84)   | 9.47<br>(0.73)      |
| 2                                   | 6.80<br>(2.07)                | 7.67<br>(1.84)    | 7.73<br>(1.72)      | 7.07<br>(1.74)   | 4.83<br>(2.12)      |
| 5                                   | 7.03<br>(1.73)                | 6.60<br>(1.81)    | 6.80<br>(1.84)      | 7.10<br>(1.71)   | 5.40<br>(1.49)      |
| 8                                   | 6.83<br>(1.83)                | 7.03<br>(1.27)    | 7.00<br>(1.51)      | 6.90<br>(1.83)   | 5.23<br>(1.99)      |
| 11                                  | 7.47<br>(1.59)                | 7.27<br>(1.89)    | 7.00<br>(1.93)      | 6.37<br>(1.65)   | 6.03<br>(1.94)      |
| 14                                  | 6.33<br>(2.18)                | 6.50<br>(1.83)    | 6.50<br>(1.96)      | 5.47<br>(1.88)   | 6.37<br>(1.83)      |
| $r^3$                               | -0.63                         | -0.73             | -0.79               | -0.89            | -0.33               |

<sup>1</sup>: Average of two replicates and 15 panelists.

<sup>2</sup>: Rating scale, 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.

<sup>3</sup>: Correlation of rancid odor intensity with heating time.

<sup>4</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

## Table 25. cont.

- 
- 5: Significant ( $p < 0.0001$ ) effect of heating time (days), and significant ( $p < 0.0001$ ) differences among shortenings.
- 6: Standard deviations are the values in brackets.

rating (Fig. 21), which indicated that rancid odor is easier to detect than rancid taste.

From the orthogonal contrast study of odor rating, significant ( $P < 0.0001$ ) differences were found between unheated and heated samples of the oils and liquid shortenings. Furthermore, samples heated from 2 to 11 days, showed insignificant ( $P < 0.1661$  to  $0.6611$ ) differences in their odor ratings, however, samples heated to 14 days had significantly ( $P < 0.0138$ ) lower ratings as compared to the 11 days heated samples. The odor ratings of cottonseed oil and cottonseed oil liquid shortening samples, heated to 5 days were significantly ( $P < 0.0109$  and  $0.0356$ , respectively) lower than those heated only to 2 days, however, no significant ( $P < 0.0662$  to  $0.5744$  and  $P < 0.2581$  to  $1.000$ , respectively) differences among samples heated between 5 to 14 days were found. In the case of peanut oil and soybean oil liquid shortening, no significant ( $P < 0.0514$  to  $0.9422$  and  $P < 0.0782$  to  $0.7125$ , respectively) differences among heated samples were found.

After two days of heating, SB/OLS received the lowest scores because of a perceived intense painty odor. The odor was reduced and eliminated with continuous heating up to 14 days as indicated by the sensory rating. The origin of this odor was probably due to the extraction of soybean oil from premature soybean seeds. Peanut oil was the only shortening among the five to show significant ( $\alpha = 0.1$ )

### PLOT OF RANCID ODOR RATING VS HEATING TIME

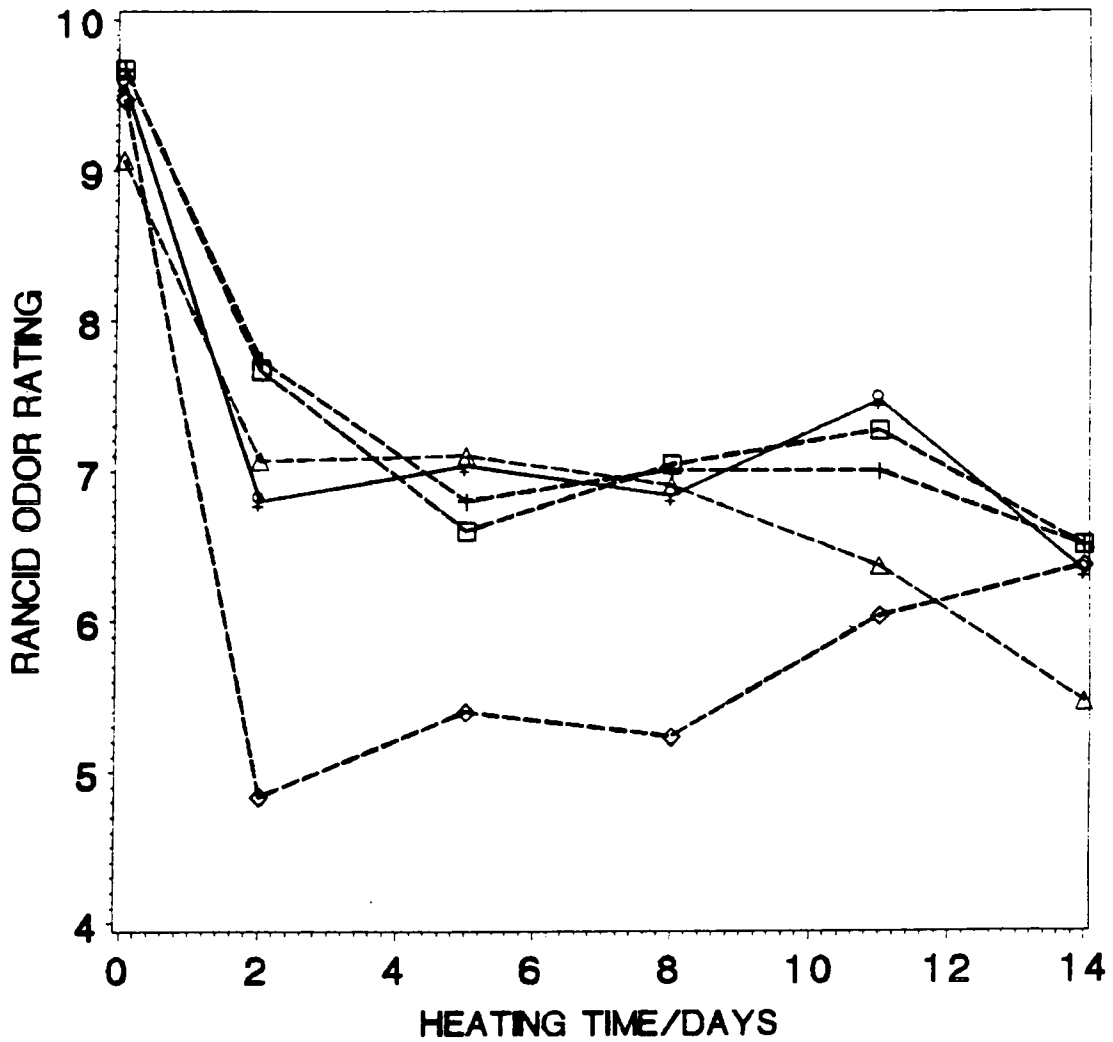


Fig. 21. Effect of static heating Time on rancid odor rating of Corn (♀), Cottonseed (□), Peanut oils (Δ), Cottonseed (+) and Soybean (◇) Oil Liquid Shortenings. Lower rating indicates rancidity.



linearity. Its correlation coefficient of rancid odor rating with heating time was  $r=0.89$ .

In general, corn oil, cottonseed oil and cottonseed oil liquid shortening had stable rancid odors between day two to fourteen of static heating. However, peanut oil odor started deteriorating after day five of heating. The lowest rating after two days of heating was received by soybean oil liquid shortening; yet, its odor improved steadily thereafter.

#### 4.1.10.2 EFFECT OF 5 AND 11 DAYS OF STATIC HEATING ON THE ODOR OF FIVE SHORTENINGS.

By comparing the average rancid odor ratings among the five shortenings statically heated in duplicate up to five and eleven days, the following was observed. On the average, there were significant ( $P<0.0001$ ) differences between shortening rancid odor ratings (Table 26); heating times, nevertheless, had no significant ( $P<0.529$ ) effect on rancid odor rating between five and eleven days of heating. After five days, the ratings were in the range of 4.73 to 7.33 (Table 26). Duncan's test showed no significant ( $\alpha=0.05$ ) differences between C/O, CS/O, CS/OLS and P/O; however SB/OLS was significantly ( $\alpha=0.05$ ) lower than the other four. This indicated that its odor deteriorated at a faster rate than the other shortenings. After eleven days

Table 26. Effect of two static heating times on average rancid odor intensity of five different shortenings.<sup>2</sup>

| RANCID ODOR RATING <sup>1,2,3</sup> |                               |                     |                     |                   |                                |
|-------------------------------------|-------------------------------|---------------------|---------------------|-------------------|--------------------------------|
| HEATING TIME<br>(DAYS)              | SHORTENING TYPES <sup>4</sup> |                     |                     |                   |                                |
|                                     | C/O                           | CS/O                | CS/OLS              | P/O               | SB/OLS                         |
| 5                                   | 6.33(A)<br>(1.94)             | 7.13(A)<br>(1.47)   | 7.33(A)<br>(1.42)   | 6.33(A)<br>(2.29) | 4.73(B) <sup>5</sup><br>(2.22) |
| 11                                  | 6.27(B)<br>(1.84)             | 6.70(A,B)<br>(1.82) | 7.10(A,B)<br>(2.14) | 7.57(A)<br>(1.65) | 4.93(C)<br>(2.46)              |

<sup>1</sup>: Average of two replicates and 15 panelists.

<sup>2</sup>: Rating scale, 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.

<sup>3</sup>: Standard deviations are the values in brackets.

<sup>4</sup>: C/O (corn oil), CS/O (cottonseed oil), CS/OLS (cottonseed oil liquid shortening), P/O (peanut oil) and SB/OLS (soybean oil liquid shortening).

<sup>5</sup>: Duncan's multiple range test: means with the same letter are not significantly different for one heating period (rows).

of static heating, the rancid odor rating was in the range of 4.93 to 7.57. Duncan's test showed no significant ( $\alpha=0.05$ ) differences among P/O, CS/O and CS/OLS; and among CS/O, CS/OLS and C/O, there were, however, significant ( $\alpha=0.05$ ) differences between SB/OLS and the other four shortenings. In both trials, soybean oil liquid shortening had the lowest rating. This finding was reported in previous studies by Blumenthal et al. (1976).

In general, from both odor and flavor analyses, SB/OLS had the lowest rating, and cottonseed oil and its liquid shortening was significantly competitive ( $\alpha=0.05$ ) with peanut oil. Furthermore, flavor and odor analyses could not be considered as good indicators of heat abuse, since both test failed to indicated final cut-off level of unacceptability.

#### 4.1.1.11 THE LINEAR REGRESSION STUDY

##### 4.1.1.11.1 THE CORRELATION COEFFICIENTS AMONG NINE SUBJECTIVE AND OBJECTIVE ANALYSES APPLIED TO FIVE STATICALLY HEATED SHORTENINGS

Tables 27-31 explain the relationships between nine tests used to evaluate the quality of five types of commercial shortenings heated statically at 0, 2, 5, 8, 11 and 14

Table 27. Correlation coefficients among free fatty acid (FFA), polar component, dielectric constant (FOS), viscosity, quantitative high performance thin layer chromatography (QHPTLC), volatile profile analysis, contact angle measurement and sensory analyses of rancid flavor and odor intensities of statically heated corn oil.

| PHYSICAL, CHEMICAL AND SENSORY ANALYSES |                  |                  |                  |                  |                  |                  |                   |                     |
|---|------------------|------------------|------------------|------------------|------------------|------------------|-------------------|---------------------|
| TESTS                                   | FFA <sup>1</sup> | FOS <sup>2</sup> | VIS <sup>3</sup> | TLC <sup>4</sup> | VPA <sup>5</sup> | CON <sup>6</sup> | ODOR <sup>7</sup> | FLAVOR <sup>8</sup> |
| FFA <sub>9</sub>                        | --               | --               | --               | 0.96             | 0.72             | 0.78             | -0.67             | -0.34               |
| POL <sup>9</sup>                        | 0.98             | 1.00             | 0.97             | 0.99             | 0.80             | 0.73             | -0.73             | -0.23               |
| FOS                                     | 0.99             | --               | 0.97             | 0.99             | 0.79             | 0.76             | -0.72             | -0.29               |
| VIS                                     | 0.99             | --               | --               | 0.93             | 0.65             | 0.77             | -0.59             | -0.37               |
| VPA                                     | --               | --               | --               | 0.86             | --               | 0.55             | -0.97             | -0.20               |
| CON                                     | --               | --               | --               | --               | --               | --               | -0.65             | -0.64               |

<sup>1</sup>: Free fatty acid (%oleic acid).

<sup>2</sup>: FoodOil-Sensor readings (dielectric constants).

<sup>3</sup>: Viscosity (CP) determined at 40°C.

<sup>4</sup>: Polar component% measured by quantitative high performance thin layer chromatography.

<sup>5</sup>: Volatile profile analysis (gas chromatographic method).

<sup>6</sup>: Contact angle of the shortenings at 28.5 ± 0.5 °C measured on FT-120-WW surface.

<sup>7</sup>: Rancid odor evaluation at room temperature.

<sup>8</sup>: Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings.

<sup>9</sup>: Polar component%.

Table 28. Correlation coefficients among free fatty acid (FFA), polar component, dielectric constant (FOS), viscosity, quantitative high performance thin layer chromatography (QHPTLC), volatile profile analysis, contact angle measurement and sensory analyses of rancid flavor and odor intensities of statically heated cottonseed oil.

| PHYSICAL, CHEMICAL AND SENSORY ANALYSES |                  |                  |                  |                    |                  |                  |                   |                     |
|---|------------------|------------------|------------------|--------------------|------------------|------------------|-------------------|---------------------|
| TESTS                                   | FFA <sup>1</sup> | FOS <sup>2</sup> | VIS <sup>3</sup> | HPTLC <sup>4</sup> | VPA <sup>5</sup> | CON <sup>6</sup> | ODOR <sup>7</sup> | FLAVOR <sup>8</sup> |
| FFA <sub>9</sub>                        | --               | --               | --               | 0.94               | 0.84             | 0.61             | -0.70             | -0.63               |
| POL <sub>9</sub>                        | 0.97             | 1.00             | 0.95             | 0.98               | 0.89             | 0.57             | -0.81             | -0.79               |
| FOS                                     | 0.97             | --               | 0.96             | 0.98               | 0.90             | 0.60             | -0.81             | -0.78               |
| VIS                                     | 0.99             | --               | --               | 0.91               | 0.81             | 0.64             | -0.68             | -0.59               |
| VPA                                     | --               | --               | --               | 0.95               | --               | 0.65             | -0.87             | -0.70               |
| CON                                     | --               | --               | --               | --                 | --               | --               | -0.83             | -0.26               |

<sup>1</sup>: Free fatty acid (%oleic acid).

<sup>2</sup>: FoodOil-Sensor readings (dielectric constants).

<sup>3</sup>: Viscosity (CP) determined at 40°C.

<sup>4</sup>: Polar component% measured by quantitative high performance thin layer chromatography.

<sup>5</sup>: Volatile profile analysis (gas chromatographic method).

<sup>6</sup>: Contact angle of the shortenings at 28.5 ± 0.5 °C measured on FT-120-WW surface.

<sup>7</sup>: Rancid odor evaluation at room temperature.

<sup>8</sup>: Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings.

<sup>9</sup>: Polar component%.

Table 29. Correlation coefficients among free fatty acid (FFA), polar component, dielectric constant (FOS), viscosity, quantitative high performance thin layer chromatography (QHPTLC), volatile profile analysis, contact angle measurement and sensory analyses of rancid flavor and odor intensities of statically heated cottonseed oil liquid shortening.

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PHYSICAL, CHEMICAL AND SENSORY ANALYSES

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| TESTS            | FFA <sup>1</sup> | FOS <sup>2</sup> | VIS <sup>3</sup> | HPTLC <sup>4</sup> | VPA <sup>5</sup> | CON <sup>6</sup> | ODOR <sup>7</sup> | FLAVOR <sup>8</sup> |
|------------------|------------------|------------------|------------------|--------------------|------------------|------------------|-------------------|---------------------|
| FFA <sub>9</sub> | --               | --               | --               | 0.92               | 0.98             | 0.63             | -0.70             | -0.67               |
| POL <sup>9</sup> | 0.98             | 1.00             | 0.86             | 0.97               | 0.99             | 0.61             | -0.81             | -0.80               |
| FOS              | 0.98             | --               | 0.87             | 0.97               | 1.00             | 0.64             | -0.82             | -0.78               |
| VIS              | 0.92             | --               | --               | 0.83               | 0.86             | 0.74             | -0.53             | -0.49               |
| VPA              | --               | --               | --               | 0.96               | --               | 0.67             | -0.83             | -0.79               |
| CON              | --               | --               | --               | --                 | --               | --               | -0.60             | -0.60               |

---

<sup>1</sup>: Free fatty acid (%oleic acid).

<sup>2</sup>: FoodOil-Sensor readings (dielectric constants).

<sup>3</sup>: Viscosity (CP) determined at 40°C.

<sup>4</sup>: Polar component% measured by quantitative high performance thin layer chromatography.

<sup>5</sup>: Volatile profile analysis (gas chromatographic method).

<sup>6</sup>: Contact angle of the shortenings at 28.5 ± 0.5 °C measured on FT-120-WW surface.

<sup>7</sup>: Rancid odor evaluation at room temperature.

<sup>8</sup>: Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings.

<sup>9</sup>: Polar component%.

Table 30. Correlation coefficients among free fatty acid (FFA), polar component, dielectric constant (FOS), viscosity, quantitative high performance thin layer chromatography (QHPTLC), volatile profile analysis, contact angle measurement and sensory analyses of rancid flavor and odor intensities of statically heated peanut oil.

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PHYSICAL, CHEMICAL AND SENSORY ANALYSES

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| TESTS            | FFA <sup>1</sup> | FOS <sup>2</sup> | VIS <sup>3</sup> | HPTLC <sup>4</sup> | VPA <sup>5</sup> | CON <sup>6</sup> | ODOR <sup>7</sup> | FLAVOR <sup>8</sup> |
|------------------|------------------|------------------|------------------|--------------------|------------------|------------------|-------------------|---------------------|
| FFA <sub>9</sub> | --               | --               | --               | 0.97               | 0.98             | 0.80             | -0.91             | -0.82               |
| POL <sup>9</sup> | 0.99             | 1.00             | 0.98             | 0.99               | 0.97             | 0.75             | -0.93             | -0.82               |
| FOS              | 0.99             | --               | 0.97             | 0.99               | 0.98             | 0.74             | -0.94             | -0.82               |
| VIS              | 0.98             | --               | --               | 0.95               | 0.93             | 0.86             | -0.87             | -0.74               |
| VPA              | --               | --               | --               | 0.97               | --               | 0.70             | -0.98             | -0.78               |
| CON              | --               | --               | --               | --                 | --               | --               | -0.55             | -0.35               |

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1: Free fatty acid (%oleic acid).

2: FoodOil-Sensor readings (dielectric constants).

3: Viscosity (CP) determined at 40°C.

4: Polar component% measured by quantitative high performance thin layer chromatography.

5: Volatile profile analysis (gas chromatographic method).

6: Contact angle of the shortenings at 28.5 ± 0.5 °C measured on FT-120-WW surface.

7: Rancid odor evaluation at room temperature.

8: Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings.

9: Polar component%.

Table 31. Correlation coefficients among free fatty acid (FFA), polar component, dielectric constant (FOS), viscosity, quantitative high performance thin layer chromatography (QHPTLC), volatile profile analysis, contact angle measurement and sensory analyses of rancid flavor and odor intensities of statically heated soybean oil liquid shortening.

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PHYSICAL, CHEMICAL AND SENSORY ANALYSES

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| TESTS            | FFA <sup>1</sup> | FOS <sup>2</sup> | VIS <sup>3</sup> | HPTLC <sup>4</sup> | VPA <sup>5</sup> | CON <sup>6</sup> | ODOR <sup>7</sup> | FLAVOR <sup>8</sup> |
|------------------|------------------|------------------|------------------|--------------------|------------------|------------------|-------------------|---------------------|
| FFA <sup>9</sup> | --               | --               | --               | 0.93               | 0.87             | 0.67             | -0.16             | -0.51               |
| POL <sup>9</sup> | 0.92             | 1.00             | 0.98             | 1.00               | 0.96             | 0.66             | -0.43             | -0.59               |
| FOS              | 0.94             | --               | 0.99             | 1.00               | 0.97             | 0.69             | -0.43             | -0.58               |
| VIS              | 0.91             | --               | --               | 0.99               | 0.95             | 0.73             | -0.45             | -0.59               |
| VPA              | --               | --               | --               | 0.96               | --               | 0.76             | -0.48             | -0.39               |
| CON              | --               | --               | --               | --                 | --               | --               | -0.61             | -0.02               |

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<sup>1</sup>: Free fatty acid (%oleic acid).

<sup>2</sup>: FoodOil-Sensor readings (dielectric constants).

<sup>3</sup>: Viscosity (CP) determined at 40°C.

<sup>4</sup>: Polar component% measured by quantitative high performance thin layer chromatography.

<sup>5</sup>: Volatile profile analysis (gas chromatographic method).

<sup>6</sup>: Contact angle of the shortenings at 28.5 ± 0.5 °C measured on FT-120-WW surface.

<sup>7</sup>: Rancid odor evaluation at room temperature.

<sup>8</sup>: Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings.

<sup>9</sup>: Polar component%.



days, respectively. A high positive correlation ranging from 0.93 to 1.00 was found in corn oil (Table 27) between Free Fatty Acid% (FFA), FoodOil-Sensor Reading (FOS), Viscosity in Centipoise (VIS), High Performance Thin Layer Chromatography-Polar Component% (HPTLC) and Polar Component% (POLAR). Also, a high correlation occurred between Volatile Profile Analysis (VPA) and Polar Component% (POLAR) and between VPA and Polar Component% as determined by QHPTLC, 0.80 and 0.86; and between Rancid Odor Rating (ODOR) and VPA, 0.97. High positive correlations (Table 28) were found between FFA, FOS, VIS, HPTLC, VPA and POLAR in cottonseed oil with a range between 0.81-1.00. Rancid ODOR rating had high negative correlations (-0.81, -0.81, -0.87, and -0.83) with POLAR, FOS, VPA and CON (contact angle), respectively. Cottonseed oil liquid shortening (Table 29) also had high correlations between FFA, FOS, VIS, HPTLC, POLAR and VPA with a 0.83-1.00 range. Moreover ODOR had shown good negative (-0.81, -0.82, and -0.83) correlations with POLAR, FOS, and VPA respectively. FLAVOR showed only one high negative (-0.80) correlation with POLAR.

There are several advantages from obtaining good correlations among the tests. These correlations are particularly important when considering that the two standard point of references, polar component test and sensory evaluations of flavor and odor, are time consuming,

require training, costly and/or require large number of trained subjects. Therefore finding an acceptable a substitute evaluation method is considerably important. On the other hand, the dielectric constant test with FOS for example, is fast, simple and requires no reagents. Therefore, establishing high correlation coefficients between the FOS test with either the polar component test or sensory tests, would be advantageous, particularly after establishing a cut-off level for each particular heating conditions and and types of shortening type. This correlation also has an economic advantage, particularly with respect to developing countries.

In the case of peanut oil (Table 30), high positive correlations were found between FFA, FOS, VIS, HPTLC, POLAR and VPA in the range of 0.93-1.00. Contact angle tests showed good correlations with FFA ( $r=0.80$ ) and VIS (0.86). Also ODOR tests showed good correlations with FFA, POLAR, FOS, VIS and VPA, (-0.91, -0.93, -0.94, -0.87 and -0.98), respectively. Moreover, the rancid flavor intensity test (FLAVOR) was highly correlated with FFA, POLAR and FOS, with a correlation coefficient of -0.82 for all three tests. Nevertheless, soybean oil liquid shortening (Table 31) showed high positive correlations between FFA, FOS, VIS, HPTLC, VPA and POLAR within the 0.87-1.00 range. Since high correlation coefficients between all these tests were found for all five shortenings, standard curves can be

established from linear regression models between any two analytical methods.

#### 4.1.1.11.2 RELATIONS BETWEEN NINE PHYSICAL, CHEMICAL AND SENSORY TESTS APPLIED TO STATICALLY HEATED FRYING SHORTENINGS.

Relationships between highly correlated tests in peanut oil (P/O), cottonseed oil liquid shortening (CS/OLS), cottonseed oil (CS/O) soybean oil liquid shortening (SB/OLS) and corn oil (C/O) are discussed in this section. Specific tests included are Volatile Profile Analysis (VPA), Polar Component% (POL), Polar Component% by Quantitative High Performance Thin Layer Chromatography (TLC), Sensory Evaluation of Rancid Odor Intensity (ODOR), FoodOil-Sensor reading (FOS), Free Fatty Acid-% Oleic Acid (FFA), Viscosity in Centipoise (VIS), Contact Angle Measurement (CON) and Sensory Evaluation of Rancid Flavor Intensity (FLAV). The linear regression equations with the high correlation coefficients are presented in Tables 32-36, and their linear regression plots (standard curves) are presented in Fig. 13-34 in Appendix G.

When high a correlation coefficient is obtained between only two tests, the data of one test can be used to predict the data of the other test.

Table 32. Linear regression equations and correlation coefficients among seven tests on corn oil under static heating conditions.

| Tests <sup>1</sup> | Linear Regression Equations <sup>3</sup> | r <sup>2</sup> |
|--------------------|--|----------------|
| VPA vs POL         | $y = 2070^C + 158^D x$                   | 0.80           |
| VPA vs TLC         | $y = 834^C + 142^D x$                    | 0.86           |
| VPA vs ODOR        | $y = 17614^D - 1697^A x$                 | -0.97          |
| TLC vs FOS         | $y = -6.73^C + 10.21^B x$                | 0.99           |
| TLC vs FFA         | $y = 14.18^C + 131.94^B x$               | 0.96           |
| TLC vs VIS         | $y = -17.47^D + 0.98^A x$                | 0.93           |
| VIS vs FOS         | $y = 14.01^C + 9.51^E x$                 | 0.97           |
| VIS vs POL         | $y = 27.24^C + 1.10^E x$                 | 0.97           |
| VIS vs FFA         | $y = 32.87^E + 126.67^E x$               | 0.99           |
| FFA vs POL         | $y = -0.05^{A,B} + 0.009^B x$            | 0.98           |
| FOS vs FFA         | $y = 2.04^B + 13.04^D x$                 | 0.99           |
| FOS vs POL         | $y = 1.39^B + 0.115^A x$                 | 1.00           |
| TLC vs POL         | $y = 6.93^C + 1.210^B x$                 | 0.99           |

<sup>1</sup>: Tests:

- VPA (total volatile profile).
- POL (polar component%).
- TLC (polar component%).
- ODOR (sensory evaluation of rancid odor intensity).
- FOS (FoodOil-Sensor reading).
- FFA (free fatty acid - %oleic acid).
- VIS (viscosity in centipoise).

<sup>2</sup>: Correlation coefficient.

<sup>3</sup>: Duncan's multiple range test: same letters have no significant difference ( $\alpha = 0.05$ ).

Table 33. Linear regression equations and correlation coefficients among eight tests on cottonseed oil used under static heating conditions.

| Tests <sup>1</sup> | Linear Regression Equations <sup>3</sup> | r <sup>2</sup> |
|--------------------|--|----------------|
| VPA vs POL         | $y = 138^D + 250^B x$                    | 0.89           |
| VPA vs TLC         | $y = -12304^A + 470^C x$                 | 0.95           |
| VPA vs ODOR        | $y = 23564^C - 2301^C x$                 | -0.88          |
| VPA vs VIS         | $y = 1704^D + 181.6^A x$                 | 0.81           |
| VPA vs FOS         | $y = -2736^D + 2077^A x$                 | 0.90           |
| TLC vs FOS         | $y = 19.83^A + 4.54^E x$                 | 0.98           |
| TLC vs FFA         | $y = 31.68^A + 64.59^E x$                | 0.94           |
| TLC vs VIS         | $y = 16.40^A + 0.42^C x$                 | 0.91           |
| VIS vs FOS         | $y = 12.42^D + 9.87^D x$                 | 0.96           |
| VIS vs POL         | $y = 25.87^D + 1.20^C x$                 | 0.95           |
| VIS vs FFA         | $y = 36.63^D + 150.56^C x$               | 0.99           |
| FFA vs VPA         | $y = 2869^B + 28131^A x$                 | 0.84           |
| FFA vs POL         | $y = -0.08^B + 0.008^{A, B} x$           | 0.97           |
| FOS vs FFA         | $y = 2.62^A + 14.19^B x$                 | 0.97           |
| FOS vs POL         | $y = 1.35^C + 0.12^A x$                  | 1.00           |
| TLC vs POL         | $y = 25.83^A + 0.56^E x$                 | 0.97           |
| ODOR vs POL        | $y = 9.65^A - 0.087^A x$                 | -0.81          |
| ODOR vs FOS        | $y = 10.6^A - 0.710^A x$                 | -0.81          |
| CON vs ODOR        | $y = 53.3 - 1.39x$                       | -0.83          |

<sup>1</sup>: Tests:

- VPA (total volatile profile).
- POL (polar component%).
- TLC (polar component%).
- ODOR (sensory evaluation of rancid odor intensity).
- FOS (FoodOil-Sensor reading).
- FFA (free fatty acid - %oleic acid).
- VIS (viscosity in centipoise).
- CON (contact angle measurement).

<sup>2</sup>: Correlation coefficient.

Table 33. cont.

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<sup>3</sup>: Duncan's multiple range test: same letters have no significant difference ( $\alpha = 0.05$ ).

Table 34. Linear regression equations and correlation coefficients among eight tests on cottonseed oil liquid shortening used under static heating conditions.

| Tests <sup>1</sup> | Linear Regression Equations <sup>3</sup> | r <sup>2</sup> |
|--------------------|--|----------------|
| VPA vs POL         | $y = 2597^A + 204^C x$                   | 0.99           |
| VPA vs TLC         | $y = 2058^B + 205^C x$                   | 0.96           |
| VPA vs ODOR        | $y = 21043^C - 1833^D x$                 | -0.83          |
| VPA vs VIS         | $y = 5904^A + 11.88^D x$                 | 0.86           |
| VPA vs FOS         | $y = 259^B + 1691^C x$                   | 1.00           |
| TLC vs FOS         | $y = -6.76^C + 7.76^C x$                 | 0.97           |
| TLC vs FFA         | $y = 10.66^D + 105.89^C x$               | 0.91           |
| TLC vs VIS         | $y = 16.09^A + 0.05^E x$                 | 0.83           |
| VIS vs FOS         | $y = -265.30^E + 106.77^A x$             | 0.87           |
| VIS vs POL         | $y = -116.78^E + 12.83^A x$              | 0.86           |
| VIS vs FFA         | $y = -55.81^A + 1656^A x$                | 0.92           |
| FFA vs VPA         | $y = 3907^A + 24093^D x$                 | 0.98           |
| FFA vs POL         | $y = -0.05^{A,B} + 0.01^{A,B} x$         | 0.98           |
| FOS vs FFA         | $y = 2.17^A + 14.17^C x$                 | 0.98           |
| FOS vs POL         | $y = 1.37^A + 0.12^{A,B} x$              | 1.00           |
| TLC vs POL         | $y = 3.77^D + 0.94^C x$                  | 0.98           |
| ODOR vs POL        | $y = 9.23^B - 0.075^A x$                 | -0.81          |
| ODOR vs FOS        | $y = 10.10^{A,B} - 0.630^A x$            | -0.82          |
| FLAV vs POL        | $y = 9.49^A - 0.0343^A x$                | -0.80          |

1: Tests:

- VPA (total volatile profile).
- POL (polar component%).
- TLC (polar component%).
- ODOR (sensory evaluation of rancid odor intensity).
- FOS (FoodOil-Sensor reading).
- FFA (free fatty acid - %oleic acid).
- VIS (viscosity in centipoise).
- CON (contact angle measurement).

2: Correlation coefficient.

Table 34. cont.

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<sup>3</sup>: Duncan's multiple range test: same letters have no significant difference ( $\alpha = 0.05$ ).



Table 35. Linear regression equations and correlation coefficients among nine tests on peanut oil used under static heating conditions.

| Tests <sup>1</sup> | Linear Regression Equations <sup>3</sup> |                          | r <sup>2</sup> |
|--------------------|--|--------------------------|----------------|
| VPA vs POL         | y = 141 <sup>D</sup>                     | + 259 <sup>A</sup> x     | 0.97           |
| VPA vs TLC         | y = -5695 <sup>D</sup>                   | + 325 <sup>B</sup> x     | 0.97           |
| VPA vs ODOR        | y = 22751 <sup>B</sup>                   | - 2453 <sup>D</sup> x    | -0.98          |
| VPA vs VIS         | y = 247.7 <sup>D</sup>                   | + 176.25 <sup>B</sup> x  | 0.93           |
| VPA vs FOS         | y = -860 <sup>C</sup>                    | + 1981 <sup>B</sup> x    | 0.98           |
| TLC vs FOS         | y = 14.93 <sup>B</sup>                   | + 6.06 <sup>D</sup> x    | 0.99           |
| TLC vs FFA         | y = 22.55 <sup>B</sup>                   | + 75.79 <sup>D</sup> x   | 0.97           |
| TLC vs VIS         | y = 1.83 <sup>B</sup>                    | + 0.54 <sup>B</sup> x    | 0.95           |
| VIS vs FOS         | y = 26.51 <sup>A</sup>                   | + 10.37 <sup>C</sup> x   | 0.97           |
| VIS vs POL         | y = 31.45 <sup>B</sup>                   | + 1.37 <sup>C</sup> x    | 0.98           |
| VIS vs FFA         | y = 38.56 <sup>C</sup>                   | + 135.29 <sup>D</sup> x  | 0.98           |
| FFA vs VPA         | y = 1486 <sup>D</sup>                    | + 25396 <sup>C</sup> x   | 0.97           |
| FFA vs POL         | y = -0.05 <sup>A, B</sup>                | + 0.01 <sup>A, B</sup> x | 0.99           |
| FOS vs FFA         | y = 1.21 <sup>C</sup>                    | + 12.77 <sup>E</sup> x   | 0.99           |
| FOS vs POL         | y = 0.49 <sup>A</sup>                    | + 0.13 <sup>A, B</sup> x | 1.00           |
| TLC vs POL         | y = 17.84 <sup>D</sup>                   | + 0.80 <sup>D</sup> x    | 0.99           |
| ODOR vs POL        | y = 9.11 <sup>B</sup>                    | - 0.1 <sup>A</sup> x     | -0.93          |
| ODOR vs FFA        | y = 8.51                                 | - 9.46 x                 | -0.91          |
| ODOR vs VIS        | y = 11                                   | - 0.066 <sup>A</sup> x   | -0.87          |
| ODOR vs FOS        | y = 9.5 <sup>B</sup>                     | - 0.7600 <sup>A</sup> x  | -0.93          |
| FLAV vs POL        | y = 9.03 <sup>B</sup>                    | - 0.0377 <sup>A</sup> x  | -0.82          |
| FLAV vs FFA        | y = 8.80                                 | - 3.65x                  | -0.82          |
| FLAV vs FOS        | y = 9.13                                 | - 0.276x                 | -0.82          |
| CON vs VIS         | y = 26.8                                 | + 0.159x                 | -0.86          |

Table 35. cont.

- 
- 1: Tests:
    - VPA (total volatile profile).
    - POL (polar component%).
    - TLC (polar component%).
    - ODOR (sensory evaluation of rancid odor intensity).
    - FOS (FoodOil-Sensor reading).
    - FFA (free fatty acid - %oleic acid).
    - VIS (viscosity in centipoise).
    - CON (contact angle measurement).
    - FLAV (sensory evaluation of rancid flavor intensity).
  - 2: Correlation coefficient.
  - 3: Duncan's multiple range test: same letters have no significant difference ( $\alpha = 0.05$ ).

Table 36. Linear regression equations and correlation coefficients among seven tests on soybean oil liquid shortening used under static heating conditions.

| Tests <sup>1</sup> | Linear Regression Equations <sup>3</sup> | r <sup>2</sup> |
|--------------------|--|----------------|
| VPA vs POL         | $y = 2435^B + 139^E x$                   | 0.96           |
| VPA vs TLC         | $y = 2182^A + 88^E x$                    | 0.96           |
| VPA vs VIS         | $y = 3033^B + 40.28^C x$                 | 0.95           |
| VPA vs FOS         | $y = 1708^A + 1070^D x$                  | 0.97           |
| TLC vs FOS         | $y = -4.96^D + 12.12^A x$                | 1.00           |
| TLC vs FFA         | $y = 5.57^E + 304.97^A x$                | 0.93           |
| TLC vs VIS         | $y = -13.25^C + 0.46^C x$                | 0.99           |
| VIS vs FOS         | $y = 19.36^B + 25.81^B x$                | 0.99           |
| VIS vs POL         | $y = 37.03^A + 3.36^B x$                 | 0.98           |
| VIS vs FFA         | $y = 42.32^B + 638.4^B x$                | 0.91           |
| FFA vs VPA         | $y = 2723^C + 25733^B x$                 | 0.87           |
| FFA vs POL         | $y = -0.007^A + 0.004^A x$               | 0.92           |
| FOS vs FFA         | $y = 0.87^C + 24.98^A x$                 | 0.93           |
| FOS vs POL         | $y = 0.68^A + 0.13^C x$                  | 1.00           |
| TLC vs POL         | $y = 2.49^E + 1.60^A x$                  | 1.00           |

<sup>1</sup>: Tests:

- VPA (total volatile profile).
- POL (polar component%).
- TLC (polar component%).
- ODOR (sensory evaluation of rancid odor intensity).
- FOS (FoodOil-Sensor reading).
- FFA (free fatty acid - %oleic acid).
- VIS (viscosity in centipoise).

<sup>2</sup>: Correlation coefficient.

<sup>3</sup>: Duncan's multiple range test: same letters have no significant difference ( $\alpha = 0.05$ ).

Duncan's multiple range test was applied to detect significance ( $\alpha=0.05$ ) between intercepts and slopes for all five frying shortenings. Twenty-four high correlation coefficients and linear regressions for peanut oil are reported in Table 32. Nineteen high correlation coefficients and linear regressions in cottonseed oil liquid shortening and cottonseed oil are reported in Tables 33 and 34, respectively. Fifteen and thirteen linear regressions and correlation coefficients of soybean oil liquid shortening and corn oil are reported in Tables 35 and 36, respectively.

The results indicated that peanut oil, cottonseed oil, cottonseed oil liquid shortening, soybean oil liquid shortening and corn oil had 25, 19, 19, 15 and 13 high correlations between objective and subjective analyses, respectively. Also, these shortenings have the following number of triglycerides: 27, 20, 22, 17 and 14. By coincidence it was found that the number of high correlations is proportional to the number of triglycerides (Tables 22-26). Hence, a high positive correlation between the analytical tests and number of triglycerides of ( $r=0.987$ ), or number of FFA ( $r=0.975$ ) was established with a linear regression equation of  $y = 0.167 + 0.918x$ . From this principle it can be suggested that the linear behavior of a particular oil or fat during subjective and objective

analyses might be predicted from the number of triglycerides or fatty acids.

Since triglycerides and their fatty acids are substrates for many chemical reactions, it is concluded that the higher the number of fatty acids or triglycerides in a particular shortening, the greater is the sensitivity of this shortening to this treatment; hence the more linear is the analytical data. Predictive models may be developed with those types of shortenings. It would be interesting to observe the behavior of peanut oil with the peroxide value and TBA test, because both tests failed to provide good correlation with heating time (Stevenson et al., 1984).

From the ten quality analyses, important relationships can be separated into polar and non-polar fractions. Triglyceride analysis of non-polar fraction would provide a clue about the rate at which individual triglycerides are degraded. Also, this analysis would indicate of any possible exchange of triglycerides with the cooked product. The polar component% as evaluated by open column chromatography (standard method), or by Quantitative High Performance Thin Layer Chromatography (QHPTLC) resulted in an increase in free fatty acid content, dielectric constant, polymers (as evaluated by increases in viscosity), total volatiles or specific volatiles and lower ratings of oil odor and flavor. However, the net balance

between polar and non-polar components would affect the final values of oil contact angles.

#### 4.1.1.12 OTHER OBSERVATIONS FROM THE STATIC HEATING EXPERIMENTS.

As a result of oxidation and polymerization, shortening color changed from light yellow or light green to dark orange.

#### 4.1.2 COMMERCIAL FRYING CONDITIONS ON SHORTENING QUALITY.

##### 4.1.2.1 EFFECT OF CHICKEN NUGGET/FILET FRYING CONDITIONS AND SHORTENING TYPE ON SHORTENING YIELD.

Shortening yield was estimated by dividing the weight (lb) of total raw chicken cooked by the weight (lb) of total shortening used (absorbed) to cook the chicken products, over defined time interval.

Changes in shortening yields for chicken nuggets and filets were investigated after 2, 5 and 7 DAYS of commercial frying conditions using peanut oil (P/O), cottonseed oil liquid shortening (CS/OLS) and soybean oil liquid shortening (SB/OLS) (Tables 37 and 38). From the General Linear Model (GLM) procedure, it was found that

Table 37. Changes in heating time (days), Total Heating Time (THT), Total Frying Time (TFT), Chicken Nugget Weight (CHKW), added shortening weight (OILW) and shortening yield (OILY) during commercial frying with Peanut Oil (P/O), Cottonseed Oil Liquid Shortening (CS/OLS), and Soybean Oil Liquid Shortening (SB/OLS).

| SHORTENING <sup>7,8</sup><br>TYPE | DAYS <sup>6</sup> | THT <sup>1</sup><br>(hr) | TFT <sup>2</sup><br>(hr) | CHKW <sup>3</sup><br>(lb) | OILW <sup>4</sup><br>(lb) | OILY <sup>5</sup> |
|-----------------------------------|-------------------|--------------------------|--------------------------|---------------------------|---------------------------|-------------------|
| P/O                               | 2                 | 19.88                    | 2.11                     | 254.3                     | 36.45                     | 6.98              |
| P/O                               | 5                 | 49.01                    | 4.19                     | 453.8                     | 59.55                     | 7.62              |
| P/O                               | 7                 | 69.23                    | 6.13                     | 651.0                     | 86.00                     | 7.57              |
| CS/OLS                            | 2                 | 19.97                    | 2.13                     | 241.15                    | 24.00                     | 10.05             |
| CS/OLS                            | 5                 | 50.20                    | 5.00                     | 583.90                    | 56.90                     | 10.26             |
| CS/OLS                            | 7                 | 67.58                    | 6.57                     | 749.25                    | 80.05                     | 9.36              |
| SB/OLS                            | 2                 | 19.80                    | 1.67                     | 159.00                    | 32.10                     | 4.95              |
| SB/OLS                            | 5                 | 50.47                    | 4.98                     | 538.50                    | 72.45                     | 7.43              |
| SB/OLS                            | 7                 | 71.68                    | 7.88                     | 926.50                    | 110.70                    | 8.37              |

<sup>1</sup>: Total Heating Time (hr) = the period at which shortening temperature is at 325°F.

<sup>2</sup>: Total Frying Time (hr) = # batches of chicken nuggets x 2.5 min / 60 min/hr.

<sup>3</sup>: Weight=(lb) of chicken nuggets.

<sup>4</sup>: Weight=(lb) of added shortening.

<sup>5</sup>: Shortening yield = total weight (lb) of chicken nuggets / total weight (lb) of shortening used.

<sup>6</sup>: P/O: DAYS significantly ( $P < 0.0342$ ) effect shortening yield. CS/OLS: DAYS had no significant ( $P < 0.2384$ ) effect on shortening yield. SB/OLS: DAYS significantly ( $P < 0.0012$ ) effect shortening yield.

<sup>7</sup>: Significant ( $P < 0.0004$ ) differences among shortenings on shortening yield.

Table 37. cont.

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<sup>8</sup>: Duncan's Multiple Range Test: on the average shortening yields were in the following increasing order CS/OLS> P/O> SB/OLS.



Table 38. Changes in heating time (days), Total Heating Time (THT), Total Frying Time (TFT), Chicken Filet Weight (CHKW), added shortening weight (OILW) and shortening yield (OILY) during commercial frying with Peanut Oil (P/O), Cottonseed Oil Liquid Shortening (CS/OLS), and Soybean Oil Liquid Shortening (SB/OLS).

| SHORTENING <sup>7,8</sup><br>TYPE | DAYS <sup>6</sup> | THT <sup>1</sup><br>(hr) | TFT <sup>2</sup><br>(hr) | CHKW <sup>3</sup><br>(lb) | OILW <sup>4</sup><br>(lb) | OILY <sup>5</sup> |
|-----------------------------------|-------------------|--------------------------|--------------------------|---------------------------|---------------------------|-------------------|
| P/O                               | 2                 | 20.69                    | 3.30                     | 220.30                    | 24.25                     | 9.08              |
| P/O                               | 5                 | 48.53                    | 6.59                     | 399.30                    | 43.05                     | 9.28              |
| P/O                               | 7                 | 69.04                    | 9.68                     | 597.30                    | 62.70                     | 9.53              |
| CS/OLS                            | 2                 | 20.73                    | 3.33                     | 215.80                    | 19.10                     | 11.30             |
| CS/OLS                            | 5                 | 50.87                    | 8.40                     | 525.00                    | 47.45                     | 11.06             |
| CS/OLS                            | 7                 | 68.65                    | 11.11                    | 707.75                    | 68.60                     | 10.32             |
| SB/OLS                            | 2                 | 19.68                    | 2.72                     | 166.05                    | 17.60                     | 9.43              |
| SB/OLS                            | 5                 | 50.37                    | 8.37                     | 510.55                    | 54.95                     | 9.29              |
| SB/OLS                            | 7                 | 72.07                    | 13.59                    | 874.60                    | 79.10                     | 11.06             |

<sup>1</sup>: Total Heating Time (hr) = the period at which shortening temperature is at 325°F.

<sup>2</sup>: Total Frying Time (hr) = # batches of chicken filets x 3.5 min / 60 min/hr.

<sup>3</sup>: Weight (lb) of chicken filets.

<sup>4</sup>: Weight (lb) of added shortening.

<sup>5</sup>: Shortening yield = total weight (lb) of chicken filets / total weight (lb) of shortening used.

<sup>6</sup>: P/O: DAYS significantly ( $P < 0.0025$ ) effect shortening yield. CS/OLS: DAYS significantly ( $P < 0.0281$ ) effect on shortening yield. SB/OLS: DAYS had no significant ( $P < 0.1372$ ) effect shortening yield.

<sup>7</sup>: Significant ( $P < 0.005$ ) differences among shortenings on shortening yield.

Table 38. cont.

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8: Duncan's Multiple Range Test: on the average shortening yields were in the following increasing order CS/OLS > SB/OLS > P/O.

there are significant ( $P < 0.0001$ ,  $0.0001$  and  $0.0245$ ) differences among products, shortening types and heating time, respectively, in their effects on shortening yield. Also, significant ( $0.0142$ ) interaction between shortening type and products were found.

It was also observed that heating time (DAYS) had significant ( $P < 0.0342$  and  $0.0012$ ) effects on the changes in shortening yield for peanut oil and soybean oil liquid shortening, respectively, while no significant ( $P < 0.2384$ ) effect was observed by cottonseed oil liquid shortening under chicken nugget frying conditions. Moreover, under chicken filet frying conditions, heating time (DAYS) HAD significant ( $P < 0.0025$  and  $0.0281$ ) effects on shortening yield with P/O and CS/OLS but no significant ( $P < 0.1372$ ) effect was observed with SB/OLS on shortening yield.

On the average, from Duncan's multiple range test it was observed that shortening yield decreased in the following order: CS/OLS, SB/OLS and P/O under chicken filet frying conditions and CS/OLS, P/O and SB/OLS under chicken nugget frying conditions.

Also, different shortenings were significantly different in their shortening yield under chicken nugget ( $P < 0.0009$ ) and chicken filet ( $P < 0.004$ ) frying conditions. The shortening yield of CS/OLS was significantly higher than that of P/O and SB/OLS, which indicated that CS/OLS was absorbed to lesser extent by the chicken products as

compared to P/O and SB/OLS. In order to confirm that frying methods affect the moisture content of fried chicken products, which accordingly, affect the rate of oil replenishment, hence its stability. Analysis of Variance and Duncan's multiple range test were conducted and it was found that pressure-fried chicken filet samples had a significantly ( $\alpha=0.5$ ) higher (59.35%) moisture content as compared to open-vat fried chicken nugget samples (54.98%) as reported in Table 39. This finding was reported earlier (Mostert and Stadelman, 1964). Nevertheless, it is important to mention that the initial moisture content in battered, breaded and uncooked chicken nuggets is significantly ( $\alpha=0.5$ ) higher (68.22%) than the moisture contents of chicken filets (65.07%). This higher moisture content is due to the higher surface to volume ratio of chicken nuggets as compared to chicken filets, causing a greater batter absorption on the nuggets' surface. As a result of frying, a significant ( $P<0.0001$ ) reduction in moisture contents (from 68.22 to 54.98% and from 65.07 to 59.35%) in both chicken nuggets and chicken filets, respectively occurs. Although more moisture is removed from chicken nuggets than from chicken filets, less moisture is retained in the heating medium at 325<sup>o</sup>F. Therefore, it is anticipated that the method of cooking (pressure vs open-vat) and not the food moisture content affects oil stability during frying.

Table 39. Effect of frying conditions on the moisture content of chicken nuggets and chicken filets fried in peanut oil used to four days.

| PRODUCT             | % MOISTURE <sup>A,B</sup>          |                       |
|---------------------|------------------------------------|-----------------------|
|                     | BEFORE FRYING                      | AFTER FRYING          |
| NUGGET <sup>D</sup> | 68.22 (A) <sup>C</sup><br>(0.2523) | 54.98 (B)<br>(0.1738) |
| FILET <sup>D</sup>  | 65.07 (B)<br>(0.2040)              | 59.98 (A)<br>(0.1866) |

A: Average of four samples.

B: Standard deviations are the values in brackets.

C: Duncan's Multiple Range Test: different letters indicates significance ( $\alpha=0.5$ ; columns).

D: Significant ( $P<0.0001$ ) reduction in moisture content as a result of frying (row).

From this study, it was concluded that cooking chicken filets under pressure retains moisture, absorbs less material from the frying media, and produces a greater shortening yield. Nonpressurized frying of chicken nuggets resulted in opposite effects. These results confirm an earlier study by Mostert and Stadelman (1964) who showed that pressurized deep fat frying of breaded and non-breaded chicken legs and thighs resulted in minimum cooking losses and maximum moisture retention. Therefore, both frying methods and shortening types affect the shortening yield, hence, the turnover rate.

#### 4.1.2.2 EFFECT OF COMMERCIAL FRYING CONDITIONS OF CHICKEN NUGGETS AND FILETS ON THE CHEMICAL, PHYSICAL AND SENSORY PROPERTIES OF PEANUT OIL AND COTTONSEED, AND SOYBEAN OIL LIQUID SHORTENINGS.

The results of eight of the chemical, physical and sensory analyses are summarized in Tables 40 and 41. Peanut oil (P/O), cottonseed oil liquid shortening (CS/OLS) and soybean oil liquid shortening (SB/OLS) were used to fry battered and breaded chicken nuggets under atmospheric pressure (open vessel) at 325°F for 2.5 min per batch and chicken filets under pressurized cooking at 325°F for 3.5 min per batch. Changes in the independent variables, such

Table 40. Effect of commercial frying conditions on the chemical, physical and sensory properties of peanut oil and cottonseed and soybean oil liquid shortenings used to cook chicken nuggets.

| CHEMICAL, PHYSICAL AND SENSORY RESULTS |                   |                  |                  |                  |                  |                  |                  |                  |                   |
|--|-------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|-------------------|
| SHORT <sup>1</sup>                     | DAYS <sup>2</sup> | POL <sup>3</sup> | TLC <sup>4</sup> | FOS <sup>5</sup> | FFA <sup>6</sup> | VIS <sup>7</sup> | CON <sup>8</sup> | ODR <sup>9</sup> | FLA <sup>10</sup> |
| P/O                                    | 0                 | 4.86<br>(0.01)   | 11.79<br>(1.05)  | 1.12<br>(0.00)   | 0.20<br>(0.00)   | 37.10<br>(1.02)  | 42.25<br>(2.20)  | 9.63<br>(2.30)   | 8.40<br>(1.50)    |
| P/O                                    | 2                 | 6.14<br>(0.07)   | 14.84<br>(2.09)  | 1.51<br>(0.01)   | 0.25<br>(0.02)   | 37.87<br>(1.82)  | 43.75<br>(1.80)  | 5.97<br>(0.30)   | 8.14<br>(0.60)    |
| P/O                                    | 5                 | 8.80<br>(0.21)   | 18.27<br>(1.79)  | 1.79<br>(0.08)   | 0.51<br>(0.05)   | 37.63<br>(2.62)  | 45.15<br>(2.40)  | 6.47<br>(1.22)   | 8.70<br>(2.20)    |
| P/O                                    | 7                 | 9.20<br>(0.53)   | 21.14<br>(3.89)  | 2.00<br>(0.18)   | 0.90<br>(0.45)   | 38.06<br>(2.32)  | 47.48<br>(1.40)  | 6.84<br>(0.34)   | 7.57<br>(0.92)    |
| CS/S                                   | 0                 | 9.40<br>(0.02)   | 21.61<br>(2.89)  | 2.64<br>(0.19)   | 0.05<br>(0.55)   | 48.20<br>(3.82)  | 39.35<br>(2.40)  | 9.74<br>(1.24)   | 8.63<br>(1.01)    |
| CS/S                                   | 2                 | 11.89<br>(0.07)  | 22.29<br>(2.21)  | 2.66<br>(0.02)   | 0.31<br>(1.05)   | 45.37<br>(2.33)  | 39.40<br>(1.87)  | 6.73<br>(1.83)   | 8.10<br>(0.47)    |
| CS/S                                   | 5                 | 12.71<br>(1.10)  | 23.39<br>(4.28)  | 3.00<br>(0.01)   | 0.52<br>(1.15)   | 47.49<br>(3.53)  | 40.03<br>(2.19)  | 6.37<br>(0.34)   | 7.53<br>(1.55)    |

cont.

Table 40. cont.

|      |   |                 |                 |                |                |                 |                 |                |                |
|------|---|-----------------|-----------------|----------------|----------------|-----------------|-----------------|----------------|----------------|
| CS/S | 7 | 13.05<br>(1.08) | 24.80<br>(2.84) | 3.29<br>(0.05) | 0.73<br>(0.79) | 45.92<br>(2.98) | 40.88<br>(1.88) | 6.37<br>(1.35) | 8.17<br>(1.95) |
| SB/S | 0 | 4.56<br>(0.02)  | 15.41<br>(1.89) | 1.26<br>(0.66) | 0.08<br>(0.86) | 50.83<br>(2.58) | 40.58<br>(2.30) | 8.80<br>(0.24) | 8.87<br>(1.01) |
| SB/S | 2 | 6.40<br>(1.11)  | 16.33<br>(2.19) | 1.68<br>(1.65) | 0.26<br>(0.05) | 56.86<br>(2.62) | 40.33<br>(3.40) | 5.77<br>(1.22) | 7.97<br>(1.20) |
| SB/S | 5 | 8.39<br>(0.21)  | 17.26<br>(1.79) | 2.00<br>(0.08) | 0.51<br>(0.05) | 53.25<br>(1.33) | 40.03<br>(2.21) | 6.10<br>(2.70) | 8.10<br>(2.01) |
| SB/S | 7 | 9.01<br>(0.53)  | 20.64<br>(2.89) | 5.89<br>(0.18) | 0.94<br>(0.45) | 52.52<br>(1.32) | 43.30<br>(1.40) | 6.00<br>(0.34) | 8.20<br>(0.92) |

1 : Shortenings: are peanut oil and cottonseed and soybean oil liquid shortenings.

2 : One working day averages 9.9 hr.

3 : Polar component%.

4 : Polar component% measured by quantitative high performance thin layer chromatography.

5 : FoodOil-Sensor readings (dielectric constants).

6 : Free fatty acid (%oleic acid).

7 : Viscosity (CP) determined at 40°C.



Table 40. cont.

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- 8 : Contact angle of the shortenings at  $28.5 \pm 0.5$  °C measured on FT-120-WW surface.
- 9 : Rancid odor evaluation at room temperature.  
Scale: 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.
- 10 : Rancid flavor evaluation of chicken nuggets cooked in commercially used shortenings. Same scale as in 9.

Table 41. Effect of commercial frying conditions on the chemical, physical and sensory properties of peanut oil, cottonseed and soybean oil liquid shortenings used to cook chicken filets.

| CHEMICAL, PHYSICAL AND SENSORY RESULTS |                   |                  |                  |                  |                  |                  |                  |                  |                   |
|--|-------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|-------------------|
| SHORT <sup>1</sup>                     | DAYS <sup>2</sup> | POL <sup>3</sup> | TLC <sup>4</sup> | FOS <sup>5</sup> | FFA <sup>6</sup> | VIS <sup>7</sup> | CON <sup>8</sup> | ODR <sup>9</sup> | FLA <sup>10</sup> |
| P/O                                    | 0                 | 4.88<br>(0.73)   | 11.81<br>(3.29)  | 1.12<br>(0.48)   | 0.20<br>(0.75)   | 37.91<br>(2.42)  | 42.63<br>(1.46)  | 9.47<br>(0.84)   | 8.50<br>(0.32)    |
| P/O                                    | 2                 | 6.45<br>(0.61)   | 17.84<br>(1.49)  | 1.71<br>(0.07)   | 0.32<br>(0.04)   | 37.60<br>(2.22)  | 44.68<br>(2.80)  | 5.73<br>(1.24)   | 8.17<br>(2.30)    |
| P/O                                    | 5                 | 10.71<br>(0.04)  | 22.77<br>(2.04)  | 2.06<br>(0.02)   | 0.90<br>(0.03)   | 37.72<br>(1.52)  | 46.73<br>(1.60)  | 6.40<br>(0.30)   | 7.74<br>(0.70)    |
| P/O                                    | 7                 | 12.96<br>(0.02)  | 26.46<br>(1.03)  | 2.45<br>(0.01)   | 1.87<br>(0.01)   | 38.50<br>(1.00)  | 48.03<br>(2.10)  | 6.90<br>(2.20)   | 7.84<br>(1.71)    |
| CS/S                                   | 0                 | 9.40<br>(0.37)   | 22.99<br>(2.51)  | 2.64<br>(0.02)   | 0.05<br>(0.05)   | 48.20<br>(3.01)  | 39.98<br>(1.67)  | 9.33<br>(1.73)   | 8.70<br>(0.57)    |
| CS/S                                   | 2                 | 13.10<br>(0.06)  | 24.08<br>(2.29)  | 2.95<br>(0.29)   | 0.38<br>(0.35)   | 47.21<br>(2.82)  | 36.83<br>(1.40)  | 5.60<br>(1.54)   | 7.67<br>(1.31)    |

cont.

Table 41. cont.

|      |   |                 |                 |                |                |                 |                 |                |                |
|------|---|-----------------|-----------------|----------------|----------------|-----------------|-----------------|----------------|----------------|
| CS/S | 5 | 14.35<br>(1.12) | 29.01<br>(1.28) | 3.41<br>(0.10) | 1.08<br>(1.65) | 47.23<br>(2.53) | 38.80<br>(2.38) | 5.77<br>(0.54) | 7.47<br>(1.25) |
| CS/S | 7 | 17.07<br>(1.06) | 31.93<br>(1.84) | 3.68<br>(0.45) | 1.87<br>(0.29) | 45.39<br>(1.98) | 42.00<br>(1.78) | 7.13<br>(1.55) | 7.47<br>(1.35) |
| SB/S | 0 | 4.56<br>(0.22)  | 15.44<br>(1.69) | 1.26<br>(0.05) | 0.08<br>(0.07) | 50.83<br>(1.53) | 40.55<br>(2.11) | 8.53<br>(2.80) | 8.63<br>(2.06) |
| SB/S | 2 | 7.00<br>(0.57)  | 19.65<br>(2.69) | 1.62<br>(0.48) | 0.34<br>(0.25) | 55.87<br>(1.42) | 40.10<br>(1.60) | 6.23<br>(0.24) | 7.74<br>(0.12) |
| SB/S | 5 | 10.14<br>(1.12) | 21.58<br>(2.29) | 2.16<br>(1.55) | 1.15<br>(0.15) | 55.89<br>(1.62) | 39.95<br>(2.40) | 6.14<br>(1.62) | 7.84<br>(1.27) |
| SB/S | 7 | 13.47<br>(0.02) | 25.98<br>(1.49) | 2.70<br>(0.65) | 2.27<br>(0.66) | 50.40<br>(2.88) | 42.55<br>(2.33) | 7.13<br>(0.27) | 7.87<br>(1.04) |

1 : Shortenings: are peanut oil, cottonseed and soybean oil liquid shortenings.

2 : One working day averages 9.9 hr.

3 : Polar component%.

4 : Polar component measured by quantitative high performance thin layer chromatography.

5 : Food-Oil sensor readings (dielectric constants).

Table 41. cont.

- 
- 6 : Free fatty acid (% oleic acid).
- 7 : Viscosity (CP) determined at 40°C.
- 8 : Contact angle of the shortenings at  $28.5 \pm 0.5$  °C measured on FT-120-WW surface.
- 9 : Rancid odor evaluation at room temperature.  
Scale: 10: bland; 9 and 8: slightly rancid; 7 and 6: moderately rancid; 5, 4 and 3: strongly rancid; and 2 and 1: extremely rancid.
- 10 : Rancid flavor evaluation of chicken filets cooked in commercially used shortenings. Same scale as in 9.

as heating time (DAYS), Total Heating Time (THT) is defined as the total hours the temperature of frying shortening is kept at 325°F, Total Frying Time (TFT) is defined as number of batches of chicken product X cooking period (2.5 minutes for chicken nuggets and 3.5 minutes for chicken filets), weight (lb) of chicken nuggets (CHKW) or filets and weight (lb) of added shortening (OILW) are summarized in Tables 38 and 39.

#### 4.1.2.2.1 FREE FATTY ACID-% Oleic Acid (FFA).

The FFA of unheated and commercially used P/O, CS/OLS and SB/OLS were reported in Table 40 for chicken nugget and Table 41 for chicken filet frying conditions. Following the GLM procedure, it was found that shortening type (SHORT.), heating time (DAYS), Total Heating Time (THT) and weight (lb) of added shortenings had significant effect on the increase in FFA content (Table 42 and Fig. 22), however, TFT, CHKW, and DAYS x SHORT. had no significant effects on increasing FFA content.

When the FFA content was measured for the same shortenings, but under chicken filet frying conditions, it was found that DAYS and THT had significant (Table 42 and Fig. 22) effects on the increase in FFA%.

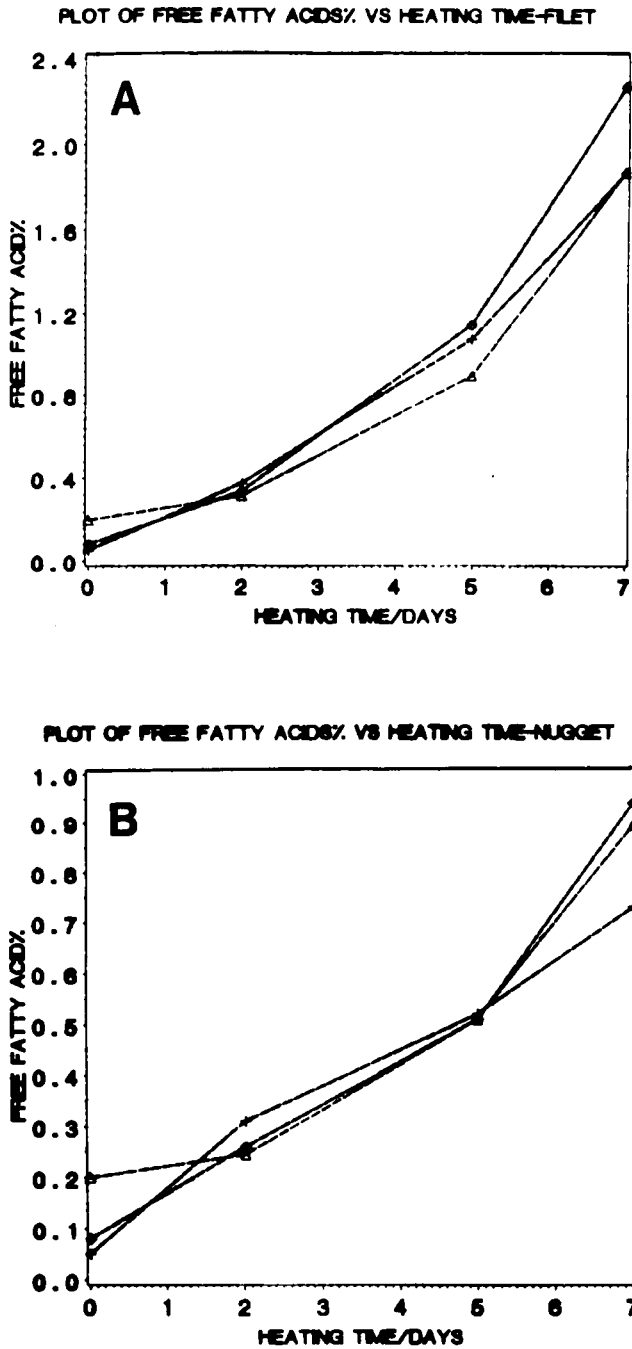


Fig. 22. Effect of Heating Time (Days) on Free Fatty Acid-(%Oleate) of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

Table 42. Effect of commercial frying conditions of chicken nuggets and filets on the chemical, physical and sensory properties of Peanut Oil (P/O), Cottonseed (CS/OLS), and Soybean Oil Liquid Shortening (SB/OLS) using General Linear Models (GLM) procedure<sup>20</sup>.

|                     | SHORT <sup>3</sup> | DAYS <sup>4</sup> | THT <sup>5</sup> | TFT <sup>6</sup> | CHKW <sup>7</sup> | OILW <sup>8</sup> | DAYxSHORT <sup>9</sup> |
|---------------------|--------------------|-------------------|------------------|------------------|-------------------|-------------------|------------------------|
| POL <sup>12</sup>   | 0.0001             | 0.0002            | 0.419            | 0.0006           | 0.004             | 0.481             | 0.0001                 |
| NU <sup>10</sup>    | 0.0001             | 0.0002            | 0.419            | 0.0006           | 0.004             | 0.481             | 0.0001                 |
| FL <sup>11</sup>    | 0.0001             | 0.547             | 0.103            | 0.413            | 0.616             | 0.066             | 0.0009                 |
| TLC <sup>1,13</sup> | 0.0001             | 0.171             | 0.042            | 0.0003           | 0.0002            | 0.020             | 0.0016                 |
| NU                  | 0.0001             | 0.280             | 0.454            | 0.870            | 0.971             | 0.302             | 0.224                  |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| FOS <sup>14</sup>   | 0.0001             | 0.0001            | 0.959            | 0.110            | 0.010             | 0.200             | 0.0001                 |
| NU                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| FFA <sup>15</sup>   | 0.0001             | 0.0001            | 0.0001           | 0.8992           | 0.6244            | 0.0001            | 0.639                  |
| NU                  | 0.4506             | 0.0001            | 0.0001           | 0.7788           | 0.8749            | 0.1533            | 0.963                  |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| VIS <sup>16</sup>   | 0.0001             | 0.0001            | 0.107            | 0.187            | 0.060             | 0.0001            | 0.0001                 |
| NU                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| CON <sup>2,17</sup> | 0.628              | 0.0002            | 0.0006           | 0.002            | 0.0002            | 0.917             | 0.121                  |
| NU                  | 0.838              | 0.0001            | 0.307            | 0.111            | 0.086             | 0.0001            | 0.001                  |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |
| ODOR <sup>18</sup>  | 0.0001             | 0.0001            | 0.060            | 0.140            | 0.005             | 0.0001            | 0.0001                 |
| NU                  | 0.057              | 0.0001            | 0.006            | 0.034            | 0.005             | 0.062             | 0.0001                 |
| FL                  | 0.0001             | 0.0001            | 0.0001           | 0.0001           | 0.0001            | 0.0001            | 0.0001                 |

cont.

Table 42. cont.

|                    |    |       |        |        |       |        |        |        |
|--------------------|----|-------|--------|--------|-------|--------|--------|--------|
| FLAV <sup>19</sup> | NU | 0.986 | 0.0007 | 0.637  | 0.015 | 0.0001 | 0.0003 | 0.0001 |
|                    | FL | 0.031 | 0.001  | 0.0008 | 0.075 | 0.399  | 0.097  | 0.0001 |

1: (QHPTLC) plate: significance for nuggets ( $p < 0.010$ ) and filets ( $p < 0.0001$ ), (QHPTLC) plate x shortening type for nuggets ( $p < 0.646$ ) and for filets ( $p < 0.432$ ).

2: Significance of pellet surface on contact angles for nuggets ( $p < 0.649$ ) and filets ( $p < 0.997$ ), pellet x shortening type for nuggets ( $p < 0.362$ ) and filets ( $p < 0.719$ ).

3: Shortening type effect.

4: Heating time (Days) effect.

5: Total heating time (hr) effect.

6: Total frying time (hr) effect.

7: Chicken nugget or filet weight (lb) effect.

8: Weight of added frying shortening (lb) effect.

9: Interaction between heating time (Days) and shortening type.

10: Chicken nugget.

11: Chicken filet.

12: Polar component%.



Table 42. cont.

- 
- 13 : Polar component% as determined by QHPTLC.
  - 14 : FoodOil-Sensor reading.
  - 15 : Free Fatty Acid - %Oleic acid.
  - 16 : Viscosity in centipoise at 40°C.
  - 17 : Contact angle measurements of three types of shortenings at  $28.5 \pm 0.5$  °C.
  - 18 : Sensory analysis of rancid odor intensity.
  - 19 : Sensory analysis of rancid flavor intensity.
  - 20 : Test of significance.

The higher FFA content produced under commercial frying conditions is probably due to the presence of moisture emanating from the chicken portions. The availability of water molecules in the frying shortening, supplies triglycerides with substrate needed for their hydrolysis. Therefore free fatty acids (Mistry and Min, 1987) and mono- and diglycerides (Fritsch, 1981) are released in the frying media influencing shortening stability. However, a greater accumulation of FFA in shortenings used to fry chicken filets was observed and is probably due to:

1. Pressure cooking of chicken filets would result in maintaining a higher moisture content in the filets and frying shortening.

2. Chicken filets are cooked for 3.5 minutes per batch at 325°F, however the cooking time for chicken nuggets is 2.5 minutes at the same temperature.

The application of the FFA test on commercially used shortening has one advantage. The presence of food moisture in the shortening does not interfere with the FFA titration value as it does with the dielectric constant test.

Probably the FFA test would remain as one of the better analytical procedure to evaluate frying oil stability in the food industry.

#### 4.1.2.2.2 POLAR COMPONENT%.

Polar component% (POL) in the unheated and used frying shortenings (Fig. 23) were reported in Table 40 for chicken nugget frying conditions, and Table 41 for chicken filet frying conditions. The results from the General Linear Model (GLM) procedure were reported in Table 42.

Shortening type (SHORT.), heating time in days (DAYS), Total Frying Time (TFT) and weight (lb) of uncooked chicken products (CHKW), significantly ( $P < 0.0001$ ,  $0.0002$ ,  $0.0006$  and  $0.004$ , respectively) affected the increase in polar component% under chicken nuggets frying conditions for all three shortenings (Fig. 23 and Tables 40 and 42).

Moreover, the significant ( $P < 0.0001$ ) interaction between heating time (DAYS) and shortening types indicated that different shortenings have different polar component% at different heating times. A similar finding ( $P < 0.0024$ ) was found under static heating conditions. No significant ( $P < 0.419$ , and  $0.481$ ) effects were found from THT and OILW on changes in polar component%. The initial and the final polar component% for CS/OLS were significantly ( $\alpha = 0.05$ ) higher than the P/O and SB/OLS as determined by Duncan's test. From the General Linear Model (GLM) procedure, it was found that shortening type significantly ( $P < 0.0001$ ) affect changes in polar component% (Table 42). The GLM procedure also indicated that DAYS, THT (hr), TFT (hr),

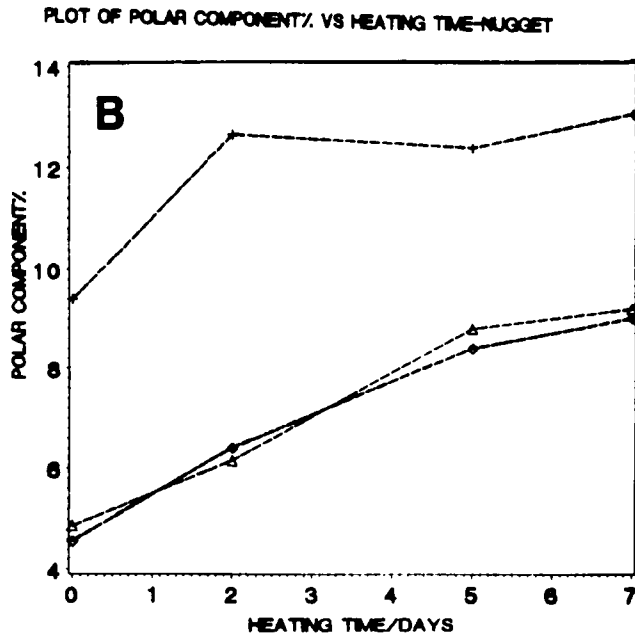
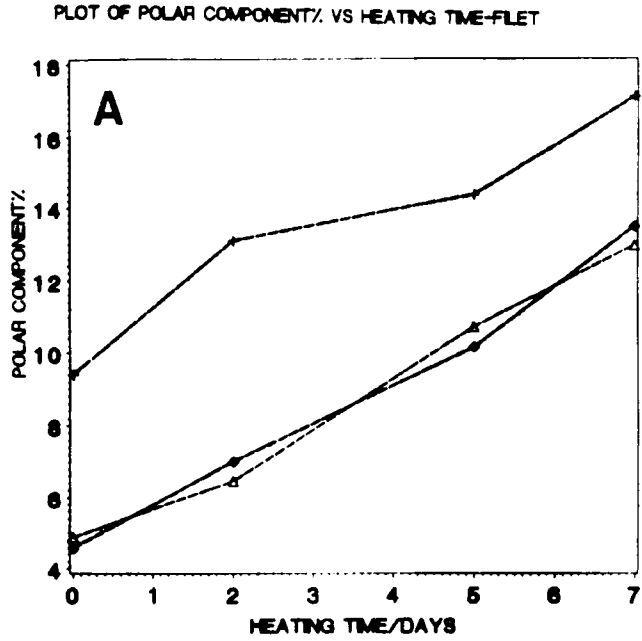


Fig. 23. Effect of Heating Time (Days) on Polar Component% of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

CHKW (lb) and OILW (lb) have no significant ( $P < 0.547$ , 0.103, 0.413, 0.616, 0.066, respectively) effect on polar component% under chicken filet frying conditions. A significant ( $P < 0.0009$ ) interaction between DAYS x SHORT. indicated that different shortenings have different polar component% (stability) at different heating times (DAYS).

Under the commercial frying conditions, the final polar component contents of all shortenings were unexpectedly lower after seven working days than those obtained under static heating conditions, because shortenings are usually abused more in the presence of food. Furthermore, less polar component% accumulated under chicken nugget frying conditions. The possible reasons for these lower values under commercial conditions are as follows:

1. Some of these polar components are carried away with the cooked chicken portions thereby reducing their presence in the frying media. This mechanism is probably more effective with chicken nuggets (Table 40) than chicken filets (Table 41), because, nuggets have a higher surface to volume ratio. Also, open vessel cooking allows more nugget moisture displacement with the frying shortening. The replenishment of frying shortening serves an effective role as a dilution factor particularly with chicken nugget frying conditions.

2. Under static heating conditions shortenings were heated at higher temperature, 360°F, while under commercial frying conditions shortenings were heated at a lower temperature, 325°F.

3. Gum buildup on the vessel and the shortening and air interface may enhance further removal of polar polymers from the frying media. When a basket of uncooked chicken portions is lowered into the frying shortening, the shortening level will rise to cover the gum buildup region. Consequently, more polar or nonpolar polymers are removed from the cooking shortening. At the end of the frying period, the basket is lifted out of the hot shortening allowing the liquid to return to its original level. Meanwhile, the gum is exposed to additional air and heat permitting polymer fixation (baking) on the surface and forming a thicker gum region. Subsequent cooking process repetitions would probably have a limited positive filtering effect on the cooking media.

It would be interesting to replace the stainless steel liner with teflon at the gum region because teflon has a very low surface free energy when compared to stainless steel. Consequently, less polymers would stick to the steel and more polymers would accumulate in the shortening because less would be adsorbed on the teflon surface. Nevertheless, the opposite effect of steel should not be excluded.

4. During cooking a blanket of steam produced from the chicken portions would reduce the oxygen contact with the shortening. Nevertheless, the moisture content of the shortening would be sufficiently high to reduce the rate of oxidative deterioration, but would probably enhance the hydrolytic deterioration.

In general the polar component percentages of cottonseed oil liquid shortening were relatively higher than those of both soybean oil liquid shortening and peanut oil. However, this difference was less observed under the static heating conditions even though statically heated oils accumulated more polar component as previously reported.

#### 4.1.2.2.3 VOLATILE PROFILE ANALYSIS-GAS CHROMATOGRAPHIC METHOD.

This test was not conducted under these conditions.

#### 4.1.2.2.4 DIELECTRIC CONSTANT-FOODOIL-SENSOR READING (FOS).

FOS readings of unheated and commercially used P/O, CS/OLS and SB/OLS were reported in Table 40 for chicken nugget and Table 41 for chicken filet frying conditions.

From the GLM procedure, it was found that shortening type (SHORT.), heating time (DAYS), Total Heating Time (THT), Total Frying Time (TFT), weight (lb) of chicken product (CHKW), weight (lb) of added shortening (OILW) and DAYS x SHORT. were all significant (Fig. 24 and Table 42). FOS readings increased significantly for shortening used to fry chicken filets by SHORT., DAYS, CHKW and (DAYS x SHORT.) (Fig. 24). However, THT, TFT and OILW had no significant effect on changes in FOS reading. One of the more important precautions in the measurement of dielectric constant is to obtain moisture free samples because moisture produces spurious results (Paradis and Nawar, 1981a,b). Therefore, shortening samples were collected in the morning before frying was started. Since 0.00 test oil was used to zero the instrument instead of unheated shortening, therefore the readings obtained for unheated samples are more related to the polar component content of the original frying shortenings. Furthermore, any quality defect in the unheated shortening can be detected with the instrument.

If changes in dielectric constants instead of absolute values were needed for a particular analysis, then the differences between FOS readings could be used. Therefore, the modification has its advantages over the conventional method.



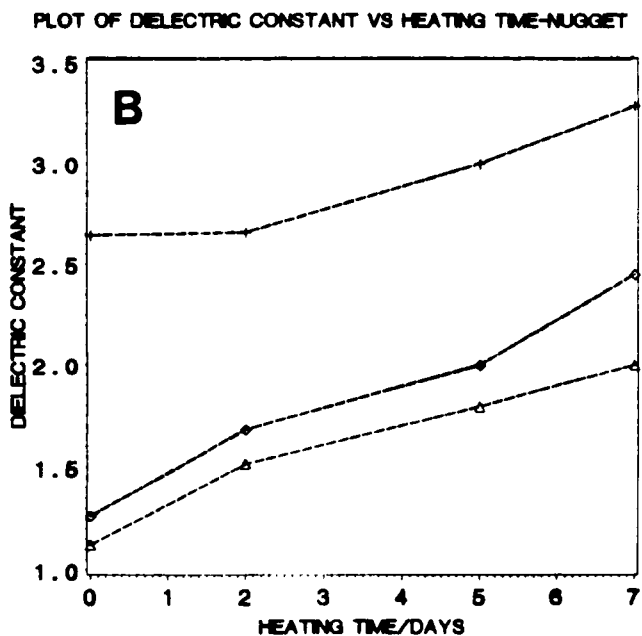
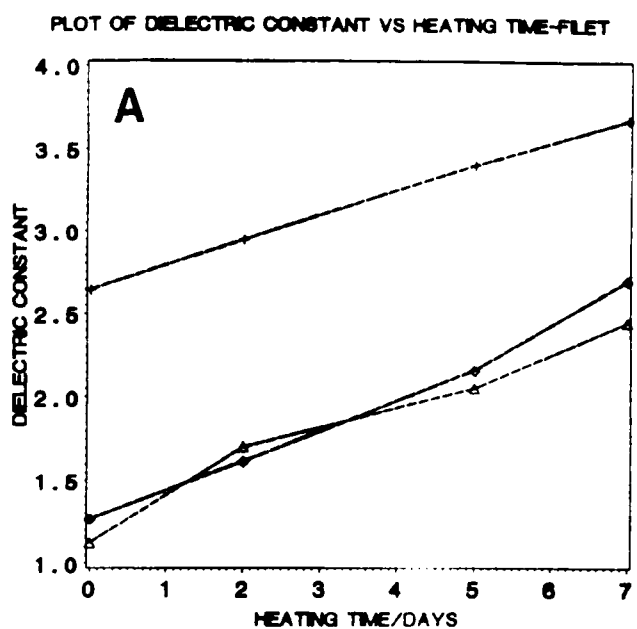


Fig. 24. Effect of Heating Time (Days) on Dielectric constants of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

#### 4.1.2.2.5 VISCOSITY.

The viscosity measurements of unheated and commercially used frying shortenings in centipoise (cp) were conducted at 40°C and presented in Table 40 for chicken nugget and Table 41 for chicken filet frying conditions. From the GLM procedure, it was found that all the independent variables, shortening type (SHORT.), heating time (DAYS), Total Heating Time (THT), Total Frying Time (TFT), weight (lb) of chicken product (CHKW), and weight (lb) OILW had significant ( $P < 0.0001$ ) effect on the changes in viscosity of all shortenings (Fig. 25 and Table 42). Moreover, a significant interaction ( $P < 0.0001$ ) of DAYS x SHORT. was found for all three shortenings. When P/O, CS/OLS, and SB/OLS were used to fry chicken filets, the changes in viscosity of these shortenings were similar to those shortenings used for chicken nuggets preparation. Using the GLM procedure, it was found that the independent variables SHORT., DAYS, and OILW had a significant ( $P < 0.000$ ) effect on changes in viscosity. However, THT, TFT, and CHKW had no significant effect on its measurement. Furthermore, a significant interaction ( $P < 0.0001$ ) was found for DAYS x SHORT. which indicated that different shortenings had different viscosities at different heating times.

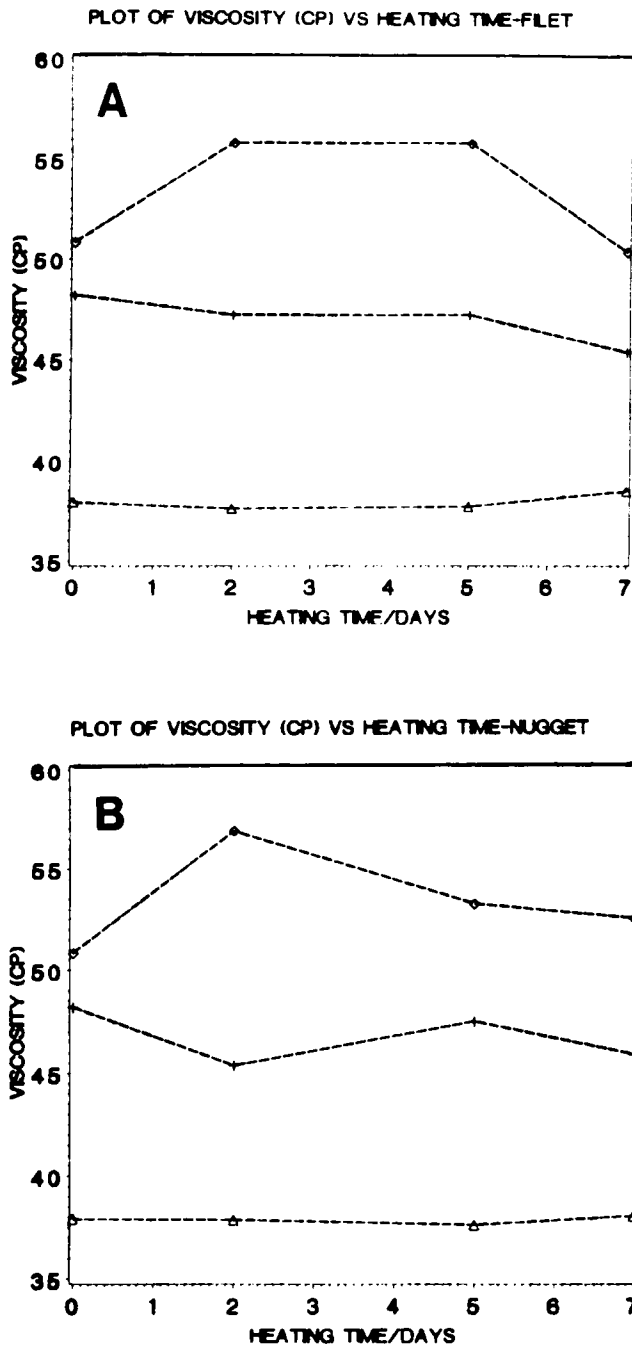


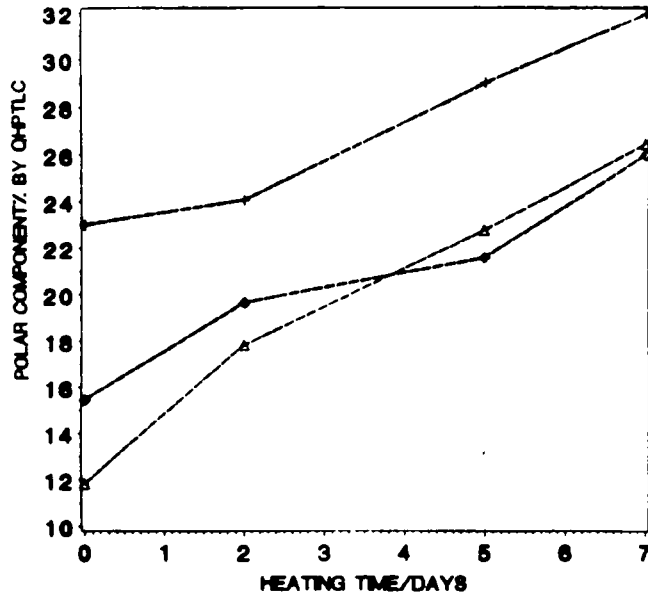
Fig. 25. Effect of Heating Time (Days) on Viscosity (CP) of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

Since changes in viscosity were not linear and the differences between the initial and final viscosity were not significant ( $\alpha=0.05$ ), therefore this test cannot be accepted to evaluate the quality of P/O, CS/OLS and SB/OLS under commercial frying conditions. From these results, it is suggested that the increase in FOS reading is due to the increase in polar component percent excluding polymers. Furthermore, the increase in dielectric constant values is probably due to the accumulation of smaller weight molecules. However, it is unknown whether these molecules are volatiles, non-volatiles or both.

#### 4.1.2.2.6 POLAR COMPONENT% BY QUANTITATIVE HIGH PERFORMANCE THIN LAYER CHROMATOGRAPHY (QHPTLC).

Polar component% as determined by QHPTLC for unheated and commercially used P/O, CS/OLS and SB/OLS were reported in Table 40 for chicken nugget and Table 41 for chicken filet frying conditions. From the GLM procedure, it was found that shortening type (SHORT.), Total Heating Time (THT), Total Frying Time (TFT), weight (lb) of chicken products (CHKW), weight (lb) of added oil (OILW) and QHPTLC plates, significantly affect the increase in polar component% of fried chicken nuggets (Tables 40, 42 and Fig. 26). A significant interaction ( $P<0.0016$ ) was found

PLOT OF POLAR COMPONENT% BY QHPTLC VS HEATING TIME-FLET



PLOT OF POLAR COMPONENT% BY QHPTLC VS HEATING TIME-NUGGET

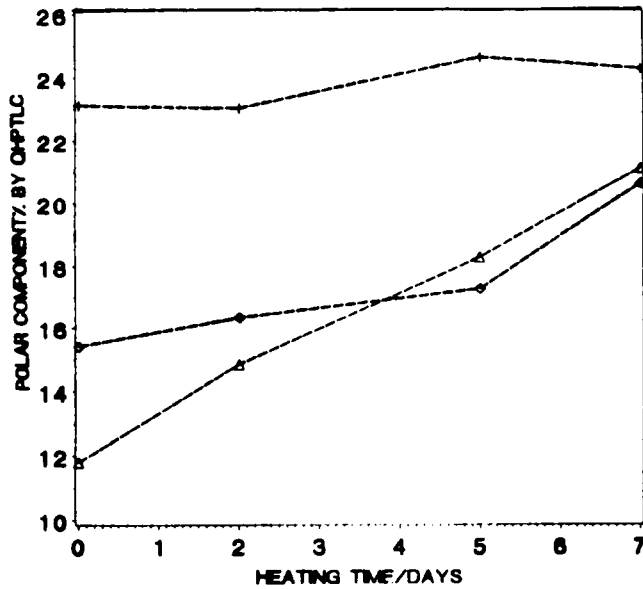


Fig. 26. Effect of Heating Time (Days) on Polar Component% by QHPTLC of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

between DAYS and shortening type which indicated that different shortenings had different polarities at different heating DAYS. Even though heating DAYS had no significant ( $P < 0.171$ ) effect on polar component% by the QHPTLC method. No significant ( $P < 0.646$ ) interaction between the QHPTLC plates and shortening type was found.

Polar component% measured by the QHPTLC procedure for shortenings used to fry chicken filets is reported in Table 41 with the test of significance in Table 42. Both shortening and individual QHPTLC plates had significant ( $P < 0.0001$ ) effects on changes in polar component%, however no significant effects were observed from DAYS, THT, TFT, CHKW and OILW. These results are identical to the results obtained by the polar component test. No significant ( $P < 0.432$ ) interaction was found between plates and shortening types because individual plates have the same effect on all shortening types used. The QHPTLC method requires less time and reagents when compared to the standard polar component test, however, the technique requires expensive instrumentation and some training.

#### 4.1.2.2.7 CONTACT ANGLE.

The contact angles (con) of unheated and commercially used P/O, CS/OLS, and SB/OLS were reported in Table 40 for

chicken nuggets and Table 41 for chicken filet frying conditions. From the GLM procedure, it was found that the independent variables heating time (DAYS), Total Heating Time (THT), Total Frying Time (TFT), and weight (lb) of chicken product (CHKW) had a significant effect on the changes in contact angles of the frying shortening as measured on the FT-120-WW surface (Fig. 27). Also a significant ( $P < 0.0001$ ) interaction occurred between DAYS and shortening type. From this study, no significant ( $P < 0.646$ ) interaction between pellet and shortening type occurred, indicating that different shortenings would probably provide the same contact angle on different pellets.

Under the chicken filet cooking conditions using the GLM procedure, it was found that the independent variables DAYS and OILW had a significant effect on the contact angles of shortenings on the FT-120-WW surface. However, pellet, SHORT., THT, TFT, and CHKW had no significant effect on changes in contact angle measurement. A significant interaction between DAYS and shortenings was found, however no significant ( $P < 0.719$ ) interaction between pellet and shortening type was found, which indicated that different shortenings had identical effects on contact angle measurements on different pellets.

Application of the contact angle test under commercial frying conditions was slightly more successful than its

application under static heating conditions. When shortenings were compared under static heating conditions, peanut oil was the only shortening to show more linearity and the final contact angle of fourteen-day heated sample was higher than the original value of the unheated sample. Under commercial frying conditions, the contact angles of peanut oil manifested excellent linearity and the difference between fresh and seven-day heated samples was significantly noticeable (Fig. 27). Also, when cottonseed oil liquid shortening was used to fry chicken nuggets, its contact angles were significantly linear, however the difference between contact angles of unheated and heated samples were less than the differences in peanut oil.

These results indicated that the frying shortening type plays major role in determining the success of contact angle measurement under the specified conditions. The test requires simple instrumentation and training, and the measurement takes only five minutes to evaluate frying oil quality.

The application of a specific electromagnetic field on the sessile drop before and during the measurement may provide a solution to the non linear behavior of the frying shortening. Because, the electromagnetic field would control the polarity at the liquid solid interface, which might provide more stable measurement. Furthermore, the preparation of a new mixture between mono-, distearin,



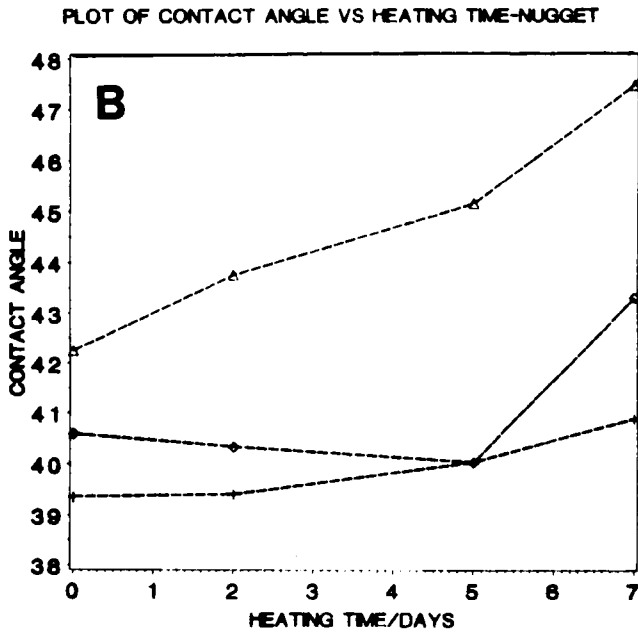
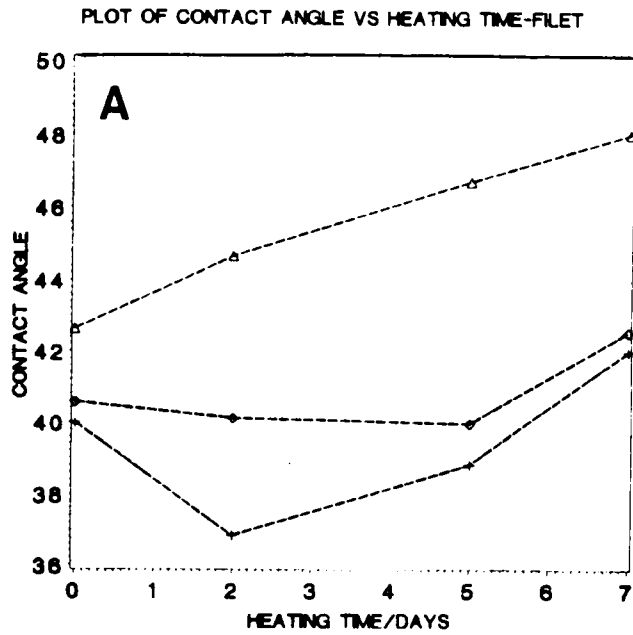


Fig. 27. Effect of Heating Time (Days) on Contact Angle Measurements of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets.

calcium stearate and polypropylene, may produce more efficient measurements of contact angle.

#### 4.1.2.2.8 HIGH TEMPERATURE GAS CHROMATOGRAPHIC ANALYSES TRIGLYCERIDES.

##### 4.1.2.2.8.1 EFFECT OF COMMERCIAL FRYING CONDITIONS ON TRIGLYCERIDES IN PEANUT OIL.

###### 4.1.2.2.8.1.1 Effect of Commercial Chicken Nuggets Frying conditions on Triglycerides in Peanut Oil (P/O).

Thirty triglyceride peaks were separated from unheated commercial brand peanut oil in less than 14 minutes (Table 43), and Fig. 35 in Appendix H). Also see Appendix A for names. In general, heating times had no significant ( $P < 0.9963$ ) effects on the changes in triglyceride profile. The major triglycerides in unheated peanut oil were POO, PLS, PLO, PLL, SOO, SLS + OOO, SLO, UNKNOWN<sub>2</sub>, OLO + SLL and OLL. Under the chicken nugget frying conditions PLS, UNKNOWN<sub>1</sub>, and UNKNOWN<sub>2</sub> were decreased in concentration to an undetected amount. Significance was determined using the General Linear Model (GLM) procedure. Total Frying Time (TFT) had no significant effect on triglyceride (peak

Table 43. Effect of Commercial Chicken Nugget Frying Time (Days) on Triglycerides in Peanut Oil.

| Triglyceride Type    | Triglycerides (Peak Area) <sup>SD,A</sup> |                |                |                |                |       |       |       |
|----------------------|---|----------------|----------------|----------------|----------------|-------|-------|-------|
|                      | t <sub>R</sub>                            | 0              | 2              | 5              | 7              | +D%   | S1    | S2    |
| POP                  | 4.03                                      | 4559<br>(58)   | 7461<br>(39)   | 7602<br>(60)   | 7455<br>(32)   | +63.5 | 0.033 | 0.266 |
| PLP                  | 4.22                                      | 5533<br>(82)   | 8484<br>(75)   | 8880<br>(24)   | 8667<br>(100)  | +56.6 | 0.127 | 0.567 |
| POS                  | 5.05                                      | 8540<br>(23)   | 3676<br>(13)   | 2871<br>(17)   | 3007<br>(76)   | -64.8 | 0.066 | 0.505 |
| POO                  | 5.24                                      | 28855<br>(182) | 38786<br>(485) | 40242<br>(14)  | 38967<br>(330) | +35.0 | 0.050 | 0.777 |
| PLS                  | 5.48                                      | 10347<br>(169) | ND<br>-        | ND<br>-        | ND<br>-        | -100  | -     | -     |
| PLO                  | 5.52                                      | 36156<br>(165) | 42728<br>(205) | 45270<br>(283) | 43892<br>(124) | +21.4 | 0.412 | 0.766 |
| UNKNOWN <sub>1</sub> | 5.67                                      | 2432<br>(84)   | ND<br>-        | ND<br>-        | ND<br>-        | -100  | -     | -     |

cont.

Table 43. cont.

|                      |      |                |                |                |                |       |       |       |
|----------------------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| PLL                  | 5.73 | 12673<br>(42)  | 13878<br>(140) | 16379<br>(124) | 15761<br>(171) | +24.4 | 0.653 | 0.407 |
| POA+SOS              | 6.30 | 1610<br>(45)   | 2170<br>(108)  | 1523<br>(118)  | 2087<br>(36)   | +29.6 | 0.716 | 0.562 |
| S00                  | 6.50 | 21653<br>(97)  | 10824<br>(199) | 9436<br>(40)   | 9657<br>(16)   | -55.4 | 0.046 | 0.552 |
| SLS+000              | 6.86 | 51374<br>(121) | 58669<br>(375) | 60985<br>(149) | 59504<br>(123) | +15.8 | 0.045 | 0.786 |
| SLO                  | 6.94 | 11988<br>(139) | 12403<br>(209) | 10737<br>(941) | 11262<br>(192) | - 6.1 | 0.842 | 0.865 |
| UNKNOWN <sub>2</sub> | 7.10 | 13074<br>(296) | ND<br>-        | ND<br>-        | ND<br>-        | -100  | -     | -     |
| OLO+SLL              | 7.22 | 62771<br>(129) | 74212<br>(448) | 76232<br>(185) | 74242<br>(188) | +18.3 | 0.049 | 0.841 |
| OLL                  | 7.53 | 45576<br>(199) | 43336<br>(57)  | 44698<br>(196) | 43650<br>(390) | - 4.2 | 0.431 | 0.834 |
| LLL                  | 7.80 | 8087<br>(25)   | 8346<br>(22)   | 8235<br>(50)   | 8394<br>(25)   | + 3.8 | 0.880 | 0.562 |
| POBe                 | 8.05 | 2857<br>(72)   | 3661<br>(32)   | 3312<br>(25)   | 3164<br>(78)   | +10.8 | 0.286 | 0.707 |

cont.

Table 43. cont.

|           |       |              |              |              |              |       |       |       |
|-----------|-------|--------------|--------------|--------------|--------------|-------|-------|-------|
| AOO       | 8.26  | 4021<br>(40) | 3206<br>(27) | 3369<br>(39) | 3470<br>(67) | -13.7 | 0.230 | 0.504 |
| PLBe+CaOO | 8.42  | 3853<br>(80) | 3386<br>(19) | 2976<br>(21) | 3254<br>(35) | -15.6 | 0.073 | 0.119 |
| ALo       | 8.61  | 3236<br>(55) | 3490<br>(19) | 3742<br>(77) | 3546<br>(21) | + 9.6 | 0.363 | 0.858 |
| OLGa      | 8.90  | 4700<br>(71) | 3989<br>(20) | 4099<br>(11) | 4007<br>(70) | -14.7 | 0.125 | 0.964 |
| GaLL      | 9.31  | 2518<br>(92) | 2208<br>(21) | 2393<br>(52) | 2287<br>(72) | - 9.2 | 0.327 | 0.391 |
| SOBe+POLg | 10.04 | 1831<br>(97) | 1793<br>(4)  | 1881<br>(21) | 1632<br>(7)  | -10.9 | 0.543 | 0.865 |
| BeOO      | 10.31 | 6703<br>(89) | 6163<br>(11) | 6110<br>(78) | 5942<br>(95) | -11.4 | 0.244 | 0.622 |
| PLLg      | 10.48 | 2020<br>(95) | 1999<br>(15) | 2017<br>(19) | 1972<br>(2)  | - 2.4 | 0.915 | 0.586 |
| BeLO      | 10.78 | 8239<br>(12) | 7463<br>(86) | 7677<br>(17) | 7410<br>(65) | -10.1 | 0.155 | 0.736 |
| BeLL      | 11.23 | 4350<br>(55) | 3996<br>(39) | 4308<br>(18) | 4030<br>(10) | - 7.4 | 0.666 | 0.989 |

cont.

Table 43. cont.

|       |       |              |              |              |              |       |       |       |
|-------|-------|--------------|--------------|--------------|--------------|-------|-------|-------|
| LgOO  | 12.80 | 2983<br>(21) | 2248<br>(40) | 2321<br>(59) | 2462<br>(74) | -17.4 | 0.696 | 0.956 |
| LgLO  | 13.36 | 3496<br>(76) | 3015<br>(57) | 2834<br>(68) | 3075<br>(44) | -12.0 | 0.739 | 0.814 |
| LgLL  | 13.91 | 1613<br>(24) | 1926<br>(34) | 2287<br>(94) | 1030<br>(55) | -36.1 | 0.296 | 0.654 |
| Total |       | 378733       | 376677       | 375547       | 376715       |       | 0.352 | 0.881 |

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
 L: linoleic acid (C<sub>18</sub>:2), A: arachidic acid (C<sub>20</sub>:0), Ga: gadoleic acid  
 (C<sub>20</sub>:1), Be: behenic acid (C<sub>22</sub>:0) and Lg: lignoceric acid (C<sub>24</sub>:0).

ND: not detected assumed = zero.

S1: significance of heating time (day).

S2: significance of Total Frying Time (TFT).

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 7 days of commercial frying.

A: average of two replicates.

areas) concentration, however, heating time (DAYS) had a significant effect on POP, POO, SOO, SLS+OOO and (OLO + SLL). No significant effect was observed with heating time (Day) on changes in the remaining triglycerides (Table 43). After 7 days of frying, POP, PLP, POO, PLO, PLL, POA + SOS, SLS + OOO, OLO + SLL, LLL, POBe and ALO increased in concentrations in the range of 3.8 to 63.5%. However, on the contrary, POS, PLS, UNKNOWN<sub>1</sub>, SOO, SLO, UNKNOWN<sub>2</sub>, OLL, AOO, PLBe + GaOO, OLGa, GaLL, SOBe + POLg, BeOO, PLLg, BeLO, BeLL, LgOO, LgLO and LgLL had noticeable decreases in their peak areas in the range of 2.4 to 100%.

The triglyceride, SLO, remained relatively stable throughout the heating and frying processes. Its peak area did not change significantly, only 6.1%. Some (PLP, POO, PLO, PLL and SLO) triglycerides were commonly found in chicken fat (Fig. 29) and appeared relatively stable. No relationship could be established between the stereochemistry of unsaturated fatty acids in the TGs and their rate of degradation, since the location of unsaturated fatty acids in the second triglyceride position did not always provide absolute protection.

Triglycerides with 1 to 6 double bonds showed increase in peak areas, while others with 1 to 5 double bonds showed reduction in their peak area under chicken nugget frying conditions. Therefore, triglyceride with six double bonds

showed accumulation, contradicting previous findings (Yoshida and Thompson, 1984).

The chain length of saturated fatty acids on the first and third position, such as palmitic acid, may provide better protection to unsaturated fatty acids on the second position than stearic, arachidic, behenic or lignoceric acids under these conditions.

The rate of deterioration of a particular triglyceride is greatly influenced by the quality and the quantity of other triglycerides in the frying media.

Total triglyceride (peak area) concentration was essentially stable throughout the heating period.

#### 4.1.2.2.8.1.2 Effect of Commercial Chicken Filet Frying Conditions on Triglycerides in Peanut Oil.

Thirty triglycerides peaks were separated from unheated peanut oil in less than 14 minutes (Table 44 and Fig. 28), however, one peak (unknown) was detected after 5 and 7 DAYS of commercial frying. The identified triglycerides of unheated peanut oil were discussed in the previous section and reported in Table 44.

In general, from the General Linear Model (GLM) procedure, it was found that heating time had no significant ( $P < 0.9976$ ) effect on the changes in triglyceride profile. And it was found that total frying



Table 44. Effect of Commercial Chicken Filet Frying Time (Days) on Triglycerides in Peanut Oil.

| Triglyceride Type | Triglycerides (Peak Area) SD,A |                     |                |                |                |       |       |       |
|-------------------|--------------------------------|---------------------|----------------|----------------|----------------|-------|-------|-------|
|                   | t <sub>R</sub>                 | Heating Time (Days) |                |                |                |       | +D%   | S1    |
|                   | 0                              | 2                   | 5              | 7              |                |       |       |       |
| POP               | 4.03<br>(38)                   | 4559<br>(49)        | 7625<br>(50)   | 7802<br>(33)   | 9139<br>(33)   | +100  | 0.151 | 0.262 |
| PLP               | 4.22<br>(62)                   | 5533<br>(76)        | 8897<br>(25)   | 8750<br>(104)  | 10073<br>(104) | +82.0 | 0.101 | 0.404 |
| UNKNOWN1          | 4.33<br>(24)                   | 724<br>(12)         | 856<br>(11)    | 941<br>(66)    | 1546<br>(66)   | +100  | -     | -     |
| POS               | 5.05<br>(82)                   | 8540<br>(45)        | 2912<br>(14)   | 3134<br>(30)   | 3431<br>(30)   | -59.8 | 0.008 | 0.634 |
| POO               | 5.24<br>(265)                  | 28855<br>(105)      | 39718<br>(383) | 39480<br>(224) | 40587<br>(224) | +40.7 | 0.010 | 0.504 |
| PLS               | 5.48<br>(177)                  | 10347<br>(177)      | ND             | ND             | ND             | -100  | -     | -     |

cont.

Table 44. cont.

|          |      |                |                |                |                |       |       |       |
|----------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| PLO      | 5.52 | 36156<br>(189) | 45432<br>(235) | 44736<br>(265) | 45527<br>(234) | +25.9 | 0.006 | 0.973 |
| UNKNOWN2 | 5.67 | 2432<br>(169)  | ND<br>-        | ND<br>-        | ND<br>-        | -100  | -     | -     |
| PLL      | 5.73 | 12673<br>(142) | 16643<br>(177) | 16241<br>(524) | 15742<br>(71)  | +24.2 | 0.235 | 0.390 |
| POA+SOS  | 6.30 | 1610<br>(22)   | 1681<br>(28)   | 2091<br>(18)   | 2197<br>(31)   | +36.5 | 0.927 | 0.820 |
| S00      | 6.50 | 21653<br>(57)  | 7669<br>(149)  | 9618<br>(45)   | 9347<br>(24)   | -56.8 | 0.016 | 0.871 |
| 000+SLS  | 6.86 | 51374<br>(156) | 61616<br>(345) | 58139<br>(29)  | 56888<br>(143) | +10.7 | 0.226 | 0.833 |
| SLO      | 6.94 | 11988<br>(159) | 10805<br>(109) | 11532<br>(455) | 11691<br>(222) | - 2.5 | 0.727 | 0.674 |
| UNKNOWN3 | 7.10 | 13074<br>(453) | ND<br>-        | ND<br>-        | ND<br>-        | -100  | -     | -     |
| OLO+SLL  | 7.22 | 62771<br>(133) | 7471<br>(234)  | 73873<br>(154) | 72744<br>(33)  | +15.9 | 0.063 | 0.524 |
| OLL      | 7.53 | 45576<br>(139) | 43990<br>(157) | 42806<br>(96)  | 42819<br>(320) | - 6.1 | 0.705 | 0.567 |

cont.

Table 44. cont.

|           |       |              |              |              |              |       |       |       |
|-----------|-------|--------------|--------------|--------------|--------------|-------|-------|-------|
| LLL       | 7.80  | 8087<br>(21) | 8118<br>(25) | 8028<br>(56) | 8520<br>(75) | + 5.4 | 0.026 | 0.053 |
| POBe      | 8.05  | 2857<br>(32) | 2907<br>(42) | 3023<br>(75) | 3050<br>(38) | + 6.8 | 0.420 | 0.151 |
| A00       | 8.26  | 4021<br>(12) | 3318<br>(57) | 3400<br>(34) | 3452<br>(57) | -14.2 | 0.043 | 0.463 |
| PLBe+Ga00 | 8.42  | 3853<br>(34) | 3134<br>(45) | 3004<br>(51) | 2926<br>(25) | -24.0 | 0.022 | 0.472 |
| AL0       | 8.61  | 3236<br>(35) | 3582<br>(19) | 3547<br>(37) | 3542<br>(81) | + 9.5 | 0.084 | 0.158 |
| OLGa      | 8.90  | 4700<br>(51) | 3949<br>(40) | 3971<br>(31) | 3843<br>(20) | -18.2 | 0.253 | 0.739 |
| GaLL      | 9.31  | 2518<br>(94) | 2096<br>(31) | 2181<br>(42) | 2230<br>(52) | -11.4 | 0.028 | 0.081 |
| SOBe+POLg | 10.04 | 1831<br>(7)  | 1704<br>(14) | 1810<br>(21) | 1675<br>(27) | - 8.5 | 0.407 | 0.195 |
| Be00      | 10.31 | 6703<br>(59) | 5941<br>(31) | 5891<br>(8)  | 5858<br>(55) | -12.6 | 0.505 | 0.564 |
| PLLg      | 10.48 | 2020<br>(75) | 2024<br>(15) | 1976<br>(19) | 1956<br>(12) | - 3.2 | 0.288 | 0.131 |

cont.

Table 44. cont.

|       |       |              |              |              |              |       |       |       |
|-------|-------|--------------|--------------|--------------|--------------|-------|-------|-------|
| BeLO  | 10.78 | 8239<br>(42) | 7422<br>(6)  | 7300<br>(37) | 7298<br>(5)  | -11.4 | 0.591 | 0.555 |
| BeLL  | 11.23 | 4350<br>(21) | 4116<br>(30) | 4035<br>(79) | 4040<br>(24) | - 7.1 | 0.876 | 0.803 |
| LgOO  | 12.80 | 2983<br>(45) | 2677<br>(29) | 2459<br>(8)  | 2467<br>(20) | -17.3 | 0.924 | 0.896 |
| LgLO  | 13.36 | 3496<br>(14) | 3271<br>(20) | 2964<br>(69) | 2658<br>(42) | -23.9 | 0.675 | 0.266 |
| LgLL  | 13.91 | 1613<br>(22) | 1586<br>(63) | 1251<br>(45) | 1180<br>(83) | +26.8 | 0.045 | 0.029 |
| Total |       | 378733       | 378627       | 374980       | 378626       |       | 0.666 | 0.843 |

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), A: arachidic acid (C<sub>20</sub>:0), O: oleic acid (C<sub>18</sub>:1), L: linoleic acid (C<sub>18</sub>:2), Ga: gadoleic acid (C<sub>20</sub>:1), Be: behenic acid (C<sub>22</sub>:0) and Lg: lignoëric acid (C<sub>24</sub>:0).

ND: not detected, assumed = zero.

S1: significance of heating time (day).

S2: significance of Total Frying Time (TFT).

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

Table 44. cont.

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+D%: percent increase or decrease in peak area after 7 days of commercial frying.

A : average of two replicates.

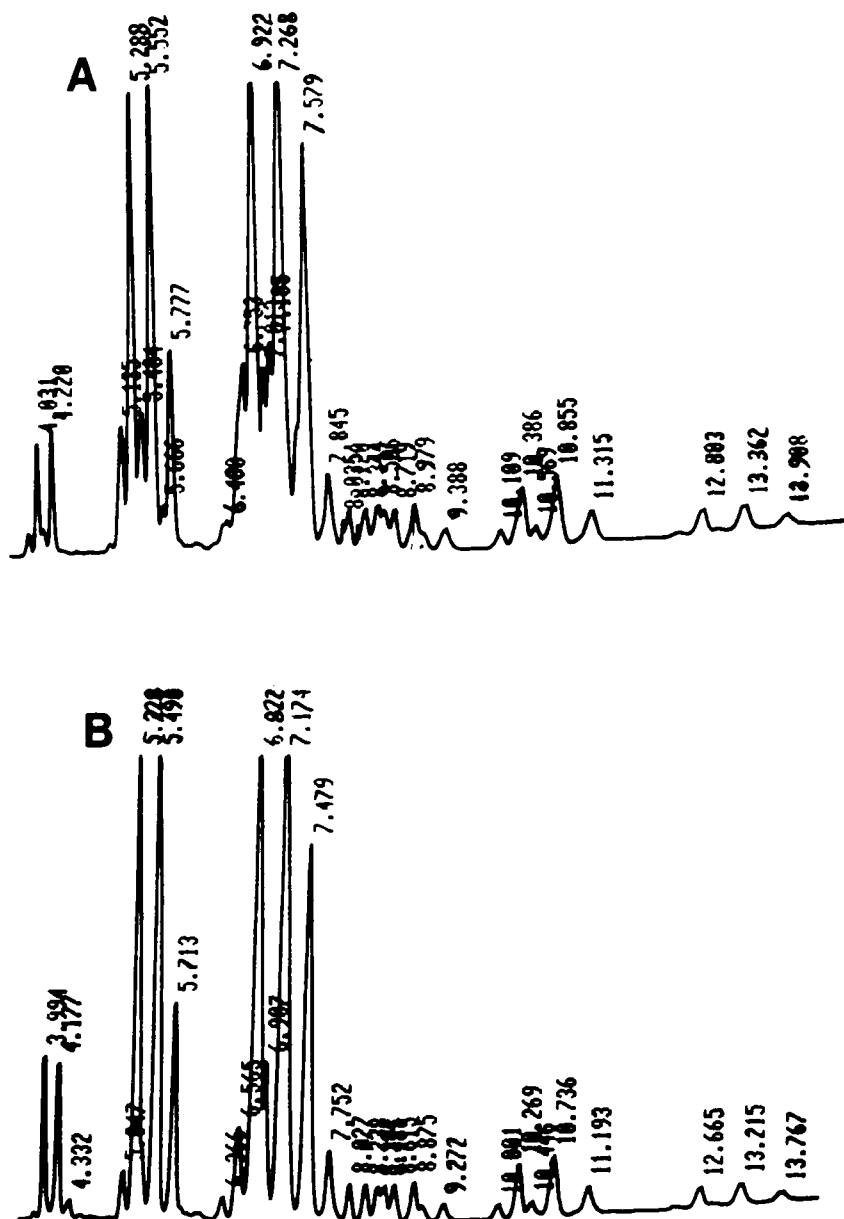


Fig. 28. Effect of commercial frying time (Days) of chicken filet on triglyceride profile in peanut oil. A-unheated; B-heated to 7 days.

**Peak identification :**

|                       |   |                     |
|-----------------------|---|---------------------|
| 1. cholesterol        | M = tetradecanoic acid,                   | C <sub>14</sub> : 0 |
| 2. PPP                | P = hexadecanoic acid,                    | C <sub>16</sub> : 0 |
| 3. PPS                | O = cis-9-octadecenoic acid,              | C <sub>18</sub> : 1 |
| 4. PPO                | L = cis, cis-9, 12-octadecadienoic acid,  | C <sub>18</sub> : 2 |
| 5. MOO                | S = octadecanoic acid,                    | C <sub>18</sub> : 0 |
| 6. PP <sub>o</sub> O  | A = eicosanoic acid,                      | C <sub>20</sub> : 0 |
| 7. PLP                | P <sub>o</sub> = cis-9-hexadecenoic acid, | C <sub>16</sub> : 1 |
| 8. PLP <sub>o</sub>   |   |                     |
| 9. MLO                |   |                     |
| 10. PSO               |   |                     |
| 11. POO               |   |                     |
| 12. PLS               |   |                     |
| 13. P <sub>o</sub> OO |   |                     |
| 14. PLO               |   |                     |
| 15. P <sub>o</sub> LO |   |                     |
| 16. PLL               |   |                     |
| 17. SSO               |   |                     |
| 18. SOO               |   |                     |
| 19. OOO               |   |                     |
| 20. SLO               |   |                     |
| 21. OOL               |   |                     |
| 22. OLL               |   |                     |

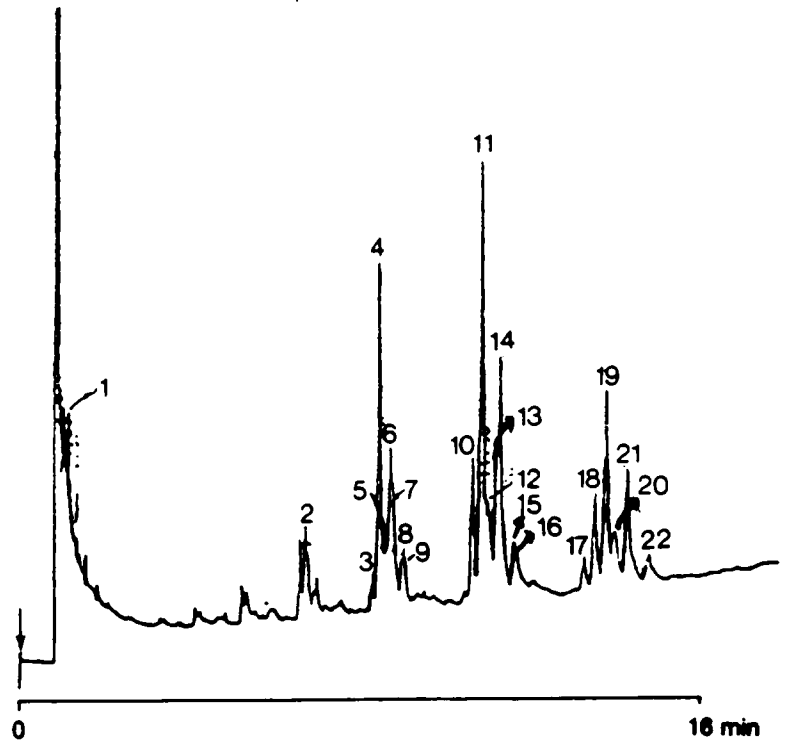


Fig. 29. Triglyceride Profile of Chicken Fat (Chrompack, 1989).

time (TFT) has no significant effect on changes in triglycerides under the conditions used in this study. However, total heating time (DAYS) had a significant effect on decreasing POS, SOO, AOO, PLBe + GaOO, GaLL and LgLL. Moreover, heating time (DAYS) had significant effect on increasing the peak areas of POO, PLO, and LLL, respectively. The significant accumulation of LLL in the triglyceride profiles indicated its relative stability under these conditions. Nevertheless, this triglyceride was not detected in the chicken fat. From this finding, it was concluded that the deterioration rate of unsaturated triglycerides is independent of their degree of unsaturation under these conditions. A previous study (Thompson et al., 1967) also supports this conclusion, while another (Yoshida and Thompson, 1984), contradicts this finding.

Although the frying period of a chicken filet batch requires 3.5 min. under pressure at 325<sup>o</sup>, the relative changes in triglycerides under these conditions were relatively similar to the relative changes in triglycerides of peanut oil used to fry chicken nuggets for 2.5 min. under atmospheric pressure and at 325<sup>o</sup>. This indicated that the triglyceride changes occurring during frying are not influenced significantly by the frying conditions. This finding contradicts an earlier study by Thompson et al. (1967), who reported that the degree of shortening



deterioration was independent of their degree of unsaturation but rather dependent upon how they were used. If chicken fat contributed to changes in the triglyceride finger print, this effect was not noticeable for either the chicken nuggets or filets.

#### 4.1.2.2.8.2 EFFECT OF COMMERCIAL FRYING CONDITIONS ON TRIGLYCERIDES IN COTTONSEED OIL LIQUID SHORTENING (CS/OLS).

##### 4.1.2.2.8.2.1 Effect of Commercial Chicken Nuggets Frying Conditions on Triglycerides of Cottonseed Oil Liquid Shortening (CS/OLS).

Twenty four triglyceride peaks were separated from unheated commercial cottonseed oil liquid shortening in less than 9 minutes (Table 45 and Fig. 36 in Appendix H). The triglycerides with high concentrations (peak areas), were PLP, PLO, PLL, OLL, and LLL, (25158, 30252, 52267, 24383 and 28848) respectively. From the General Linear Model (GLM) procedure, heating time (DAYS) had no significant ( $P < 0.9998$ ) effect on changes in triglyceride profile. Also Total Frying Time (TFT) had no significant effect on changes in triglyceride peak areas. However, heating time (DAYS) had a significant ( $P < 0.014$ ) effect in

Table 45. Effect of Commercial Chicken Nuggets Frying Time (Days) on Triglycerides in Cottonseed Oil Liquid Shortening.

| Triglyceride Type | Triglycerides (Peak Area) SD,A |                |                |               |                |       |       |       |
|-------------------|--------------------------------|----------------|----------------|---------------|----------------|-------|-------|-------|
|                   | t <sub>R</sub>                 | 0              | 2              | 5             | 7              | +D%   | S1    | S2    |
| PPP               | 3.61                           | 804<br>(77)    | 714<br>(36)    | 786<br>(46)   | 858<br>(70)    | + 6.7 | 0.404 | 0.616 |
| MOP               | 3.76                           | 1273<br>(45)   | 1994<br>(14)   | 1530<br>(33)  | 1486<br>(74)   | +16.7 | 0.824 | 0.739 |
| MLP               | 3.93                           | 1818<br>(27)   | 2272<br>(51)   | 2079<br>(42)  | 2079<br>(15)   | +14.4 | 0.702 | 0.999 |
| PPS               | 4.53                           | 1352<br>(42)   | 879<br>(53)    | 1365<br>(17)  | 963<br>(64)    | -28.8 | 0.014 | 0.011 |
| POP               | 4.68                           | 7835<br>(37)   | 9474<br>(99)   | 10288<br>(12) | 9116<br>(112)  | +16.4 | 0.713 | 0.980 |
| PLP               | 4.91                           | 25158<br>(537) | 25574<br>(389) | 27253<br>(98) | 25710<br>(844) | + 2.2 | 0.111 | 0.258 |
| PLPo+MLo          | 5.05                           | 2808<br>(15)   | 2654<br>(46)   | 3196<br>(93)  | 3021<br>(67)   | + 7.6 | 0.359 | 0.225 |

cont.

Table 45. cont.

|                      |      |                |                |                |                |       |       |       |
|----------------------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| MLL                  | 5.24 | 2215<br>(88)   | 2021<br>(210)  | 2497<br>(22)   | 2018<br>(27)   | - 8.9 | 0.501 | 0.556 |
| UNKNOWN <sub>1</sub> | 5.54 | 3270<br>(79)   | 2051<br>(56)   | 2013<br>(54)   | 2208<br>(12)   | -32.5 | 0.363 | 0.965 |
| POS                  | 5.84 | 1902<br>(64)   | 4087<br>(28)   | 3429<br>(31)   | 2905<br>(52)   | +52.7 | 0.616 | 0.777 |
| POO                  | 6.01 | 6796<br>(61)   | 12501<br>(107) | 14454<br>(171) | 9266<br>(140)  | +36.3 | 0.530 | 0.903 |
| PLS                  | 6.11 | 5632<br>(94)   | 5875<br>(39)   | 907<br>(7)     | 6137<br>(16)   | + 8.9 | 0.005 | 0.442 |
| PLO                  | 6.34 | 30252<br>(356) | 29820<br>(260) | 31197<br>(39)  | 31396<br>(29)  | + 3.8 | 0.921 | 0.814 |
| PLL                  | 6.65 | 52267<br>(222) | 48411<br>(578) | 50543<br>(72)  | 50608<br>(127) | - 3.2 | 0.849 | 0.847 |
| PoLL                 | 6.82 | 2087<br>(91)   | 2218<br>(14)   | 1796<br>(13)   | 2954<br>(17)   | +41.5 | 0.139 | 0.999 |
| UNKNOWN <sub>2</sub> | 7.28 | 3019<br>(25)   | 1734<br>(57)   | 1860<br>(33)   | 2193<br>(10)   | +27.4 | 0.317 | 0.952 |
| SOS                  | 7.38 | 727<br>(5)     | 1085<br>(85)   | 813<br>(39)    | 722<br>(25)    | 0.0   | 0.673 | 0.443 |

cont.

Table 45. cont.

|       |      |                |                |                |                |       |       |       |
|-------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| S00   | 7.48 | 1012<br>(50)   | 2866<br>(27)   | 2366<br>(51)   | 1586<br>(32)   | +56.7 | 0.652 | 0.738 |
| O00   | 7.89 | 3416<br>(14)   | 4303<br>(98)   | 3681<br>(31)   | 3565<br>(57)   | + 4.4 | 0.482 | 0.651 |
| SLO   | 7.82 | 4001<br>(79)   | 3772<br>(17)   | 3823<br>(47)   | 3701<br>(16)   | - 7.5 | 0.941 | 0.727 |
| OLO   | 8.07 | 8698<br>(22)   | 8535<br>(11)   | 8488<br>(93)   | 8339<br>(49)   | - 4.1 | 0.959 | 0.686 |
| SLL   | 8.19 | 5968<br>(76)   | 4959<br>(34)   | 4688<br>(72)   | 5409<br>(94)   | - 9.4 | 0.468 | 0.610 |
| OLL   | 8.43 | 24383<br>(754) | 22433<br>(176) | 23014<br>(198) | 22859<br>(74)  | - 6.3 | 0.819 | 0.923 |
| LLL   | 8.86 | 28848<br>(430) | 26217<br>(456) | 26559<br>(253) | 26412<br>(274) | - 8.4 | 0.824 | 0.919 |
| Total |      | 225396         | 22516          | 225152         | 225234         |       | 0.114 | 0.496 |

M: myristic acid (C<sub>14</sub>:0), P: palmitic acid (C<sub>16</sub>:0), P: palmitoleic acid (C<sub>16</sub>:1), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1), L: linoleic acid (C<sub>18</sub>:2).

S1: significance of heating time (day).

S2: significance of Total Frying Time (TFT).

Table 45. cont.

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$t_R$ : Retention time in minutes.

SD: standard deviations are the values in brackets.

$\pm D\%$ : percent increase or decrease in peak area after 7 days of commercial frying.

A: average of two replicates.

decreasing PPS peak area (28.8%) after 7 DAYS of heating. Also Total Heating Time (THT) produced a significant ( $P < 0.005$ ) increase in PLS (peak area) 8.9%. However, no significant effect was observed on the remaining triglycerides. Triglycerides which had greater peak areas, such as PLL, OLL, and LLL, experienced an insignificant decrease in peak area after 7 days of commercial frying, which indicated that triglyceride concentration did not affect the rate of triglyceride degradation. PLP and PLO had an insignificant decrease in peak area after 7 days of commercial frying. It can be concluded that:

1. In general, no significant ( $P < 0.114$ ) change in total peak areas were observed, even though the PPS triglyceride showed a noticeable reduction in its peak area (from 1352 to 963).

2. No relationship was found between the number of double bonds and rate of deterioration in cottonseed oil liquid shortening used to fry chicken nuggets at  $325^{\circ}$ .

#### 4.1.2.2.8.2.2 Effect of Commercial Chicken Filets Frying Conditions on Triglycerides in Cottonseed Oil Liquid Shortening.

Triglycerides of unheated cottonseed oil liquid shortening were reported in the previous section and in Tables

45 and 46, and Fig. 30. The changes in triglyceride profile under the chicken filet frying conditions were closely related to the changes that occurred under the commercial frying conditions of chicken nuggets. From the GLM procedure it was found that heating time had no significant ( $P < 0.9998$ ) effect on changes in triglyceride profile. Slight increases in peak areas were observed in PPP, MLP, POP, PLP, PLP<sub>o</sub> + MLO, POS, POO, PLS, PLO, P<sub>o</sub>LL, SOO, OOO, SLO and OLO. This increase was in the range of 2.9 to 34.4%. Other triglycerides such as MOP, PPS, MLL, UNKNOWN<sub>1</sub>, PLL, UNKNOWN<sub>2</sub>, SLL, OLL and LLL showed reduction in their concentrations in the range of 5.1 to 43.4%.

Some differences were observed between chicken filet and chicken nugget frying conditions. MOP decreased 22.7% in peak area under pressurized frying conditions, it showed a slight accumulation under nonpressurized frying conditions 16.7%. Also SOS was not detected under pressurized frying conditions, however, it appeared under chicken nugget frying conditions. The last separated triglyceride, LLL, decreased 8.4% under pressurized cooking and 10.6% under nonpressurized frying conditions. Peak, UNKNOWN<sub>1</sub>, was the only triglyceride which was reduced (43.4%) significantly ( $P < 0.014$ ), as a result of the 7 day chicken filet heating period under commercial frying conditions. The reduction (32.5%) in its peak area under

Table 46. Effect of Commercial Chicken Filets Frying Time (Days) on Triglycerides in Cottonseed Oil Liquid Shortening.

| Triglyceride Type | Triglycerides (Peak Area) SD,A |                |               |                |               |       |       |       |
|-------------------|--------------------------------|----------------|---------------|----------------|---------------|-------|-------|-------|
|                   | t <sub>R</sub>                 | 0              | 2             | 5              | 7             | +D%   | S1    | S2    |
| PPP               | 3.61                           | 804<br>(19)    | 767<br>(61)   | 964<br>(16)    | 1012<br>(32)  | +25.9 | 0.129 | 0.051 |
| MOP               | 3.76                           | 1273<br>(10)   | 1272<br>(14)  | 1373<br>(88)   | 984<br>(42)   | -22.7 | 0.128 | 0.069 |
| MLP               | 3.93                           | 1818<br>(15)   | 1982<br>(35)  | 2537<br>(3)    | 1994<br>(16)  | + 9.7 | 0.167 | 0.118 |
| PPS               | 4.53                           | 1352<br>(61)   | 1014<br>(29)  | 915<br>(72)    | 994<br>(73)   | -26.5 | 0.890 | 0.806 |
| POP               | 4.68                           | 7835<br>(18)   | 8770<br>(93)  | 9226<br>(108)  | 9658<br>(39)  | +23.3 | 0.910 | 0.929 |
| PLP               | 4.91                           | 25158<br>(711) | 26094<br>(18) | 25947<br>(924) | 25879<br>(47) | + 2.9 | 0.520 | 0.724 |
| PLPo+MLO          | 5.05                           | 2808<br>(9)    | 3101<br>(78)  | 3204<br>(43)   | 3423<br>(66)  | +21.9 | 0.837 | 0.601 |

cont.



Table 46. cont.

|                      |      |                |                |               |                |       |       |       |
|----------------------|------|----------------|----------------|---------------|----------------|-------|-------|-------|
| MLL                  | 5.24 | 2215<br>(25)   | 2172<br>(65)   | 2032<br>(33)  | 1910<br>(22)   | -13.8 | 0.692 | 0.300 |
| UNKNOWN <sub>1</sub> | 5.54 | 3270<br>(55)   | 1767<br>(59)   | 1790<br>(30)  | 1852<br>(24)   | -43.4 | 0.014 | 0.221 |
| POS                  | 5.84 | 1902<br>(56)   | 2346<br>(73)   | 2392<br>(20)  | 2524<br>(13)   | +32.7 | 0.213 | 0.276 |
| POO                  | 6.01 | 6996<br>(65)   | 8094<br>(30)   | 8372<br>(16)  | 9128<br>(77)   | +30.5 | 0.289 | 0.181 |
| PLS                  | 6.11 | 5632<br>(76)   | 6122<br>(94)   | 6547<br>(60)  | 6325<br>(85)   | +12.3 | 0.164 | 0.742 |
| PLO                  | 6.34 | 30252<br>(678) | 31834<br>(75)  | 32530<br>(22) | 32741<br>(543) | + 8.2 | 0.060 | 0.071 |
| PLL                  | 6.65 | 52267<br>(165) | 52083<br>(643) | 50777<br>(68) | 49604<br>(142) | - 5.1 | 0.747 | 0.644 |
| PoLL                 | 6.82 | 2087<br>(84)   | 2852<br>(92)   | 2807<br>(86)  | 2806<br>(15)   | +34.4 | 0.307 | 0.853 |
| UNKNOWN <sub>2</sub> | 7.28 | 3019<br>(62)   | 1809<br>(53)   | 1808<br>(84)  | 1924<br>(23)   | -36.3 | 0.072 | 0.453 |
| SOO                  | 7.48 | 1012<br>(56)   | 1190<br>(52)   | 1133<br>(31)  | 1351<br>(24)   | +33.5 | 0.231 | 0.510 |

cont.

Table 46. cont.

|       |      |                |                |                |                |       |       |       |
|-------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| 000   | 7.89 | 3416<br>(68)   | 3513<br>(14)   | 3656<br>(18)   | 3891<br>(97)   | +13.9 | 0.913 | 0.658 |
| SLO   | 7.82 | 4001<br>(43)   | 3725<br>(62)   | 3934<br>(20)   | 4058<br>(86)   | + 1.4 | 0.181 | 0.495 |
| OLO   | 8.07 | 8698<br>(99)   | 8708<br>(47)   | 8988<br>(12)   | 9027<br>(10)   | + 3.8 | 0.919 | 0.826 |
| SLL   | 8.19 | 5968<br>(79)   | 5618<br>(33)   | 5507<br>(23)   | 5658<br>(140)  | - 5.2 | 0.050 | 0.781 |
| OLL   | 8.47 | 24383<br>(600) | 23523<br>(401) | 23084<br>(123) | 22795<br>(771) | - 6.5 | 0.881 | 0.582 |
| LLL   | 8.86 | 28848<br>(307) | 27445<br>(581) | 26407<br>(120) | 25799<br>(128) | -10.6 | 0.915 | 0.556 |
| Total |      | 225396         | 225379         | 225422         | 225433         |       | 0.431 | 0.327 |

M: myristic acid (C<sub>14</sub>:0), P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), P: palmi~~to~~leic acid (C<sub>16</sub>:1), O: oleic acid (C<sub>18</sub>:1), L: linoleic acid (C<sub>18</sub>:2).

S1: significance of heating time (day).

S2: significance of Total Frying Time (TFT).

t<sub>R</sub>: Retention time in minutes.

Table 46. cont.

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SD : standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 7 days of commercial frying.

A : average of two replicates.

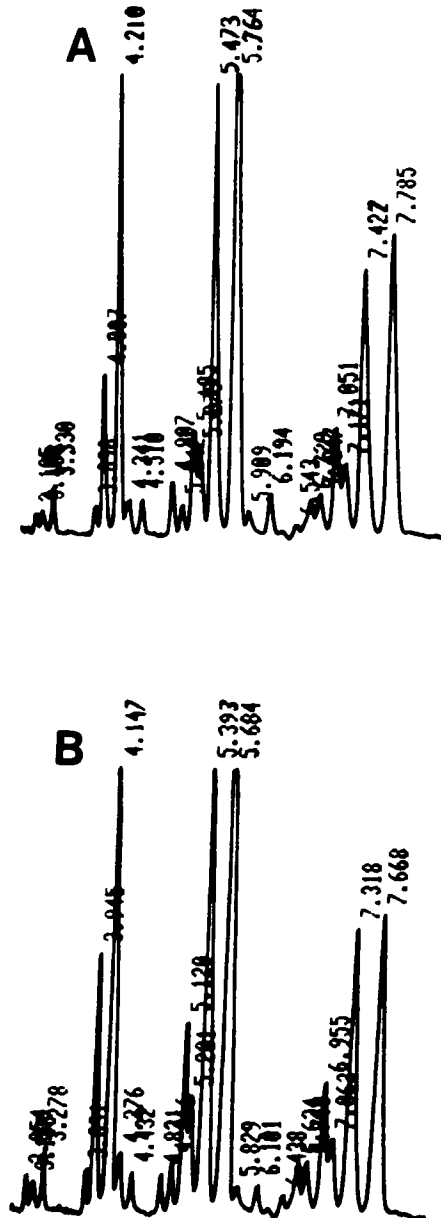


Fig. 30. Effect of commercial frying time (Days) of chicken filets on triglyceride profile in cottonseed oil liquid shortening. A-unheated; B-heated to 7 days.

nonpressurized frying was not significant ( $P < 0.363$ , Table 45).

In general, it can be concluded that:

1. The triglyceride compositions in CS/OLS under chicken filet frying conditions varied unpredictably. Some of The triglyceride composition in CS/OLS varied an unpredictably under chicken filet frying conditions. Some of the triglycerides with 1 to 5 double bonds were increased in peak area, while others with 1 and 4 to 6 double bonds showed reduction in peak area.

2. Different frying conditions, such as pressurized vs nonpressurized frying, had no significant effect on changes in triglyceride profiles.

3. No relationship was found between number of double bonds and rate of deterioration in cottonseed oil liquid shortening under both frying conditions. This indicated that frying conditions does not influence the rate of triglyceride deterioration.

4. Several triglycerides showed more accumulation during frying as compared to static heating conditions, which indicated that chicken products are probably selective in absorbing triglycerides from the heating medium. Because, TFF had no significant effect on TG profile. Furthermore, chicken breast contains only 0.32% triglycerides. Therefore, it is expected that the chicken

triglycerides do not contribute significantly to the triglyceride profile, hence to the rate of deterioration.

#### 4.1.2.2.8.3 EFFECT OF COMMERCIAL FRYING CONDITIONS ON TRIGLYCERIDES IN SOYBEAN OIL LIQUID SHORTENING (SB/OLS).

##### 4.1.2.2.8.3.1 Effect of Commercial Chicken Nuggets Frying Conditions on Triglycerides in Soybean Oil Liquid Shortening (SB/OLS).

Nineteen triglyceride peaks were separated from unheated SB/OLS in less than 10 minutes (Table 47 and Fig. 37 in Appendix H). Several triglycerides, such as POO, PLS, PLO, PLL, SOO, OOO, SLS, SLO, OOL, SLL, OLL, and LLL, were present with high concentration (peak areas). Using the General Linear Model (GLM) procedure to detect significance, it was found that heating time (DAYS) had no significant ( $P < 0.9998$ ) effect on changes in triglyceride profile. Also, Total Frying Time (TFT) had no significant effect on changes in triglyceride concentration under nonpressurized frying. However, heating time (DAYS) had a significant ( $P < 0.035$ ,  $0.016$  and  $0.011$ ) effect on increasing the  $PLL_n$  peak area and decreasing both SOO and  $LLL_n$ , respectively. Even though several triglycerides (POP, PLP, UNKNOWN<sub>1</sub>, POS, PLL, SLL and LLL) increased in peak area in

Table 47. Effect of Commercial Chicken Nuggets Frying Time (Days) on Triglycerides Soybean Oil Liquid Shortening.

| Triglyceride Type    | Triglycerides (Peak Area) SD,A |                |                |                |                |       |       |       |
|----------------------|--------------------------------|----------------|----------------|----------------|----------------|-------|-------|-------|
|                      | t <sub>R</sub>                 | 0              | 2              | 5              | 7              | +D%   | S1    | S2    |
| POP                  | 4.67                           | 6846<br>(79)   | 9173<br>(86)   | 10123<br>(38)  | 10405<br>(497) | +51.9 | 0.155 | 0.691 |
| PLP                  | 4.88                           | 6414<br>(18)   | 8131<br>(23)   | 10724<br>(118) | 9918<br>(43)   | +54.6 | 0.325 | 0.966 |
| UNKNOWN <sub>1</sub> | 5.72                           | 3729<br>(66)   | 3268<br>(61)   | 4030<br>(67)   | 3845<br>(79)   | + 3.1 | 0.513 | 0.588 |
| POS                  | 5.88                           | 5333<br>(65)   | 4988<br>(20)   | 4982<br>(75)   | 6094<br>(48)   | +14.3 | 0.322 | 0.737 |
| P00                  | 6.09                           | 33611<br>(266) | 43240<br>(680) | 41982<br>(100) | 17559<br>(256) | -47.8 | 0.139 | 0.621 |
| PLS                  | 6.10                           | 12730<br>(970) | 8811<br>(70)   | 8827<br>(66)   | 12640<br>(318) | - 0.7 | 0.649 | 0.575 |
| PLO                  | 6.38                           | 50258<br>(753) | 53213<br>(589) | 51581<br>(432) | 50584<br>(367) | + 0.7 | 0.766 | 0.654 |

cont.

Table 47. cont.

|                      |      |                |                |                 |                |       |       |       |
|----------------------|------|----------------|----------------|-----------------|----------------|-------|-------|-------|
| PLL                  | 6.63 | 23240<br>(907) | 24419<br>(23)  | 132012<br>(482) | 25092<br>(587) | + 7.9 | 0.821 | 0.859 |
| PLLn                 | 6.91 | 3713<br>(74)   | 3367<br>(7)    | 3661<br>(59)    | 3759<br>(72)   | + 1.2 | 0.035 | 0.567 |
| UNKNOWN <sub>2</sub> | 7.15 | 8661<br>(54)   | 7864<br>(11)   | 8258<br>(103)   | 7609<br>(37)   | -12.2 | 0.699 | 0.572 |
| SOS                  | 7.30 | 3102<br>(95)   | 3050<br>(59)   | 3005<br>(396)   | 3131<br>(51)   | + 0.9 | 0.919 | 0.783 |
| S00                  | 7.59 | 12599<br>(899) | 11173<br>(12)  | 10839<br>(171)  | 12222<br>(812) | - 2.9 | 0.016 | 0.946 |
| 000+SLS              | 7.89 | 43663<br>(678) | 41456<br>(286) | 44464<br>(890)  | 41507<br>(106) | - 4.9 | 0.946 | 0.952 |
| SLO                  | 8.00 | 24225<br>(643) | 32157<br>(196) | 24248<br>(114)  | 24129<br>(691) | - 0.4 | 0.704 | 0.903 |
| OOL                  | 8.28 | 67230<br>(779) | 64706<br>(777) | 63496<br>(162)  | 61829<br>(608) | - 8.0 | 0.987 | 0.731 |
| SLL                  | 8.38 | 20208<br>(909) | 21692<br>(175) | 20992<br>(94)   | 22593<br>(886) | +11.8 | 0.575 | 0.573 |
| OLL                  | 8.63 | 57603<br>(490) | 57185<br>(531) | 55528<br>(94)   | 53335<br>(488) | - 7.4 | 0.920 | 0.602 |

cont.



Table 47. cont.

|       |      |                |                |                |                |       |       |       |
|-------|------|----------------|----------------|----------------|----------------|-------|-------|-------|
| LLL   | 8.95 | 18999<br>(539) | 22293<br>(146) | 23358<br>(163) | 20976<br>(501) | +10.4 | 0.306 | 0.905 |
| LLLn  | 9.28 | 4682<br>(52)   | 2074<br>(89)   | 1993<br>(13)   | 1781<br>(29)   | -61.9 | 0.011 | 0.951 |
| Total |      | 406846         | 406934         | 406895         | 406972         |       | 0.756 | 0.569 |

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
L: linoleic acid (C<sub>18</sub>:2), Ln: linolenic acid (C<sub>18</sub>:3).

S1: significance of heating time (day).

S2: significance of Total Frying Time (TFT).

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 7 days of commercial frying.

A: average of two replicates.

the range of 3.1 to 54.6%, however, the changes were not significant. Furthermore, POO, UNKNOWN<sub>2</sub>, SOO, OOO, OOL, OLL, and LLL<sub>n</sub> showed slight, but insignificant, decreases in their peak areas in the range of 2.9 to 61.9%.

From the present study it is found that triglycerides with 1 to 6 double bonds increased with concentration, while others with 2 to 5 and 7 double bonds decreased in concentration.

The significant accumulation of PLL<sub>n</sub> and LLL, triglycerides with 5 and 6 double bonds, contradicts a previous study by Yoshida and Alexander (1984) who reported that after 100 hr of heating soybean oil, an accumulation in TGs with one to four double bonds was observed while those containing five to seven double bonds were reduced.

#### 4.1.2.2.8.3.2 Effect of Commercial Chicken Filets Frying Conditions on Triglycerides of Soybean Oil Liquid Shortening (SB/OLS).

The effect of chicken filet frying conditions on triglyceride pattern is presented in Table 48.

As a result of commercial chicken filet frying conditions, LLL<sub>n</sub> was detected in unheated SB/OLS, however it was not detected upon subsequent frying to 2, 5 and 7 DAYS (Table 48). In general, no similarities in triglyceride profiles were found under pressurized and

Table 48. Effect of Commercial Chicken Filets Frying Time (Days) on Triglycerides Soybean Oil Liquid Shortening.

| Triglyceride Type    | Triglycerides (Peak Area) SD,A |                |                |                |                |        |       |       |
|----------------------|--------------------------------|----------------|----------------|----------------|----------------|--------|-------|-------|
|                      | t <sub>R</sub>                 | 0              | 2              | 5              | 7              | +D%    | S1    | S2    |
| POP                  | 4.67                           | 6846<br>(25)   | 10446<br>(536) | 12327<br>(118) | 14847<br>(482) | +116.9 | 0.644 | 0.732 |
| PLP                  | 4.88                           | 6414<br>(48)   | 10013<br>(873) | 11685<br>(239) | 14513<br>(779) | +126.3 | 0.706 | 0.705 |
| UNKNOWN <sub>1</sub> | 5.72                           | 3729<br>(66)   | 3573<br>(23)   | 3885<br>(202)  | 3618<br>(23)   | - 2.9  | 0.587 | 0.877 |
| POS                  | 5.88                           | 5333<br>(10)   | 5418<br>(21)   | 6584<br>(62)   | 8094<br>(85)   | + 51.8 | 0.221 | 0.708 |
| POO                  | 6.09                           | 33611<br>(174) | 36394<br>(256) | 45763<br>(144) | 44370<br>(189) | + 32.0 | 0.856 | 0.772 |
| PLS                  | 6.10                           | 12730<br>(717) | 12704<br>(468) | 12937<br>(133) | 11078<br>(251) | - 12.9 | 0.422 | 0.622 |
| PLO                  | 6.38                           | 50258<br>(114) | 5536<br>(317)  | 54369<br>(98)  | 55060<br>(130) | + 9.6  | 0.448 | 0.597 |

cont.

Table 48. cont.

|                      |      |                |                |                |                  |       |        |       |
|----------------------|------|----------------|----------------|----------------|------------------|-------|--------|-------|
| PLL                  | 6.63 | 23240<br>(515) | 26254<br>(159) | 24746<br>(38)  | 24011 +<br>(876) | 3.3   | 0.197  | 0.567 |
| PLLn                 | 6.91 | 3713<br>(25)   | 3569<br>(59)   | 3547<br>(11)   | 3501 -<br>(68)   | 5.7   | 0.545  | 0.849 |
| UNKNOWN <sub>2</sub> | 7.15 | 8661<br>(120)  | 7974<br>(47)   | 7613<br>(90)   | 6093 -<br>(21)   | 29.7  | 0.748  | 0.992 |
| SOS                  | 7.30 | 3102<br>(714)  | 3093<br>(24)   | 2879<br>(87)   | 11201 +<br>(113) | 261.0 | 0.0002 | 0.737 |
| S00                  | 7.59 | 12599<br>(885) | 11144<br>(78)  | 11529<br>(26)  | 10903 -<br>(689) | 13.5  | 0.320  | 0.597 |
| 000+SLS              | 7.89 | 43663<br>(976) | 42193<br>(375) | 42270<br>(61)  | 43753 +<br>(675) | 0.2   | 0.748  | 0.636 |
| SLO                  | 8.00 | 24225<br>(284) | 30596<br>(277) | 29816<br>(218) | 26769 +<br>(273) | 10.5  | 0.294  | 0.863 |
| OOL                  | 8.28 | 67230<br>(963) | 78516<br>(104) | 81849<br>(151) | 85496 +<br>(841) | 27.2  | 0.728  | 0.618 |
| SLL                  | 8.38 | 20208<br>(652) | 9607<br>(80)   | 8522<br>(28)   | 7432 -<br>(45)   | 63.2  | 0.017  | 0.769 |
| OLL                  | 8.63 | 57603<br>(726) | 59204<br>(318) | 55447<br>(160) | 50437 -<br>(138) | 12.4  | 0.442  | 0.805 |

cont.

Table 48. cont.

|       |      |                |                |                |                  |        |       |       |
|-------|------|----------------|----------------|----------------|------------------|--------|-------|-------|
| LLL   | 8.95 | 18999<br>(509) | 23128<br>(569) | 18470<br>(113) | 18756 -<br>(239) | 1.3    | 0.631 | 0.728 |
| LlLn  | 9.28 | 4682<br>(16)   | ND<br>-        | ND<br>-        | ND<br>-          | -100.0 | -     | -     |
| Total |      | 40684          | 406882         | 406982         | 407074           |        | 0.835 | 0.890 |

P: palmitic acid (C<sub>16</sub>:0), S: stearic acid (C<sub>18</sub>:0), O: oleic acid (C<sub>18</sub>:1),  
L: linoleic acid (C<sub>18</sub>:2), Ln: linolenic acid (C<sub>18</sub>:3).

S1: significance of day.

S2: significance of Total Frying Time (TFT).

t<sub>R</sub>: Retention time in minutes.

SD: standard deviations are the values in brackets.

+D%: percent increase or decrease in peak area after 7 days of commercial heating.

A: average of two replicates.

nonpressurized frying conditions (Fig. 37 in Appendix H and Fig. 31 in this Chapter). In general, heating time had no significant ( $P < 0.9999$ ) effect on changes in triglyceride profile. Some triglycerides such as POP, PLP, POS, POO, PLO, PLL, SOS, SLO and OOL showed a tendency to accumulate under pressurized frying conditions. The range of this increase was from 3.3 to 261.0%. The SOS peak had a significant ( $P < 0.0002$ ) increase under pressurized frying conditions. Moreover, more noticeable reductions (1.3 to 100%) in UNKNOWN<sub>1</sub>, PLS, PLLn, UNKNOWN<sub>2</sub>, SOO, SLL, OLL, LLL and LLLn were realized when compared with the nonpressurized frying conditions. The reduction in SLL was also significant ( $P < 0.017$ ).

From this experiment, it is concluded that different frying conditions resulted in significant differences in the triglyceride profiles. A previous study (Thompson et al., 1967) also supports this conclusion.

It was found from the present study that in the case of soybean oil liquid shortening, the effects of pressurized cooking conditions resulted in a greater difference between triglyceride compositions, including those that are relatively stable to thermal deterioration. The pressure effect on triglycerides was not observed with the peanut oil sample, while cottonseed oil liquid shortening showed smaller differences in triglyceride composition.

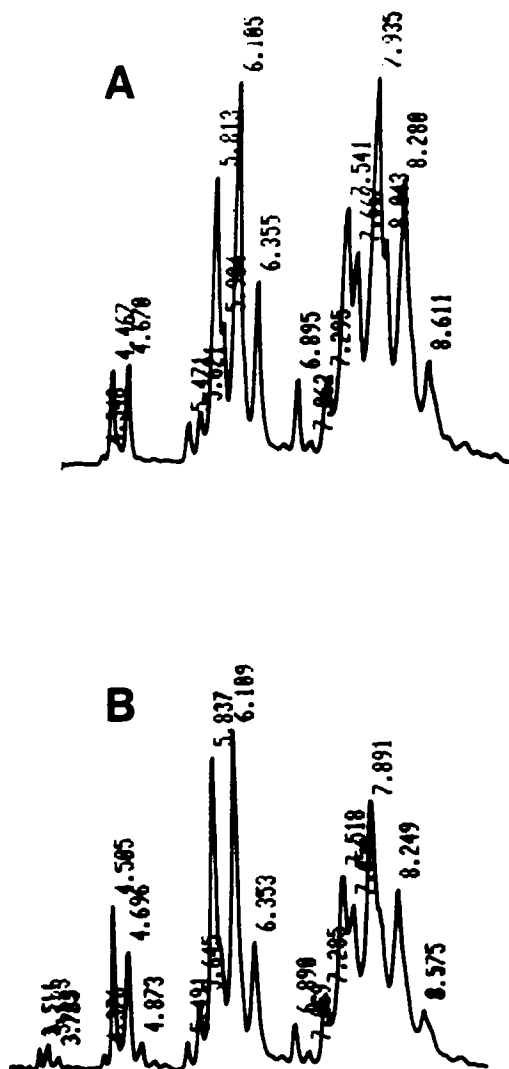


Fig. 31. Effect of commercial frying time (Days) of chicken filet on triglyceride profile in soybean oil liquid shortening. A-unheated; B-heated to 7 days.

It is unknown whether triglyceride absorption into the chicken coating was selective, thereby causing a measurable variation in the triglyceride profiles.

Pickul et al. (1985) reported that the average fat content for breast meat was 1.08% and that the fat was composed of 62.6% phospholipids, 32.2.% triglycerides, and 4.8% total cholesterol. Since triglycerides represent a minor constituent of total fats in the chicken breast, it would be anticipated that their partitioning in the frying media is not significant. Moreover, the major triglycerides in chicken fat resemble those available in the shortening.

If it is assumed that all the chicken triglycerides (0.32%) dissolved in the shortening during frying and a portion of the shortening were absorbed into the chicken (Tables 36 and 37), several conclusions can be drawn:

1. By dividing the triglyceride content in chicken nuggets or filets by the triglyceride content in the added shortening in Tables 36 and 37, it was found that more TGs are partitioned into the shortening under pressure cooking than nonpressurized frying. The exchange rate is 1.6-3.6 of chicken triglycerides to 100 absorbed shortening triglycerides under both product conditions.

2. From previous studies, it is well documented that phospholipids are present basically in the cell membrane and during cooking they will not be partitioned from the



chicken meat to the shortening. Hence, their participation in increasing the rate of shortening deterioration is expected to be insignificant. Moreover, under both frying conditions, Total Frying Time (TFT) which is related to the total chicken volume cooked, had no significant effect on changes in triglycerides compositions in all three shortenings. From these perceptions it could be anticipated that if the deterioration rate of frying shortening is enhanced in the presence of chicken nuggets or chicken filets, chicken lipids would not be the enhancing factor. However, the interactions between the shortenings and the ingredients in the fried chicken products are responsible for the production of particular decomposition products that are probably the major cause of shortening deterioration (particularly flavor).

#### 4.1.2.2.9 EFFECT OF COMMERCIAL FRYING CONDITIONS ON CHICKEN NUGGETS FRIED FOR FLAVOR.

A sensory evaluation of rancid flavor intensity of lightly salted chicken nuggets fried in previously used P/O, CS/OLS and SB/OLS shortenings was reported in Table 40 for chicken nugget (Table 40) and for chicken filet (Table 41) frying conditions. Average ratings for unheated shortenings were 8.50, 8.70 and 8.63 for P/O, CS/OLS and

SB/OLS, respectively. Under chicken nugget frying conditions, from the GLM procedure, it was found that the independent variables, heating time (DAYS), Total Frying Time (TFT), weight (lb) of chicken product (CHKW) and weight (lb) of used shortening (OILW), had significant effects on the ratings of rancid flavor intensity (Fig. 32 and Table 42), however, shortening type (SHORT.) and THT had no significant effect. Also, a significant ( $P < 0.0001$ ) interaction between DAYS and shortening type was found. The ratings of 7 day commercially used P/O, CS/OLS and SB/OLS shortenings were 7.57, 8.17 and 8.20, respectively. No significant ( $\alpha = 0.05$ ) differences were found between the different shortenings.

Under chicken filet frying conditions, from the GLM procedure, it was found that the independent variables, SHORT., DAYS and THT, had a significant effect while TFT, CHKW and OILW had no significant effects on rancid flavor intensity. Moreover, a significant ( $P < 0.0001$ ) interaction between DAYS and shortening type was found.

No significant ( $\alpha = 0.05$ ) differences were found among shortenings heated to seven days, which indicated that all shortenings performed equally.

From the orthogonal contrast study and under chicken filet frying conditions, it was found that used P/O, CS/OLS and SB/OLS had significantly ( $P < 0.0092$ ,  $0.0001$  and  $0.0384$ , respectively) lower ratings as compared to unheated

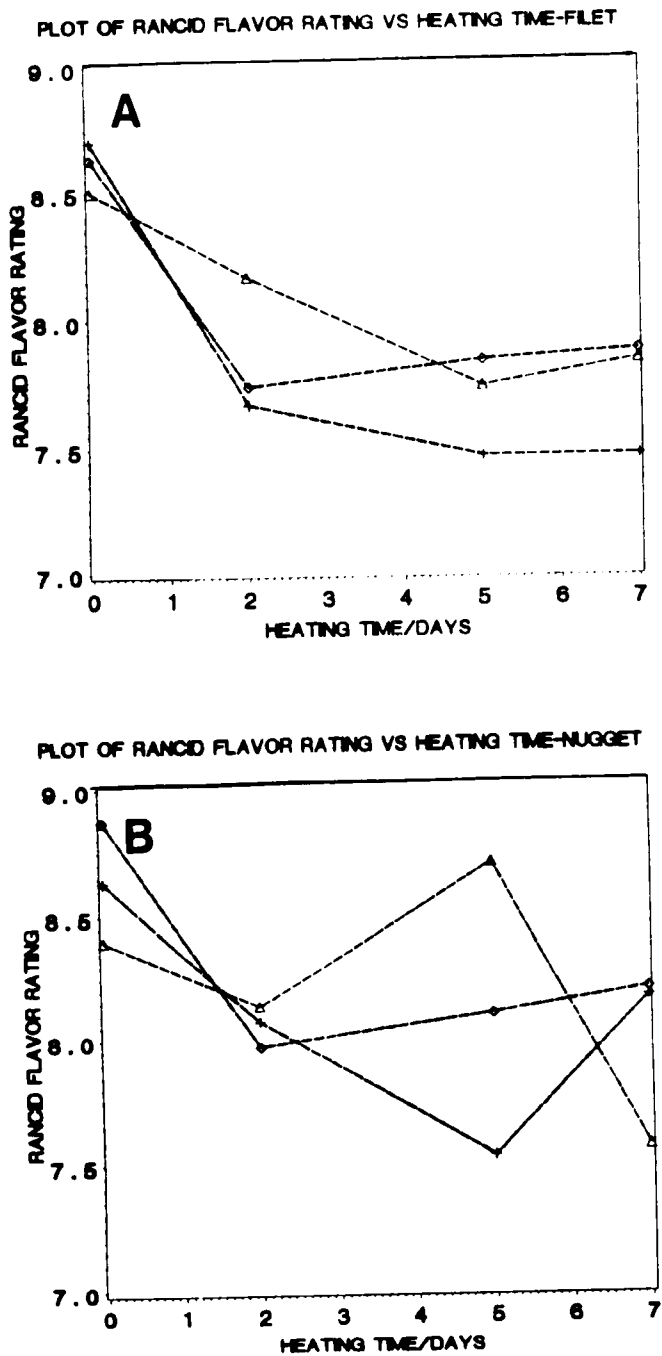


Fig. 32. Effect of Heating Time (Days) on Chicken Nuggets Cooked for Rancid Flavor Intensity Rating of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets. Lower Rating Indicative of More Rancidity.

samples. However, no significant ( $P < 0.0893$  and  $0.6749$ ;  $P < 0.2436$  and  $1.000$ ; and  $P < 0.8092$  and  $0.9449$ , respectively) differences were obtained among used samples. Moreover, from the orthogonal contrast study under chicken nugget frying conditions, it was found that used samples of P/O and CS/OLS had no significant ( $P < 0.3975$  and  $0.1796$ , respectively) differences in the ratings when compared with the fresh shortening samples, however, fresh SB/OLS had significantly ( $P < 0.0002$ ) higher flavor ratings as compared to used samples. Among the used samples of P/O, CS/OLS and SB/OLS, heated until 2 and 5 days, no significant ( $P < 0.1549$ ,  $0.3925$  and  $0.4404$ , respectively) differences in the flavor ratings were found. Similarly, insignificant ( $P < 0.3174$  and  $0.6248$ ) differences were obtained between samples of CS/OLS and SB/OLS used to 5 and 7 days, however, P/O samples used for 7 days had average flavor ratings significantly ( $P < 0.0101$ ) lower than the samples used to 5 days only.

A greater variation in the flavor ratings was obtained under the commercial frying conditions. The flavors of peanut oil and soybean shortening were almost identical under the filet cooking conditions, however, cottonseed oil shortening had the lowest ratings at day five and seven of heating. When these shortenings were used to cook chicken nuggets, peanut oil had the highest ratings at day five while cottonseed oil liquid shortening had the lowest

scores. After seven days of use, the flavor ratings of cottonseed oil liquid shortening was greater than peanut oil.

In general, the flavor, but not the odor of cottonseed oil liquid shortening was more competitive with peanut oil under statically heated conditions. However, there were no differences in odor ratings between CS/OLS and P/O in their performance under commercial frying conditions.

#### 4.1.2.2.10 EFFECT OF COMMERCIAL FRYING CONDITIONS ON THE SHORTENING ODOR.

Average rancid odor intensity scores of fifteen panelists, from unheated P/O, CS/OLS and SB/OLS were reported in Table 40 for chicken nugget and Table 41 for chicken filet frying conditions. From the GLM procedure, significant effects of the independent variables shortening type (SHORT.), heating time (DAYS), weight (lb) of chicken products (CHKW), and weight (lb) of added shortening (OILW), were found on changes in rancid odor intensity ratings (Fig. 33 and Table 42). However, Total Heating Time (THT) and Total Frying Time (TFT) had no significant effects on changes in rancid odor rating. The GLM procedure for testing significance was repeated on the same shortenings which were used as a heating medium for cooking

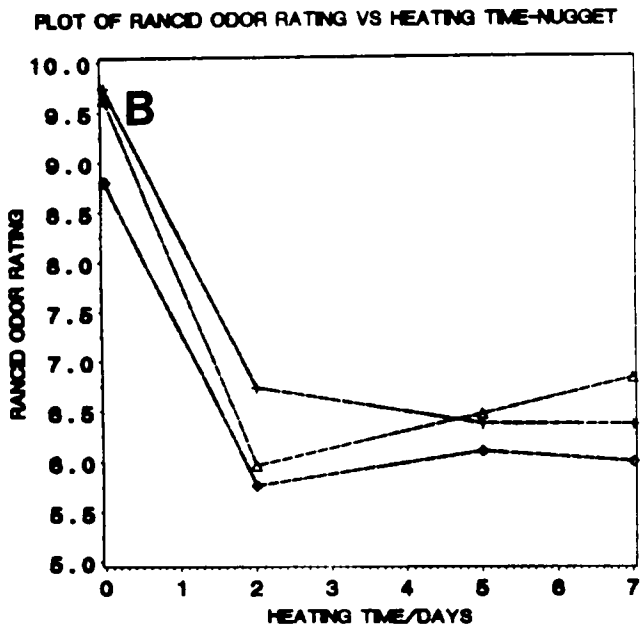
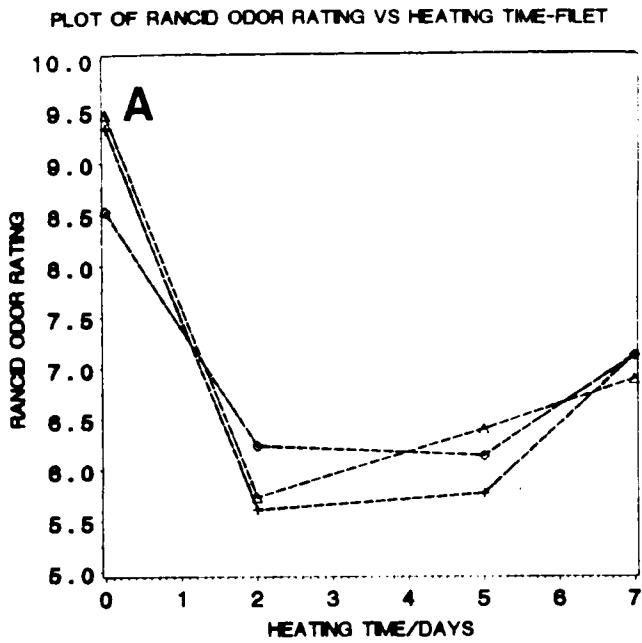


Fig. 33. Effect of Heating Time (Days) on Rancid Odor Intensity Rating of Peanut oils ( $\Delta$ ), Cottonseed (+) and Soybean ( $\diamond$ ) Oil Liquid Shortenings, Used for Commercial Frying of A- Chicken filets B- Chicken Nuggets. Lower Rating Indicative of More Rancidity.

chicken filets. The independent variables, DAYS, THT, TFT and CHKW, had significant effects on the ratings of frying shortenings rancid odor intensity. While SHORT. and OILW had no significant effects on these ratings, a significant interaction between DAYS and shortening type was found. No significant ( $\alpha=0.5$ ) differences in odor ratings of different shortenings heated to seven days under both frying conditions were found. Which indicated that all three shortenings performed equally.

From the orthogonal contrast study of flavor ratings, unused shortening samples had significantly ( $P<0.0001$ ) higher flavor ratings as compared to used samples. Moreover, used P/O samples heated to 5 days, were significantly ( $P<0.0185$ ) higher than the samples heated to 2 days only, which indicated odor instability under these conditions. Nevertheless, no significant ( $P<0.0648$ ) differences between samples heated to 5 and 7 days were found for the same oil. In the case of CS/OLS and SB/OLS, samples heated to 2 and 5 days were not significantly ( $P<0.4435$  and  $0.5538$ ) different in their flavor ratings, but significantly ( $P<0.0001$  and  $0.0004$ ) higher flavor score obtained from samples used to 7 days as compared to samples used to 5 days only. Therefore, all used shortenings did not reach their cut-off level, and were discarded before they were consumed completely.

Under chicken nugget frying conditions, used samples of P/O, CS/OLS and SB/OLS, had significantly ( $P < 0.0001$ ) lower ratings as compared to unused samples. When used samples of P/O, CS/OLS and SB/OLS were compared, no significant ( $P < 0.3043$  and  $0.4529$ ;  $P < 0.4368$  and  $0.8813$ ; and  $P < 0.1438$  and  $0.7072$ , respectively) differences were found between samples heated to 2, 5 and 7 days.

Under the commercial frying conditions of chicken nuggets and filets, sweet, burned and chicken types of odor predominated the odor of all commercially used shortenings. These flavors probably originated from the effect of heating on ingredients in the chicken products.

After two days of shortening use, a combination of sweet, burned and chicken odor caused a reduction in the rancid odor intensity rating. In general, the rancid odor intensity was reduced at day five and seven of shortening use which indicated the instability of the volatiles which are responsible for this odor. It was not possible to obtain linear regressions, or good correlations with heating time. From the behavior of these shortenings, it was expected that the sweet and burned aroma that was generated from the interaction between the shortenings and the cooked chicken products had interfered with the rancid odor rating. Therefore, all shortenings had similar odor ratings and their quality could not be determined on the basis of rancidity alone.



By comparing the results obtained from the static heating analyses with those obtained from the commercial frying results, it was concluded that the primary causative factor of flavor deterioration for any shortening used was the fried food. The shortening would contribute indirectly by interacting with the food ingredients.

#### 4.1.2.2.11 DEVELOPMENT OF PREDICTION EQUATIONS AND ITS APPLICATION IN ASSESSING QUALITY OF FRYING SHORTENING IN TERMS OF NUMBER OF DAYS UNDER THE COMMERCIAL FRYING OF CHICKEN NUGGETS AND FILETS.

Several equations were developed using the Principle Component Regression (PCR) model (Appendix 2) to predict the number of elapsed cooking days which would permit calculating the number of useful days remaining (Tables 49-54).

The prediction equations were developed by considering the objective test as a dependent variable, such as FFA, FOS reading, polar component% and contact angle measurements, however, heating time (DAYS) of use; Total Heating Time (THT); Total Frying Time (TFT); Weight (lb) of chicken nuggets or filets, CHKW; and Weight (lb) of added shortening were independent variables.

Table 49. Equations<sup>1</sup> predicting the useful days remaining for peanut oil used in frying chicken nuggets.

| Tests <sup>2</sup> | EQUATION |                 |                | R <sup>2</sup>    |      |
|--------------------|----------|-----------------|----------------|-------------------|------|
| POLAR <sup>3</sup> | =        | 4.923 + 0.327   | DAYS + 0.3815  | TFT <sup>5</sup>  | 0.86 |
| POLAR <sup>3</sup> | =        | 4.947 + 0.330   | DAYS + 0.0334  | THT <sup>6</sup>  | 0.89 |
| POLAR <sup>3</sup> | =        | 4.856 + 0.329   | DAYS + 0.00365 | CHKW <sup>7</sup> | 0.88 |
| FOS <sup>3</sup>   | =        | 1.183 + 0.0595  | DAYS + 0.0695  | TFT               | 0.88 |
| FOS <sup>3</sup>   | =        | 1.187 + 0.06    | DAYS + 0.0061  | THT               | 0.91 |
| FOS <sup>3</sup>   | =        | 1.171 + 0.06    | DAYS + 0.00067 | CHKW              | 0.90 |
| TLC <sup>3</sup>   | =        | 11.958 + 0.6395 | DAYS + 0.7467  | TFT               | 0.65 |
| TLC <sup>3</sup>   | =        | 11.839 + 0.6427 | DAYS + 0.00713 | CHKW              | 0.66 |
| CON <sup>4</sup>   | =        | 40.94 - 6.994   | DAYS + 9.091   | TFT               | 0.62 |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:  
 POLAR (polar component%).  
 FOS (FoodOil-Sensor reading).  
 TLC (polar component%).  
 CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 2.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total heating time = # hours the shortening is at 325 °F.

<sup>7</sup>: Total chicken nugget weight (lb) fried.

Table 50. Equations<sup>1</sup> predicting the useful days remaining for cottonseed oil liquid shortening used in frying chicken nuggets.

| Tests <sup>2</sup> | EQUATION  | R <sup>2</sup> |
|--------------------|---|----------------|
| FFA <sup>3</sup>   | = 0.0748 + 0.0468 DAYS + 0.0479 TFT <sup>5</sup>  | 0.97           |
| FFA <sup>3</sup>   | = 0.0751 + 0.0465 DAYS + 0.0048 THT <sup>6</sup>  | 0.98           |
| FFA <sup>3</sup>   | = 0.0791 + 0.0469 DAYS + 0.0004 CHKW <sup>7</sup> | 0.96           |
| FFA <sup>3</sup>   | = 0.0754 + 0.0466 DAYS + 0.0041 OILW <sup>8</sup> | 0.98           |
| CON <sup>4</sup>   | = 39.62 + 3.6220 DAYS + 3.619 TFT                 | 0.67           |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:  
 FFA (Free Fatty Acid - % Oleic acid).  
 CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 2.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total heating time = # hours the shortening is at 325 °F.

<sup>7</sup>: Total chicken nugget weight (lb) fried.

<sup>8</sup>: Total shortening added to the fryer (lb).

Table 51. Equations<sup>1</sup> predicting the useful days remaining for soybean oil liquid shortening used in frying chicken nuggets.

| Tests <sup>2</sup> | EQUATION |         |          |                | R <sup>2</sup>    |      |
|--------------------|----------|---------|----------|----------------|-------------------|------|
| FFA <sup>3</sup>   | =        | 0.022   | + 0.051  | DAYS + 0.049   | TFT <sup>5</sup>  | 0.72 |
| FFA <sup>3</sup>   | =        | 0.03368 | + 0.051  | DAYS + 0.00041 | CHKW <sup>6</sup> | 0.71 |
| TLC <sup>4</sup>   | =        | 15.873  | - 2.1476 | DAYS + 2.4446  | TFT               | 0.89 |
| CON <sup>4</sup>   | =        | 41.531  | - 5.94   | DAYS + 5.63    | TFT               | 0.93 |
| CON <sup>4</sup>   | =        | 42.478  | - 5.8533 | DAYS + 0.047   | CHKW              | 0.92 |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:  
 FFA (Free Fatty Acid - % Oleic acid).  
 TLC (polar component%).  
 CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 2.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total chicken nugget weight (lb) fried.

Table 52. Equations<sup>1</sup> predicting the useful days remaining for peanut oil used in frying chicken filets.

| Tests <sup>2</sup> | EQUATION |          |                           |                   | R <sup>2</sup> |
|--------------------|----------|----------|---------------------------|-------------------|----------------|
| FFA <sup>3</sup>   | =        | 0.00504  | + 0.11443 DAYS + 0.0847   | TFT <sup>5</sup>  | 0.83           |
| FFA <sup>3</sup>   | =        | 0.003186 | + 0.1161 DAYS + 0.0119    | THT <sup>6</sup>  | 0.85           |
| FFA <sup>3</sup>   | =        | 0.0030   | + 0.11387 DAYS + 0.001375 | CHKW <sup>7</sup> | 0.81           |
| FFA <sup>3</sup>   | =        | 0.0051   | + 0.1131 DAYS + 0.0129    | OILW <sup>8</sup> | 0.80           |
| POLAR <sup>3</sup> | =        | 4.51     | + 0.5957 DAYS + 0.441     | TFT               | 0.97           |
| POLAR <sup>3</sup> | =        | 4.55     | + 0.5969 DAYS + 0.061     | THT               | 0.97           |
| POLAR <sup>3</sup> | =        | 4.481    | + 0.5948 DAYS + 0.0072    | CHKW              | 0.96           |
| POLAR <sup>3</sup> | =        | 4.482    | + 0.5924 DAYS + 0.0675    | OILW              | 0.95           |
| TLC <sup>3</sup>   | =        | 12.518   | + 1.011 DAYS + 0.7487     | TFT               | 0.82           |
| TLC <sup>3</sup>   | =        | 12.61    | + 1.011 DAYS + 0.1034     | THT               | 0.82           |
| TLC <sup>3</sup>   | =        | 12.456   | + 1.0124 DAYS + 0.0122    | CHKW              | 0.82           |
| TLC <sup>3</sup>   | =        | 12.51    | + 1.001 DAYS + 0.0582     | OILW              | 0.80           |
| FOS <sup>3</sup>   | =        | 1.196    | + 0.0894 DAYS + 0.0662    | TFT               | 0.93           |
| FOS <sup>3</sup>   | =        | 1.2034   | + 0.0894 DAYS + 0.00915   | THT               | 0.94           |
| FOS <sup>3</sup>   | =        | 1.19     | + 0.0895 DAYS + 0.0011    | CHKW              | 0.93           |
| FOS <sup>3</sup>   | =        | 1.1932   | + 0.08875 DAYS + 0.010    | OILW              | 0.91           |
| CON <sup>4</sup>   | =        | 41.959   | - 5.107 DAYS + 4.381      | TFT               | 0.56           |
| CON <sup>4</sup>   | =        | 41.88    | - 3.329 DAYS + 0.0502     | CHKW              | 0.55           |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:

FFA (Free Fatty Acid - % Oleic acid).  
 POLAR (polar component%).  
 TLC (polar component%).  
 FOS (FoodOil-Sensor reading).  
 CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 3.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total heating time = # hours the shortening is at 325 °F.

Table 52. cont.

- 
- 7: Total chicken nugget weight (lb) fried.
- 8: Total shortening added to the fryer (lb).

Table 53. Equations<sup>1</sup> predicting the useful days remaining for cottonseed oil liquid shortening used in frying chicken filets.

| Tests <sup>2</sup> | EQUATION |          |           |                | R <sup>2</sup> |      |
|--------------------|----------|----------|-----------|----------------|----------------|------|
| FFA <sup>3</sup>   | =        | -0.0357  | + 0.1252  | DAYS + 0.0771  | TFT            | 0.84 |
| FFA <sup>3</sup>   | =        | -0.0584  | + 0.1276  | DAYS + 0.01295 | THT            | 0.88 |
| FFA <sup>3</sup>   | =        | -0.03539 | + 0.1252  | DAYS + 0.00121 | CHKW           | 0.84 |
| FFA <sup>3</sup>   | =        | -0.06272 | + 0.1312  | DAYS + 0.0132  | OILW           | 0.92 |
| FOS <sup>3</sup>   | =        | 2.649    | + 0.07395 | DAYS + 0.0455  | TFT            | 0.97 |
| FOS <sup>3</sup>   | =        | 2.642    | + 0.0744  | DAYS + 0.00755 | THT            | 0.98 |
| FOS <sup>3</sup>   | =        | 2.649    | + 0.0739  | DAYS + 0.00072 | CHKW           | 0.97 |
| FOS <sup>3</sup>   | =        | 2.6494   | + 0.0751  | DAYS + 0.00756 | OILW           | 1.00 |
| POLAR <sup>3</sup> | =        | 9.9178   | + 0.4956  | DAYS + 0.0503  | THT            | 0.82 |
| POLAR <sup>3</sup> | =        | 9.935    | + 0.513   | DAYS + 0.05164 | OILW           | 0.87 |
| TLC <sup>3</sup>   | =        | 22.369   | + 0.660   | DAYS + 0.4067  | TFT            | 0.76 |
| CON <sup>4</sup>   | =        | 38.8735  | + 10.013  | DAYS - 0.0953  | CHKW           | 0.66 |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:

- FFA (Free Fatty Acid - % Oleic acid).
- POLAR (polar component%).
- TLC (polar component%).
- FOS (FoodOil-Sensor reading).
- CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 3.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total heating time = # hours the shortening is at 325 °F.

<sup>7</sup>: Total chicken nugget weight (lb) fried.

<sup>8</sup>: Total shortening added to the fryer (lb).

Table 54. Equations<sup>1</sup> predicting the useful days remaining for soybean oil liquid shortening used in frying chicken filets.

| Tests <sup>2</sup> | EQUATION |          |          |                | R <sup>2</sup>    |      |
|--------------------|----------|----------|----------|----------------|-------------------|------|
| FFA <sup>3</sup>   | =        | -0.0521  | + 0.1453 | DAYS + 0.075   | TFT <sup>5</sup>  | 0.95 |
| FFA <sup>3</sup>   | =        | -0.0794  | + 0.1425 | DAYS + 0.0139  | THT <sup>6</sup>  | 0.92 |
| FFA <sup>3</sup>   | =        | -0.04648 | + 0.1461 | DAYS + 0.00119 | CHKW <sup>7</sup> | 0.95 |
| FFA <sup>3</sup>   | =        | -0.0585  | + 0.1437 | DAYS + 0.0124  | OILW <sup>8</sup> | 0.93 |
| CON <sup>4</sup>   | =        | 41.732   | - 6.0162 | DAYS - 3.3096  | TFT               | 0.93 |
| CON <sup>4</sup>   | =        | 42.01    | - 5.9977 | DAYS - 0.0519  | CHKW              | 0.94 |
| TLC <sup>4</sup>   | =        | 17.059   | - 2.2107 | DAYS + 1.81    | TFT               | 0.82 |

<sup>1</sup>: Developed using Principle Component Regression (PCR) model.

<sup>2</sup>: Tests:

- FFA (Free Fatty Acid - % Oleic acid).
- POLAR (polar component%).
- TLC (polar component%).
- FOS (FoodOil-Sensor reading).
- CON (Contact angle measurement).

<sup>3</sup>: One component dropped from the model.

<sup>4</sup>: No component dropped from the model.

<sup>5</sup>: Total frying time = # batches X 3.5 minutes / 60 minutes / hr.

<sup>6</sup>: Total heating time = # hours the shortening is at 325 °F.

<sup>7</sup>: Total chicken nugget weight (lb) fried.

<sup>8</sup>: Total shortening added to the fryer (lb).



In general, the best fit of the linear regression model was developed by dropping one or none of the components in order to obtain maximum  $R^2$  while maintaining the (t) test, between the independent and dependent variables, significant.

Table 49 contains several prediction equations used to evaluate the actual number of usable days remaining on using peanut oil that was used to fry chicken nuggets. The first equation has three variables, TFT, DAYS and POLAR. By replacing the values for TFT and POLAR in the equation, the actual number of frying days for peanut oil is determined. Therefore, the number of useful days remaining for the peanut oil is estimated.

These equations will provide a restaurant operator with a very useful tool to determine the optimum frying shortening use. It is possible that the equation could be introduced into the counter register so that the product volume and total frying time could be calculated on a real time bases.

The most recommended equations to be used with peanut oil are:  $FOS = 1.711 + 0.06 \text{ DAYS} + 0.00067 \text{ CHKW}$  for chicken nugget, and  $FOS = 1.19 + 0.0895 \text{ DAYS} + 0.0011 \text{ CHKW}$ , for chicken filet. In these equations, chicken weight (CHKW) was estimated during a high volume season, such as Christmas. Therefore, one working day in the equation (DAYS) is defined as one working day during a holiday

season. Accordingly, the cut-off level of FoodOil- Sensor reading is determined during Christmas, resulting in longer and more efficient use of the peanut oil off seasons. Because more often, the frying oil was discarded at early stages of use, particularly under chicken nugget frying conditions.

#### 4.1.2.2.12 OBSERVATIONS OBTAINED FROM THE RESTAURANT.

- a. Frying shortening replenishment for peanut oil (P/O), cottonseed liquid shortening (CS/OLS) and soybean oil shortening (SB/OLS) was reported in Tables 36 and 37 for both chicken products.
- b. Flavor desirability of cooked chicken products in addition to the odor of the shortenings were more preferred for P/O > CS/OLS > SB/OLS.
- c. Frying both chicken products with CS/OLS caused flavor retention under the coating of chicken filets and nuggets. This flavor is released suddenly after the first bite.
- d. The flavor of nuggets or filets cooked in CS/OLS was identical to those cooked in P/O. However cooking with

SB/OLS generated chicken flavors identical to Kentucky Fried Chicken products, however, a fishy aroma was generated from SB/OLS after 7 DAYS of use.

e. Gum formation at the shortening, air and vessel interface was maximum for CS/OLS > SB/OLS > P/O.

f. Fat crystals in the soybean oil liquid shortening required a higher initial temperature and longer time to melt. Nevertheless after melting, the shortening had the highest rate of thermal conductivity, because less time was required to cook the chicken products.

g. Shortenings used to fry chicken filets were much darker in color than those used to fry chicken nuggets. Moreover, as a result of longer shortening use, their color had changed from light yellow or green to dark brown. Therefore, shortenings statically heated to 14 days were lighter in color (dark orange) than those used under commercial frying conditions (dark brown). This indicated that the degradation products of the coating ingredients contributed to the browning process of the used shortenings.

4.1.2.2.13 FINAL COMMENTS.

a. Although there is no reason for searching for a universal cut-off level of frying oil quality, the modified procedure of dielectric constant test (FoodOil-Sensor test) may be considered the only candidate to be a universal test.

b. It is now appropriate that the degradation products of carbohydrates and proteins be identified in order to evaluate new factors affecting oil stability.

c. Since maximum abuse of frying oil is occurring during holidays, as Christmas, therefore, the cut-off level for any frying oil quality under any conditions, should be established during these periods; accordingly, safer and longer use of the frying oils during non-holiday seasons are guaranteed.

CHAPTER V  
CONCLUSIONS

Several conclusions could be obtained from this study:

1. Under static heating conditions, varying heating periods or shortening types had significant ( $P < 0.0001$ ) effects on the resulting data of the following tests: free fatty acid; polar component; total volatiles; dielectric constant; viscosity; polar component% by Quantitative High Performance Thin Layer Chromatography; contact angle and sensory analyses of odor and flavor; but heating time had no significant ( $P < 0.999$ ) effect on triglyceride profiles.

2. Under commercial frying conditions, less shortening was absorbed by pressure cooked chicken filets, therefore, shortening yield was significantly ( $P < 0.0001$ ) higher as compared to the open vessel frying of chicken nuggets. On the average, the shortening yield was decreased in the following order: CS/OLS > P/O > SB/OLS under chicken nugget frying conditions and CS/OLS > SB/OLS > P/O under chicken filet frying conditions. Also, under chicken nugget frying conditions, heating time (DAYS) had significant effects on shortening yields in case of P/O and SB/OLS but not with CS/OLS. Furthermore, under chicken filet frying conditions, heating time had significant effects on shortening yield in case of P/O and CS/OLS but not SB/OLS.

Therefore, product type, shortening type and heating time effect the shortening yield hence quantity of shortening absorbed.

The effect of frying conditions on the moisture content of the fried products using P/O only was investigated. From this study it was confirmed that more moisture is removed from chicken nuggets than from chicken filets; hence, less shortening yield and more shortening was absorbed under chicken nugget frying conditions. Similar results are expected from different shortening under similar conditions.

3. The rate of changes in polar component%, polar component% by QHPTLC, dielectric constant or viscosity varied significantly among different shortenings under both chicken nugget and filet frying conditions. However, the rate of changes in free fatty acid%, odor ratings varied significantly under chicken nuggets frying conditions; but were insignificant under chicken filet frying conditions. Heating time (DAYS) had a significant effect on changes in dielectric constants, free fatty acid%, viscosity, contact angle and odor rating under both frying conditions, and had a significant effect on polar component% under chicken nugget frying conditions only.

4. Under commercial frying conditions, polar component percent and polymers (measured from increases in viscosity) accumulated to a lesser extent as compared to static

heating conditions. However, the free fatty acid accumulation showed the opposite behavior.

5. From the volatile profile analysis under static heating conditions, it was found that the increase in total volatile peak areas can be used to predict the heat abuse of CS/O, CS/OLS, P/O and SB/OLS but not C/O. Pentane can be used for C/O, CS/OLS, P/O and SB/OLS, however pentanal can be used with CS/O, CS/OLS and P/O. Both t,t-2,4-decadienal and t,c-2,4-decadienal can be used with CS/O, CS/OLS and SB/OLS, however, only t,t-2,4-decadienal can be used with C/O to predict heat abuse. Unfortunately, no single index could be used to predict heat abuse of all five shortenings. A correlation coefficient range of 0.82 to 0.99 was obtained between these indices and heating time. Nevertheless, volatile profile analysis was not conducted under commercial frying conditions because of time limitation.

6. The changes in the dielectric constant procedure were successful in providing absolute values which produced high correlations ( $r=1.00$ ) with the polar component test in all five shortenings under static heating conditions. From the dielectric constant test it was found that the susceptibility of shortening samples to oxidation evaluated after eleven days of static heating was in the following increasing order: SB/OLS, P/O, C/O, CS/OLS and CS/O. This indicates that SB/OLS was the most stable under the static

heating conditions. However, when these samples were heated to fourteen days, the FOS values of P/O became identical to that of C/O, while the FOS value of CS/OLS exceeded that of CS/O's. This behavior was observed also in the polar component test.

7. The increase in viscosity (polymer accumulation), could predict shortening heat abuse under static heating conditions, however it failed to do so under commercial frying conditions; maybe because polymers were synthesized at lowers, absorbed by the chicken products or adsorbed onto the gum region of the frying vessel.

The initial and final viscosity values of all five shortenings were related under both static and commercial frying conditions. Therefore, the initial concentration of polymers, triglyceride quality and quantity and the heating conditions (static vs commercial) will effect the accumulation rate of polymers in the heating media. Furthermore, the polymerization rates for both CS/OLS >> SB/OLS were significantly higher than those of C/O, CS/O and P/O; which indicates the possible involvement of fat crystals in the polymerization process.

8. A new Quantitative High Performance Thin Layer Chromatographic method was developed to evaluate changes in polar component% in the used shortenings, and showed good correlations ( $r=0.97-1.00$ ) with the polar component test under static heating conditions. The test is fast and



requires less reagents but expensive instrumentation. Furthermore, different HPTLC plates had significant ( $P < 0.0001$ ) effects on the polar component% under static heating and commercial frying conditions, which indicated that the problem of reproducibility among HPTLC plates has not been absolutely solved. The actual polar component% could not be obtained by the QHPTLC procedure, because it magnified the polar component% values for all samples, particularly for the initial values of P/O and CS/O from 5.56% and 9.33% to 21.38% and 29.65%, respectively. Therefore, establishing standard curve between both tests under specific conditions would facilitate providing more accurate information.

9. A new contact angle measurement technique was developed to predict the heat abuse of frying shortenings. The measurement of the shortening contact angle on the FT-120-WW surface is simple, requires only five minutes waiting period, however, several days needed to prepare the surface for testing. The method exhibited more success with peanut oil under commercial frying conditions, However, more research is required before the test can be successfully introduced to the food industry.

Since used frying oil is complex mixture of multiple compounds, it unclear how different lipid classes are aligned on the FT-120-WW surface. Peanut oil was the only oil which had significant linear increase in its contact

angle and having good correlation ( $r=0.81$ ) with heating time. Both CS/O and CS/OLS had identical contact angle behavior indicating that the liquid phase only and to a great extent the triglyceride pattern affected the final contact angles on this surface. Although several factors affecting the measurements were standardized, the search for better surface which does not require preparation should be continued.

10. Aromas as, sweet, chicken and burnt odors of shortenings used under commercial frying conditions as compared to rancid odors produced under static heating conditions. From the sensory analyses under both heating conditions: volatiles produced from the interaction between food ingredients and shortenings were probably more important than the volatiles produced from the statically heated shortenings in reducing flavor quality of the tested shortenings; because rancid flavor accumulation under static heating condition was not objectionable by the panelists even after 14 days of heating. In general, the flavor of cottonseed oil liquid shortening was competitive with that of peanut oil under the same heating conditions. From the orthogonal contrast study, no significant differences among heated and unheated samples of C/O, CS/O, P/O, and SB/OLS were found, as well as, among heated samples of CS/OLS but not between unheated and heated

samples CS/OLS. This finding indicated that off-flavor is not necessarily produced from merely heating the oil.

It is important to mention that shortening heated to fourteen days, produced significant foaming as compared to shortenings heated to a lesser extent.

After eleven days of static heating, the flavor rating of SB/OLS was significantly lower than all other shortenings. However, no significant differences in flavor scores were observed after five days of heating only.

From the orthogonal contrast study of odor rating, significant differences were found between unheated and heated samples. In general, C/O, CS/O and CS/OLS had stable odor up to fourteen days of static heating; however, P/O odor deteriorated after five days of heating. SB/OLS had the lowest rating after two days of heating but it improved thereafter. Nevertheless, the odor ratings of SB/OLS were the lowest amongst other shortenings after five and eleven days of heating. These shortenings showed no significant differences in their odor rating after five days. However, no significant differences among P/O, CS/O and CS/OLS; were also found among C/O, CS/O, CS/OLS after eleven days of static heating.

11. The effects of heating periods and chicken fat on the changes in triglyceride concentrations were investigated using a high temperature gas chromatographic procedure. The chromatographic conditions of temperature

and pressure were improved in order to provide the best triglyceride separations.

The rate of deterioration of each triglyceride is controlled by the availability of other types of triglycerides in a particular shortening, the heating conditions; the degree of unsaturation; the molecular weight; and the interaction among all these factors. The stereochemistry of polyunsaturated fatty acid in triglycerides is not necessarily an important factor in determining the susceptibility to oxidation.

In general, heating time had no significant effect on changes in triglyceride profile for non of the five shortenings used under the static heating conditions.

From C/O, CS/O, P/O, CS/OLS and SB/OLS, 14, 20, 27, 23 and 17 triglycerides, were separated in less than 10, 9, 9, 15 and 10 minutes, respectively. Under static heating conditions: triglycerides with 1 to 3, 1 to 4, 0 to 4, 1 to 5 and 1 to 5 double bonds showed accumulation in C/O, CS/O, CS/OLS, P/O and SB/OLS, respectively. While those with 2 to 7, 1 to 6, 1 and 4 to 6, 2 to 6, and 3 to 6 double bonds showed a reduction in concentration with prolong heating time. By comparison, it was found that triglycerides with 4 to 7, 5 to 6, 5 to 6, 6 and 6 double bonds were unstable toward oxidative, hydrolytic and/or thermal degradations in C/O, CS/O, CS/OLS, P/O and SB/OLS. Therefore, it was not possible to specify the relation between the exact numbers

of double bonds in triglycerides with their rate of deterioration, because it varied among shortenings. LLL with six double bonds was the only unstable triglycerides in all used shortenings.

In corn oil, the presence of palmitic acid (C16:0) in PLP and POP triglycerides provided better protection to unsaturated fatty acids on the sn-2 position when compared to stearic acid (C18:0) in PLS and POS. Furthermore, in cottonseed oil, the presence of C16:0 in POP, PLO and PLL triglycerides provided better protection to unsaturated fatty acids on the sn-2 position as compared to C18:0 in POS, SLO and SLL triglycerides. This phenomenon was not observed in either cottonseed or soybean oil liquid shortenings. In peanut oil, however, C16:0 and C18:0 provided better protection to unsaturated fatty acids on the sn-2 position of triglycerides as compared to arachidic acid C20:0, behenic acid C22:0 and lignoceric acid C24:0. Therefore, it is concluded that short chain saturated fatty acids, could provide better protection to unsaturated fatty acids when compared with long chain saturated fatty acids. In other words, the molecular weight of triglycerides may effect their stability towards oxidation with specific types of shortenings.

The presence of OLL and LLL in high concentration in CS/O and CS/OLS, may have contributed to their lower

oxidative stabilities (high polar component%, total volatiles, dielectric constant and contact angle).

Under commercial frying conditions, 30, 24 and 19 triglycerides were separated from unheated P/O, CS/OLS and SB/OLS in less than 14, 9 and 10 minutes, respectively. Furthermore, it was found that heating time (DAYS) had no significant effect on changes in triglycerides.

Under commercial frying conditions, triglycerides with 1-6 double bonds were increased in concentrations while those with 1-5 double bonds decreased in concentrations for peanut oil used to fry chicken nuggets; also triglycerides with 2-6 double bonds increased in concentrations, but other triglycerides with 1-4 double bonds decreased in concentrations for peanut oil used to fry chicken filets. Therefore, triglycerides with 6 double bonds under chicken nugget frying conditions; however, under chicken filet frying conditions, triglycerides with 5 and 6 double bonds showed accumulation indicating that the higher the degree of unsaturation the higher is the relative triglyceride stability under these conditions.

In general, C16:0 provided better protection to unsaturated fatty acids on sn-2 as compared to C18:0, C20:0, C22:0 and C24:0 fatty acids.

Total Frying Time (TFT) had no significant effect on changes in triglyceride profiles. This may due to the low

concentration of triglycerides (0.32%) in the chicken filets.

From these findings and from the changes in triglyceride patterns, it was concluded that chicken fat does not contribute significantly to the triglyceride profiles of the studied shortenings.

Nevertheless, no significant or major changes in triglyceride compositions were noticed in cottonseed oil liquid shortening used to fry either chicken filets or nuggets, since PLL, OLL and LLL were the only triglycerides to be reduced (insignificantly) in peak areas. In the case of soybean oil liquid shortening, which was used to cook chicken nuggets, it was noticed that triglycerides with 1-6 double bonds increased in concentrations, while those with 2-7 double bonds decreased in concentrations, which indicated that triglycerides with 7 double bonds were not stable. When the same shortening was used to fry chicken filets, triglycerides with 1-4 double bonds increased in concentrations while those with 2-7 double bonds decreased in their relative concentrations, which indicated that triglycerides with 5,6 and 7 double bonds were not stable under the investigated conditions. Therefore, it can be concluded that a combination of shortening type, frying conditions and product type affect the modification of triglyceride profiles. Finally, since chicken products do not significantly provide the shortenings with specific

triglycerides, the possibility of selective absorption of some triglycerides should not be exempted.

12. From the correlation coefficient study, it was observed that shortenings containing high numbers of triglycerides or fatty acids, such as peanut oil, are more capable of exhibiting a significantly linear regression of any analytical data. Therefore, it is easier to predict heat abuse of peanut oil with twenty seven total triglycerides (24 high correlation coefficients), as compared to corn oil which has fourteen triglycerides (13 high correlation coefficients). Therefore, the higher the number of triglycerides in any shortening the more sensitive it is to the heat treatment at any level. Hence, the linear are the analytical data.

13. Several equations were developed to determine the useful days remaining to discard shortening under commercial frying conditions.

14. under both heating conditions: the best index to evaluate shortening quality was FoodOil-Sensor readings (dielectric constant), which was followed by the free fatty acid test. The most recommended prediction equations to be used with peanut oil under commercial frying conditions are as follows:  $FOS=1.711+0.06 \text{ DAYS}+0.00067 \text{ CHKW}$ , to be used with chicken nuggets, and  $FOS=1.19+0.0895 \text{ DAYS}+0.0011 \text{ CHKW}$ , to be used with chicken filet.



15. After fourteen days of static heating conditions SB/OLS had the lowest polar component% (29.51%) followed by C/O (32.74%), P/O (36.35%), CS/O (37.84%) and CS/OLS (41.89%). However, no significant differences in flavor ratings among C/O, CS/O, CS/OLS and P/O were observed between samples heated to eleven days, but the flavor rating of SB/OLS was significantly lower than all the five shortenings. After seven days of shortening use under chicken nugget and filet frying conditions, the polar component percentages of peanut oil were 9.2 and 12.96%, respectively, and of SB/OLS were 9.01 and 13.47%, respectively, had no significant ( $\alpha=0.5$ ) differences; however, the polar component percentages of CS/OLS under both frying conditions were significantly ( $\alpha=0.5$ ) higher than those of P/O and SB/OLS. However, no significant ( $\alpha=0.5$ ) differences in flavor and odor ratings among shortenings heated to seven days under both frying conditions. From the orthogonal contrast study it was concluded that all three shortenings were discarded before they were used completely.

16. Several observations concerning the performance of P/O, CS/OLS and SB/OLS during commercial frying conditions, were obtained from the restaurant operator, the workers and the author. From flavor evaluation study, of both chicken products, conducted at the restaurant, it was concluded that CS/OLS was much better substitute for P/O than SB/OLS.

17. Using P/O, CS/OLS and SB/OLS for 7 days under commercial frying conditions could not provide enough information to predict heat abuse (cut-off level), because longer heating periods were needed to fulfill this goal. Therefore, the used shortenings were discarded before they were consumed completely.

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APPENDIX

A

Fatty acid Nomenclature of Triglycerides

Table 1. Fatty acid Nomenclature of Triglycerides

---

|     |                  |                      |
|-----|------------------|----------------------|
| M:  | myristic acid    | (C <sub>14</sub> :0) |
| P:  | palmitic acid    | (C <sub>16</sub> :0) |
| Po: | palmitoleic acid | (C <sub>16</sub> :1) |
| S:  | stearic acid     | (C <sub>18</sub> :0) |
| O:  | oleic acid       | (C <sub>18</sub> :1) |
| L:  | linoleic acid    | (C <sub>18</sub> :2) |
| Ln: | linolenic acid   | (C <sub>18</sub> :3) |
| A:  | arachidic acid   | (C <sub>20</sub> :0) |
| Ga: | gadoleic acid    | (C <sub>20</sub> :1) |
| Be: | behenic acid     | (C <sub>22</sub> :0) |
| Lg: | lignoceric acid  | (C <sub>24</sub> :0) |

---

## APPENDIX

### B

Program used to develop Principal Component Regression Models, which can be used to predict the useful days remaining on frying shortening before it is discarded.

```

1 TITLE 'PRINCIPAL COMPONENTS FOR PEANUT OIL #1 FILET FOS';
2 * THE MACRO VARLIST SHOULD INCLUDE THE DESIRED REGRESSOR VARIABLES;
3 * THE MACRO DEVPAR SHOULD INCLUDE THE DESIRED DEPENDENT VARIABLE;
4 * THE MACRO DATASET SHOULD INCLUDE THE DESIRED DATA SET NAME VARIABLE;
5 MACRO VARLIST DAY TFT % MACRO DATASET ONE %
6 MACRO DEVPAR FOS %
7 MACRO PCR
8 PROC MATRIX ;
9   FETCH X COLNAME=XNAMES DATA=DATASET (KEEP=VARLIST);
10  FETCH Y COLNAME=YNAME DATA=DATASET (KEEP=DEVPAR);
11  VARNAMES=XNAMES||YNAME;
12  * XY IS THE COMPLETE DATASET;
13  XY=X||Y; P=NCOL(X); * P IS THE NUMBER OF MODEL VARIABLES;
14  P1=P+1;PP=3;PP=PP||P;
15  *PP IS THE MAXIMUM NUMBER OF PRINCIPAL COMPONENTS THAT CAN BE REMOVED;
16  N=NROW(XY);* N IS THE NUMBER OF OBSERVATIONS IN THE DATASET USED;
17  MEAN=XY(+, )#/N; XY=XY-J(N,1)*MEAN;
18  * XY HAS NOW BEEN CORRECTED FOR THE MEAN;
19  SS=SQRT(XY(##, ));
20  * SS CONTAINS THE CORRECTED SUMS OF SQUARES OF EACH VARIABLE;
21  Z=Y;* Z IS THE Y VECTOR NOT CORRECTED FOR THE MEAN;
22  XY=XY#/(J(N,1)*SS); Y=XY(*,P1);* Y IS NOW THE STANDARDIZED Y;
23  X=XY(*,1:P);* X IS THE STANDARDIZED X MATRIX;
24  XPX=X'*X;* XPX IS IN THE FORM OF A CORRELATION MATRIX;
25  EIGEN E M XPX; W=X#M;
26  NOTE X IS THE SCALED X MATRIX;
27  NOTE XPX IS THE CORRELATION MATRIX;PRINT XPX COLNAME=XNAMES;
28  NOTE E IS THE VECTOR OF EIGENVALUES; PRINT E;
29  * M IS THE MATRIX OF COLUMNS OF EIGENVECTORS;
30  NOTE W IS A MATRIX OF PRINCIPAL COMPONENTS ON THE COLUMNS;PRINT W;
31  * TOP IS THE MINIMUM OF 5 OR P, THE NUMBER OF ORIGINAL VARIABLES;
32  * THE FOLLOWING DO LOOP DOES PRINCIPAL COMPONENTS REGRESSION;
33  I=0; TOP=MIN(PP);
34  LOOP: IF I=TOP THEN GO TO LAST;PI=P-I; MV=J(PI,1,0); MMW=J(N,1);
35  M=W(*,1:PI); MPW=W'*M; WPY=W'*Z;MIW=INV(MPW);C=WIW*WPY;
36  WS=W';DO IR=1 TO N;MV(*,1)=WS(*,IR);MT=MV';MMW(IR,1)=MT*MIW*MV;
37  MMW(IR,1)=1-MMW(IR,1)-1#/N;END;
38  * YHAT IS THE PREDICTED Y FROM THE PRINCIPAL COMPONENTS REGRESSION;
39  * YDIFF IS A VECTOR OF THE RESPONSE VARIABLE MINUS IT'S MEAN;
40  * TOTAL IS THE CORRECTED TOTAL SUMS OF SQUARES;

```

```

41 YHAT=W*C+MEAN(1,P1); YDIFF=(Z-MEAN(1,P1))'; TOTAL=ROWSUM(YDIFF#YDIFF);
42 * R IS A VECTOR OF ORDINARY RESIDUALS;
43 * PR IS A VECTOR OF PRESS RESIDUALS;
44 R=Z-YHAT; PR=R#/(MMW;RR=R#R; SSE=ROWSUM(RR'); MSE=SSE#/(N-PI+I);
45 IF I=0 THEN SIGMA=MSE; CP=(1+PI)+(MSE-SIGMA)#(N-PI-1)#/SIGMA;
46 NOTE I IS THE NUMBER OF PRINCIPAL COMPONENTS DROPPED; PRINT I;
47 NOTE C IS THE VECTOR OF REGRESSION COEFFICIENTS OF THE PRINCIPAL COMPONENTS;
48 PRINT C;

49 NOTE SSE IS THE ERROR SUM OF SQUARES; PRINT SSE;
50 NOTE MSE IS THE MEAN SQUARE ERROR; PRINT MSE;
51 NOTE CP IS THE CP STATISTIC; PRINT CP;
52 * SE IS A VECTOR OF STANDARD ERRORS OF PREDICTION;
53 * RSQUARE IS THE COEFFICIENT OF DETERMINATION;
54 SE=SQRT(MSE*(1-MMW)); RSQUARE=(TOTAL-SSE)#/TOTAL;
55 NAMES='Y', 'OILS RES', 'PRESSRES', 'STD ERR';
56 MAT=Z||R||PR||SE; PR=PR#2; PRESS=ROWSUM(PR');
57 PRINT MAT COLNAME=NAMES; PRINT PRESS RSQUARE;
58 B=M(*,1,PI)*C(1,PI,1); BNV=B#/(SS'(1,P,1)); INTERCPT=MEAN(1,P1); I=I+1;
59 INTERCPT=INTERCPT-BNV'*MEAN'(1,P,1);
60 NOTE BNV IS THE VECTOR OF REGRESSION COEFFICIENTS IN THE NATURAL VARIABLES;
61 PRINT BNV INTERCPT;
62 GO TO LOOP; LAST;
63 %
64 DATA ONE;
65 INPUT
66 OBSNO=_N_;
67 IF OIL=1;
68 DAYSQ=DAY*DAY; DC=DAY*CHKV; DF=DAY*TFT; DH=DAY*THT; DO=DAY*OILV;
69 CARDS;

NOTE: DATA SET WORK ONE HAS 16 OBSERVATIONS AND 14 VARIABLES. 164 OBS/TRK.
NOTE: THE DATA STATEMENT USED 0.12 SECONDS AND 132K.

118 ;
119 PROC PRINT;
120 /* DATA FRFOSFL; SET ONE;
121 IF OIL=1;
122 TITLE2 'PEANUT OIL #1';
123 PRESSALL;
124 DATA FRFOSFL; SET ONE;
125 IF OIL=2;
126 TITLE2 'COTTONSEED SHORTENING #2';
127 PRESSALL;
128 DATA FRFOSFL; SET ONE;
129 IF OIL=3;
130 TITLE2 'SOYBEAN SHORTENING #3';
NOTE: THE PROCEDURE PRINT USED 0.12 SECONDS AND 224K AND PRINTED PAGE 1.

```

131 PCR;

NOTE: PROC IML WILL REPLACE PROC MATRIX AFTER VERSION 5.  
NOTE: THE PROCEDURE MATRIX USED 0.41 SECONDS AND 188K AND PRINTED PAGES 2 TO 5.  
NOTE: SAS USED 224K MEMORY.

NOTE: SAS INSTITUTE INC.  
SAS CIRCLE  
PO BOX 8000  
CARY, N.C. 27511-8000

-

APPENDIX

C

Analysis of Volatiles in Vegetable Oils

Table 2. Analysis of volatiles in vegetable oils (Dupuy et al., 1985).

| Volatile compound                 | Retention time in min. |
|-----------------------------------|------------------------|
| Acetaldehyde                      | 3.32                   |
| Pentane <sup>1</sup>              | 8.10                   |
| Hexane + 2-Butanone               | 11.02                  |
| Butanal                           | 11.23                  |
| 2-Butenal                         | 13.32                  |
| Benzene                           | 13.65                  |
| 3-Penten-2-one                    | 14.32                  |
| Acetic acid                       | 14.91                  |
| Pentanal <sup>1</sup>             | 15.39                  |
| Ethylfuran                        | 15.66                  |
| 2-Pentanol                        | 18.52                  |
| 1-Pentanol                        | 19.35                  |
| Toluene                           | 19.75                  |
| 1-Octene                          | 20.61                  |
| Hexanal <sup>1</sup>              | 21.17                  |
| 2-Octene                          | 21.59                  |
| 3-Octene                          | 22.10                  |
| 2-Hexenal                         | 24.68                  |
| Octatriene                        | 25.80                  |
| Nonane                            | 27.80                  |
| Heptanal                          | 27.96                  |
| 2-Butylfuran <sup>1</sup>         | 31.07                  |
| t-2-Heptenal <sup>1</sup>         | 31.85                  |
| 1-Hepten-3-ol                     | 33.37                  |
| 2, 3-Octanedione                  | 33.75                  |
| 2-Octanone                        | 34.23                  |
| t, c-2, 4-Heptadienal             | 34.61                  |
| Octanal                           | 35.01                  |
| t, t-2, 4-Heptadienal             | 35.54                  |
| Limonene                          | 36.83                  |
| 2-Octenal                         | 38.77                  |
| 3, 5-Octadien-2-one               | 40.10                  |
| Nonanal                           | 41.82                  |
| 2-Nonenal                         | 45.43                  |
| Benzothiazole                     | 50.34                  |
| 2-Decenal                         | 51.78                  |
| t, c-2, 4-Decadienal <sup>1</sup> | 53.78                  |
| t, t-2, 4-Decadienal <sup>1</sup> | 55.20                  |
| Tetradecane                       | 58.80                  |
| Pentadecane                       | 63.50                  |
| Hexadecane                        | 67.30                  |

cont.



Table 2 cont.

---

|             |       |
|-------------|-------|
| Heptadecane | 70.45 |
| Octadecane  | 73.95 |

---

<sup>1</sup> Marker volatiles which are produced in significant quantities.

APPENDIX

D

Typical Chromatograms of Frying Oil Volatiles

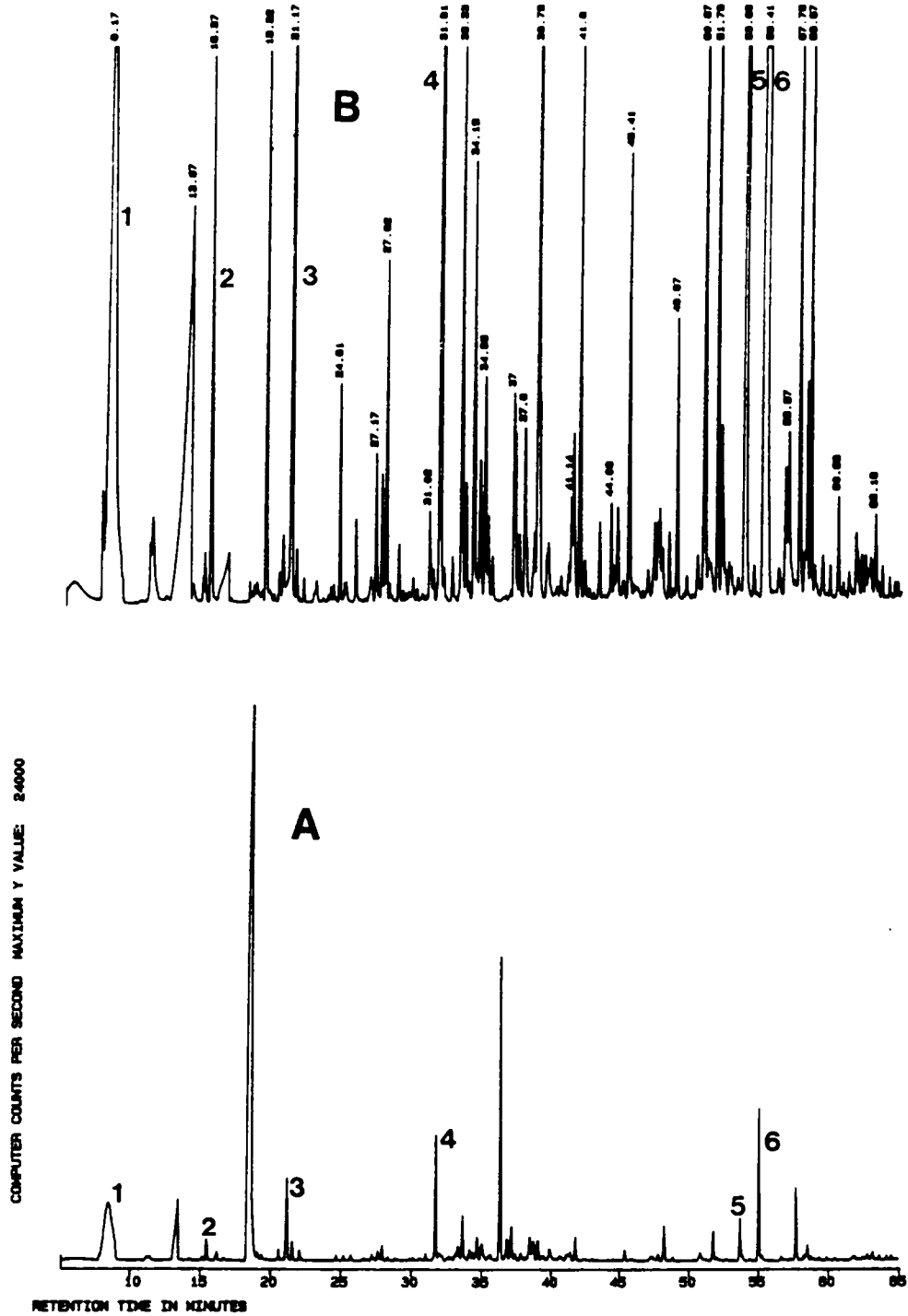


Fig. 1. Effect of static heating time (Days) on Total Volatile Profile in cottonseed oil. A-unheated; B-heated to 14 days. Volatiles: 1=Penane, 2=Penanal, 3=Hexanal, 4=t-2-Heptenal, 5=t,c-2,4-Decadienal and 6=t,t-2,4-Decadienal.

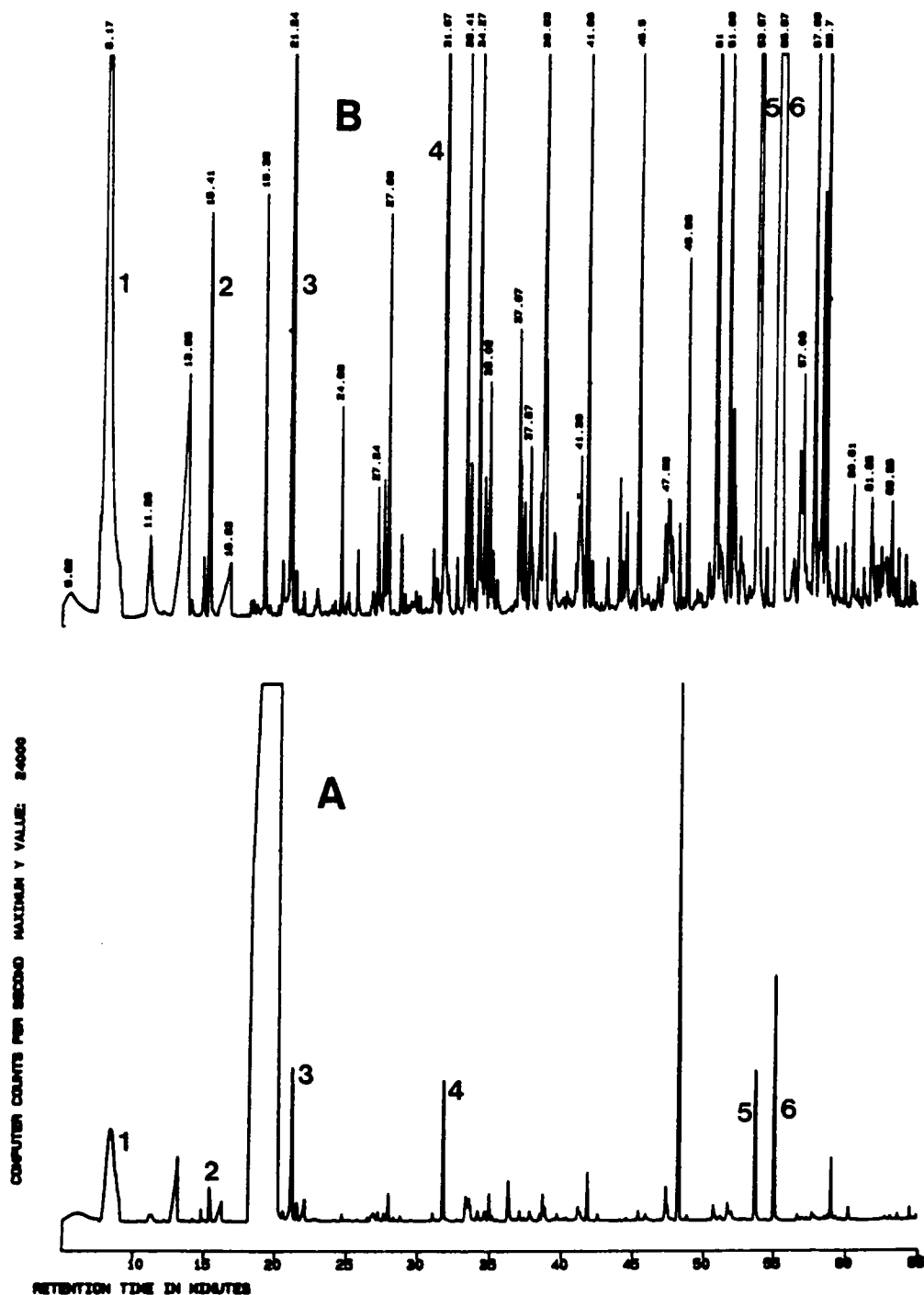


Fig. 2. Effect of static heating time (Days) on Total Volatile Profile in cottonseed oil liquid shortening. A-unheated; B-heated to 14 days. Volatiles: 1=Pentane, 2=Pentanal, 3=Hexanal, 4=t-2-Heptenal, 5=t,c-2,4-Decadienal and 6=t,t-2,4-Decadienal.

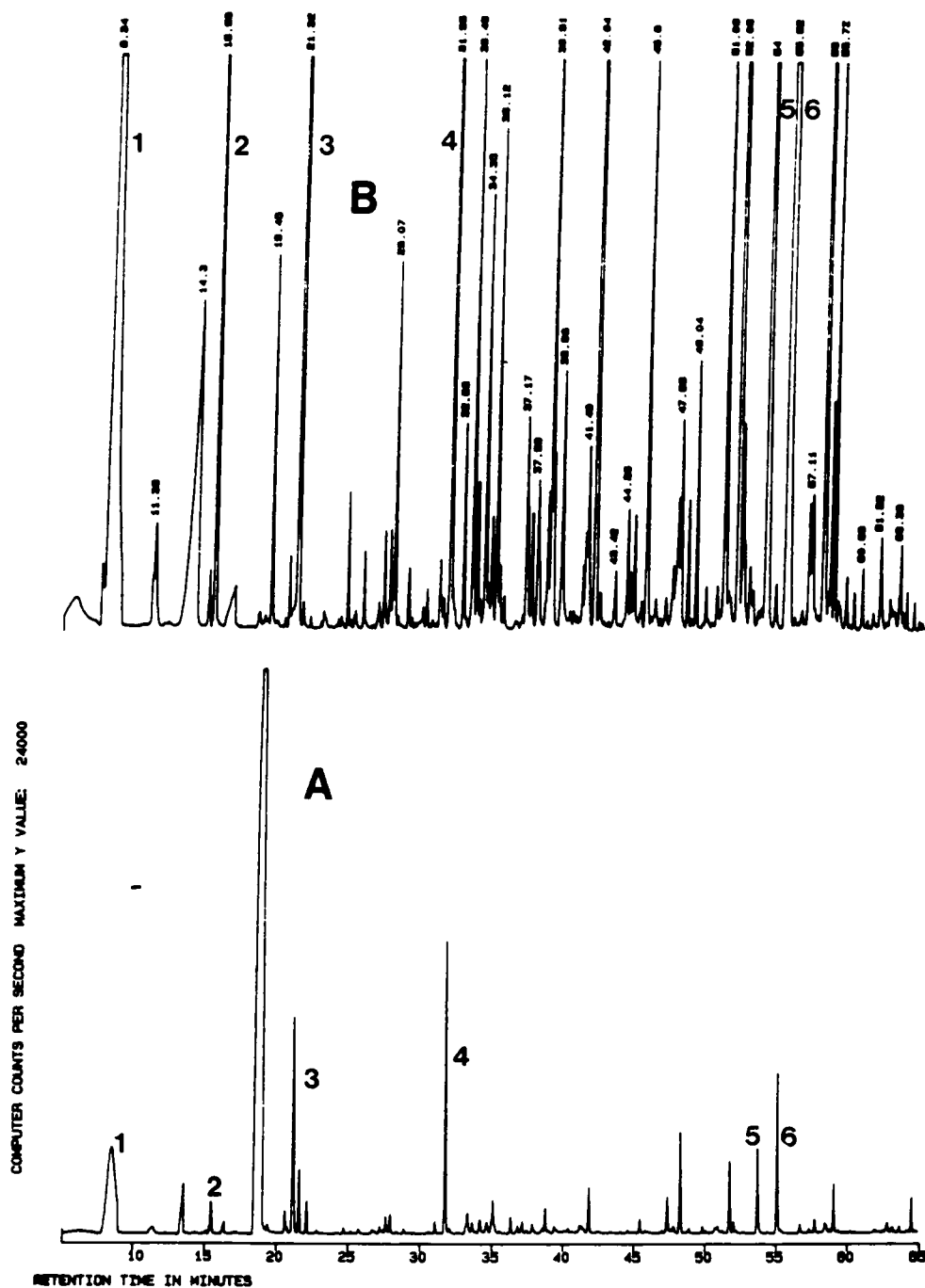


Fig. 3. Effect of static heating time (Days) on Total Volatile Profile in peanut oil. A-unheated; B-heated to 14 days. Volatiles: 1=Pentane, 2=Pentanal, 3=Hexanal, 4=t-2-Heptenal, 5=t,c-2,4-Decadienal and 6=t,t-2,4-Decadienal.

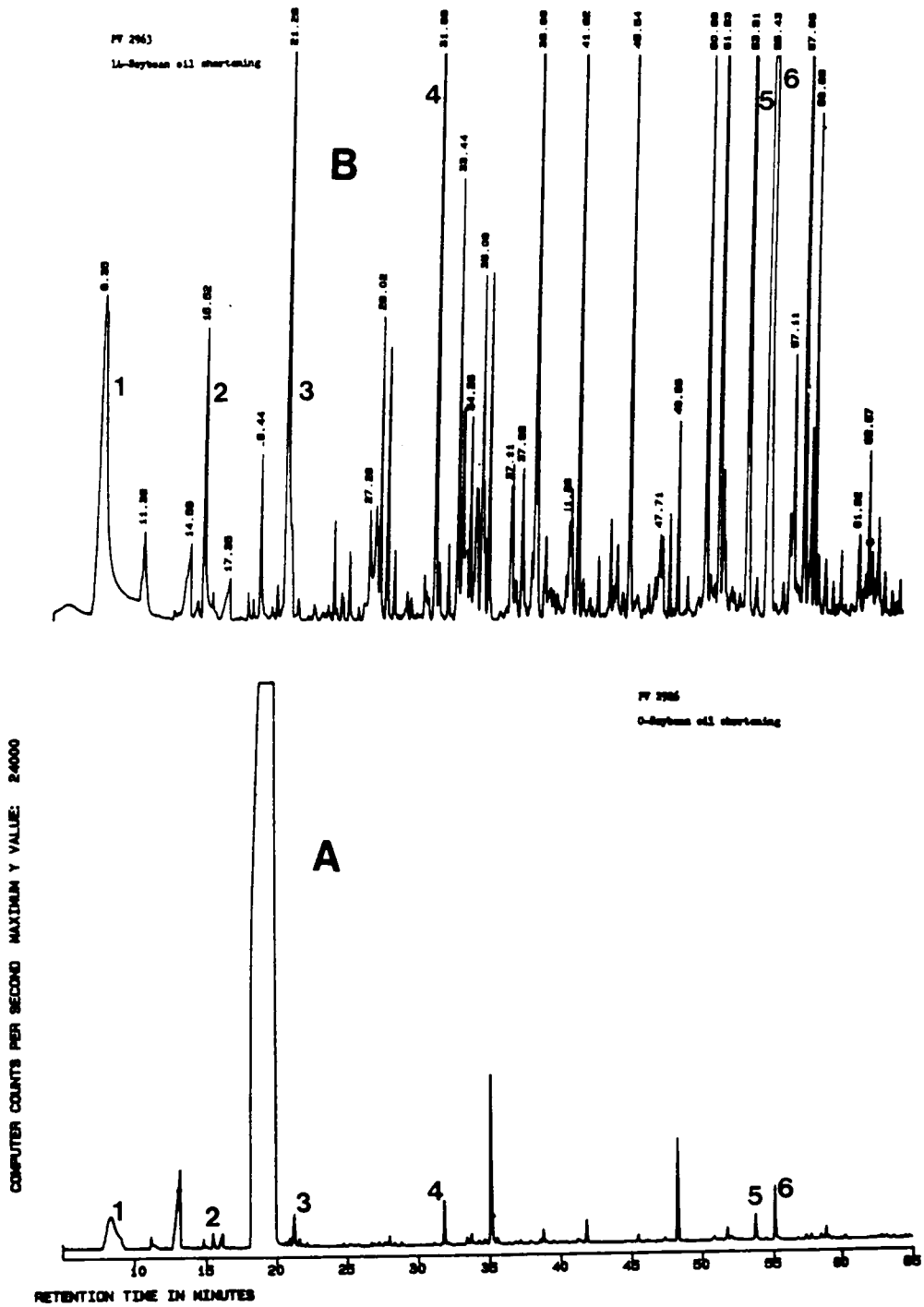


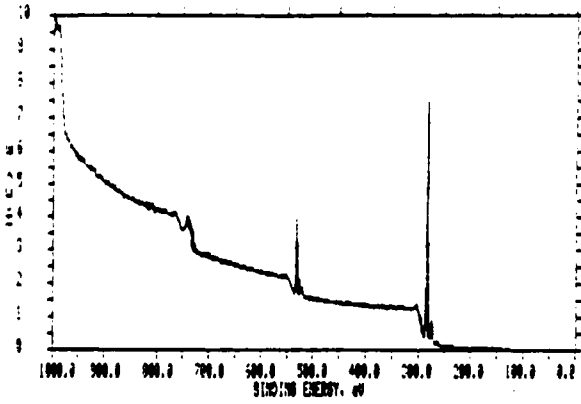
Fig. 4. Effect of static heating time (Days) on Total Volatile Profile in soybean oil liquid shortening. A-unheated; B-heated to 14 days. Volatiles: 1=Pentane, 2=Pentanal, 3=Hexanal, 4=t-2-Heptenal, 5=t,c-2,4-Decadienal and 6=t,t-2,4-Decadienal.

APPENDIX

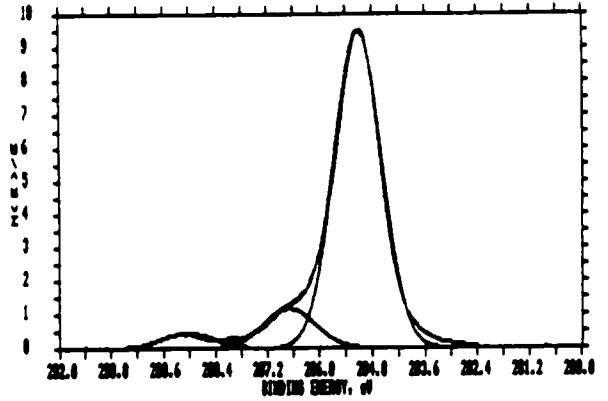
E

Typical ESCA Spectra of FT-120-WW Surface

ESCA SURVEY 4/6/80 ANGLE= 90 deg ACQ TIME=4.50 min  
 FILE: NB9 ORIGINAL  
 SCALE FACTOR, OFFSET=0.316, 0.259 t c/s PASS ENERGY=44.750 eV Mg 250 II



ESCA MULTIPLEX 4/6/80 EL=Cl RES 1 ANGLE= 90 deg ACQ TIME=3.60 min  
 FILE: NB10 ORIGINAL  
 SCALE FACTOR, OFFSET=2.440, 0.000 t c/s PASS ENERGY=17.900 eV Mg 250 II



ESCA MULTIPLEX 4/6/80 EL=Cl RES 1 ANGLE= 90 deg ACQ TIME=3.60 min  
 FILE: NB10 ORIGINAL  
 SCALE FACTOR, OFFSET=2.364, 0.930 t c/s PASS ENERGY=17.900 eV Mg 250 II

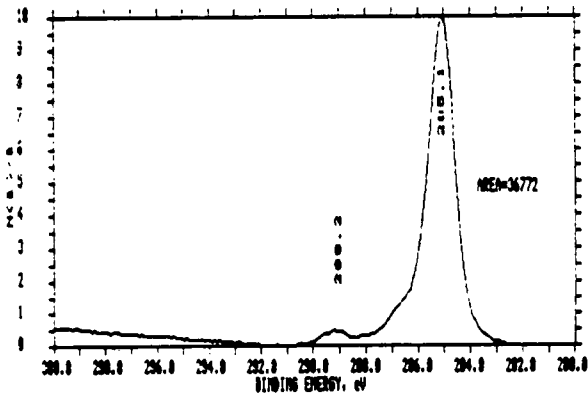
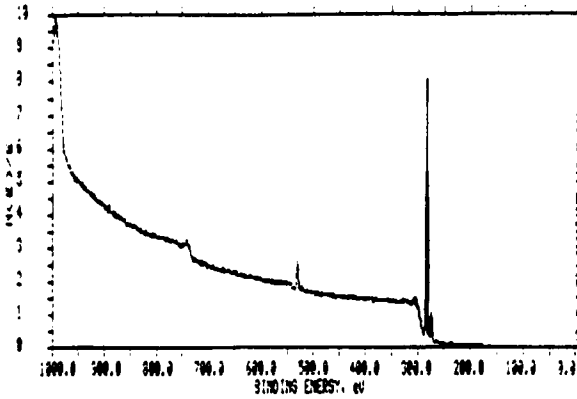


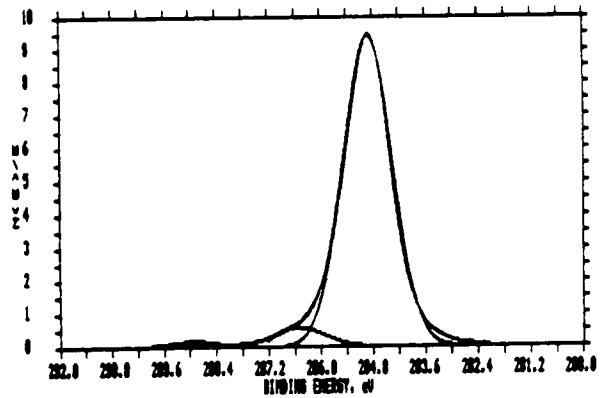
Fig. 5. Typical ESCA spectra obtained from control FT-120-WW specimen.



ESCA SURVEY 4/6/88 ANGLE= 90 deg ACQ TIME=4.17 min  
 FILE: 180 SAMPLE 180M  
 SCALE FACTOR, OFFSET=7.348, 0.228 I/c/s PASS ENERGY=44.750 eV No 250 II



ESCA MULTIPLEX 4/6/88 EL=C1 RES 1 ANGLE= 90 deg ACQ TIME=3.68 min  
 FILE: 184 SAMPLE 184M  
 SCALE FACTOR, OFFSET=2.562, 0.000 I/c/s PASS ENERGY=17.000 eV No 250 II



ESCA MULTIPLEX 4/6/88 EL=C1 RES 1 ANGLE= 90 deg ACQ TIME=3.68 min  
 FILE: 184 SAMPLE 184M  
 SCALE FACTOR, OFFSET=2.454, 0.766 I/c/s PASS ENERGY=17.000 eV No 250 II

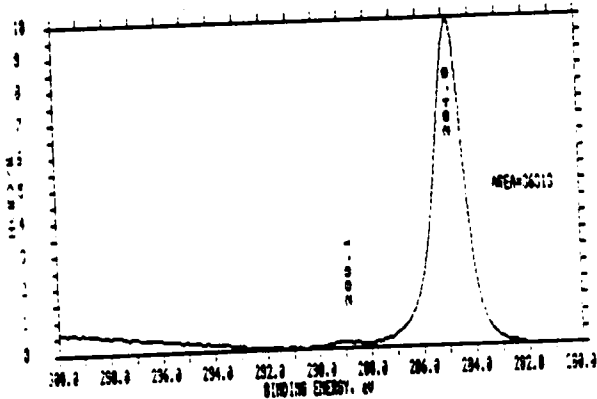
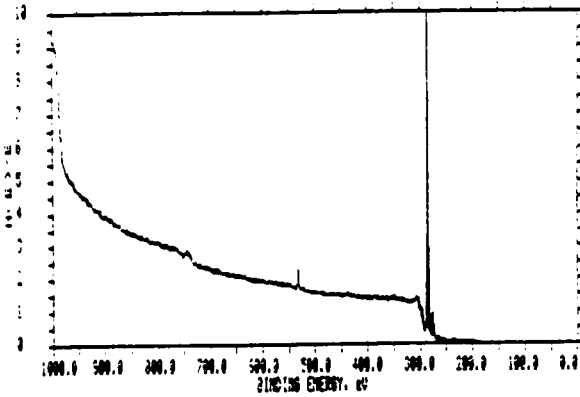
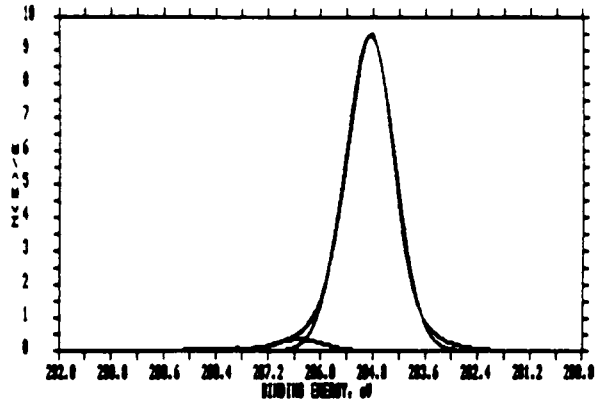


Fig. 6. Typical ESCA spectra obtained from FT-120-WW specimen after being washed with methanol.

ESCA SURVEY 4/6/88 ANGLE= 90 deg ACO TIME=4.50 min  
 FILE: HB2 SAMPLE 1  
 SCALE FACTOR, OFFSET=0.000, 0.155 k c/s PASS ENERGY=44.750 eV Ng 250 u



MULTIPLY 4/6/88 EL=C1 RES 1 ANGLE= 90 deg ACO TIME=3.60 min  
 FILE: HB2 SAMPLE 1  
 SCALE FACTOR, OFFSET=2.763, 0.000 k c/s PASS ENERGY=17.500 eV Ng 250 u



ESCA MULTIPLY 4/6/88 EL=C1 RES 1 ANGLE= 90 deg ACO TIME=3.60 min  
 FILE: HB2 SAMPLE 1  
 SCALE FACTOR, OFFSET=2.553, 0.700 k c/s PASS ENERGY=17.500 eV Ng 250 u

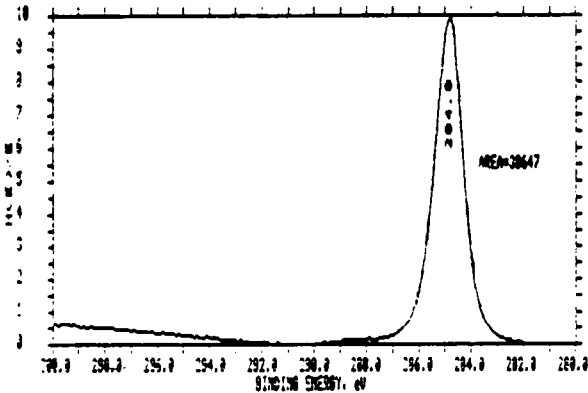
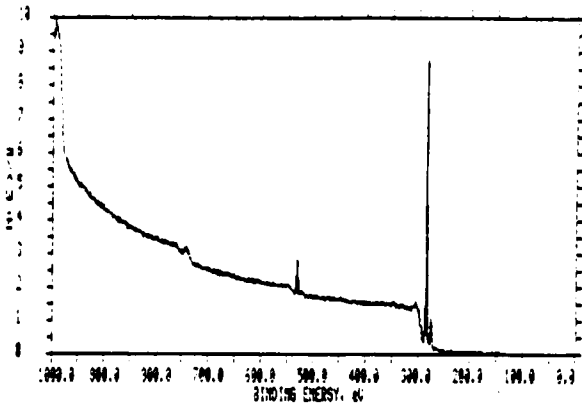
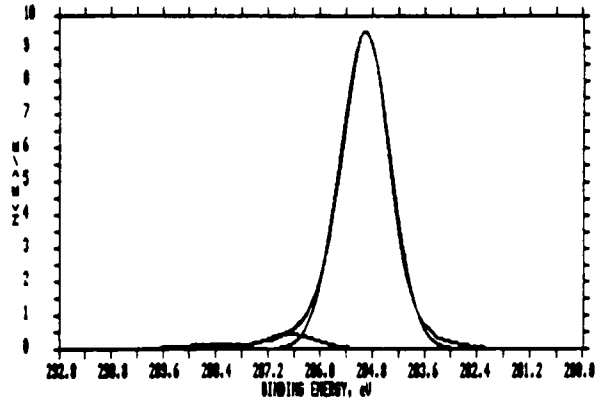


Fig. 7. Typical ESCA spectra obtained from FT-120-WW specimen directly after annealing.

ESCA SURVEY 4/6/88 ANGLE= 30 deg ACO TIME=4.59 min  
 FILE: 186 SAMPLE 5  
 SCALE FACTOR, OFFSET=7.393, 0.234 I c/s PASS ENERGY=44.750 eV Mg 250 U



ESCA MULTIPLEX 4/6/88 EL=C1 RES 1 ANGLE= 30 deg ACO TIME=3.68 min  
 FILE: 186 SAMPLE 5  
 SCALE FACTOR, OFFSET=2.577, 0.000 I c/s PASS ENERGY=17.900 eV Mg 250 U



ESCA MULTIPLEX 4/6/88 EL=C1 RES 1 ANGLE= 30 deg ACO TIME=3.68 min  
 FILE: 186 SAMPLE 5  
 SCALE FACTOR, OFFSET=2.469, 0.795 I c/s PASS ENERGY=17.900 eV Mg 250 U

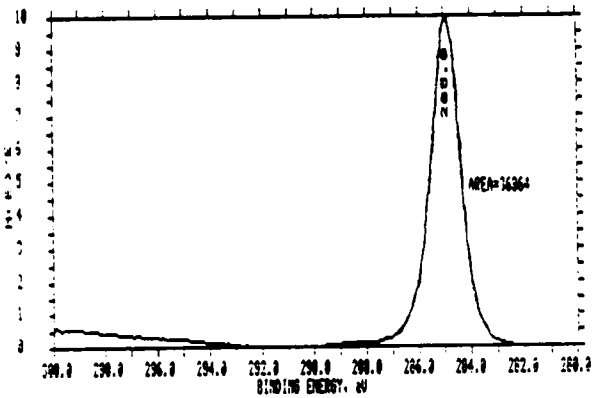
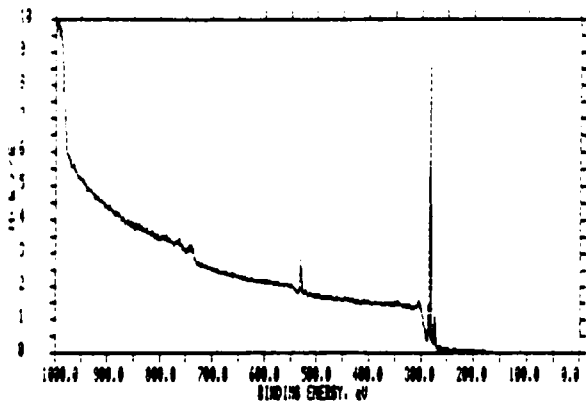
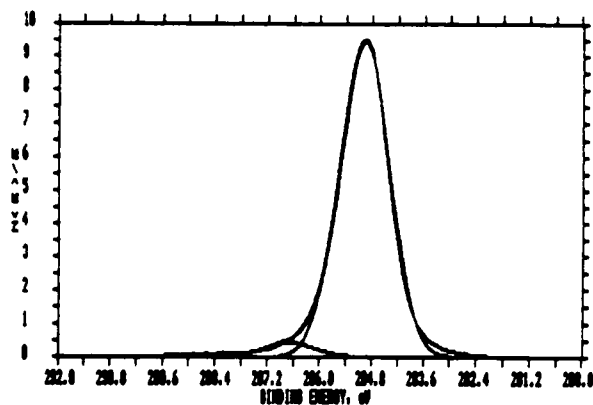


Fig. 8. Typical ESCA spectra obtained from FT-120-WW specimen stored up to 4 days after annealing.

ESCA SURVEY 4/6/88 ANGLE= 90 deg ACQ TIME=4.50 min  
 FILE: 187 SAMPLE 7  
 SCALE FACTOR, OFFSET=7.927, 0.171 k c/s PASS ENERGY=44.750 eV Mg 250 U



ESCA MULTIPLEX 4/6/88 EL=Cl RES 1 ANGLE= 90 deg ACQ TIME=3.60 min  
 FILE: 188 SAMPLE 7  
 SCALE FACTOR, OFFSET=2.307, 0.000 k c/s PASS ENERGY=17.000 eV Mg 250 U



ESCA MULTIPLEX 4/6/88 EL=Cl RES 1 ANGLE= 90 deg ACQ TIME=3.60 min  
 FILE: 188 SAMPLE 7  
 SCALE FACTOR, OFFSET=2.477, 0.747 k c/s PASS ENERGY=17.000 eV Mg 250 U

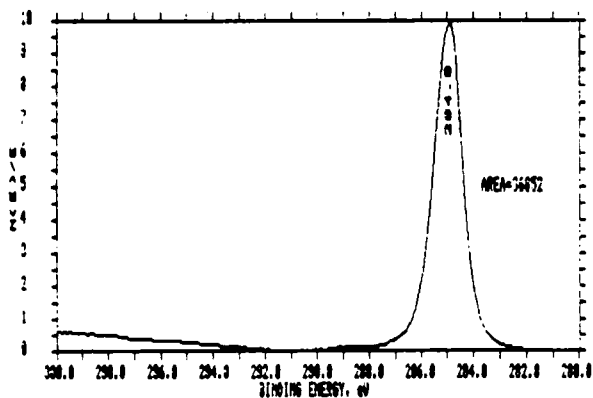


Fig. 9. Typical ESCA spectra obtained from FT-120-WW specimen stored up to 6 days after annealing.

APPENDIX

F

Typical Chromatograms of Frying Oil Triglycerides  
as Influenced by the Static Heating  
Conditions

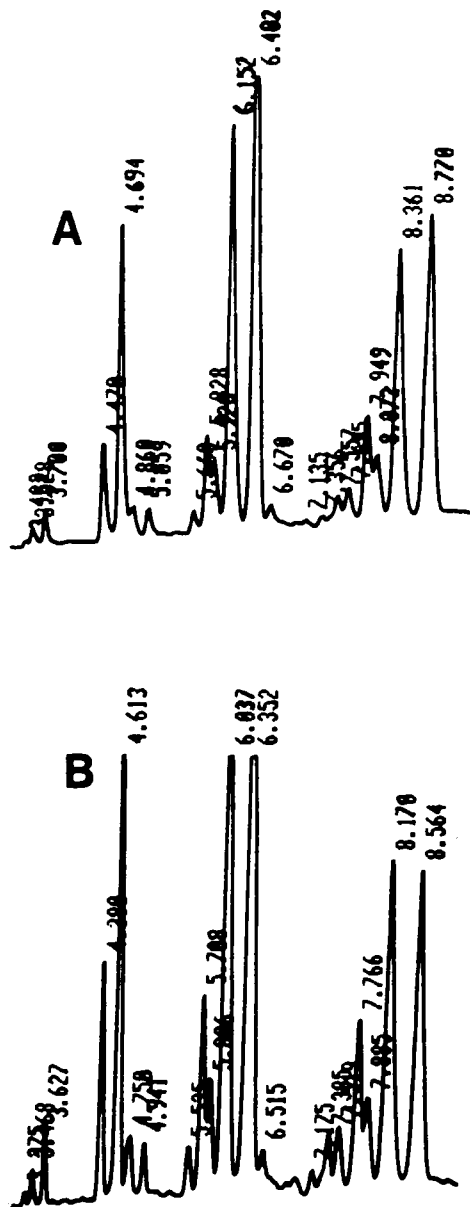


Fig. 10. Effect of static heating time (Days) on triglyceride profile of cottonseed oil. A-unheated; B-heated to 14 days.

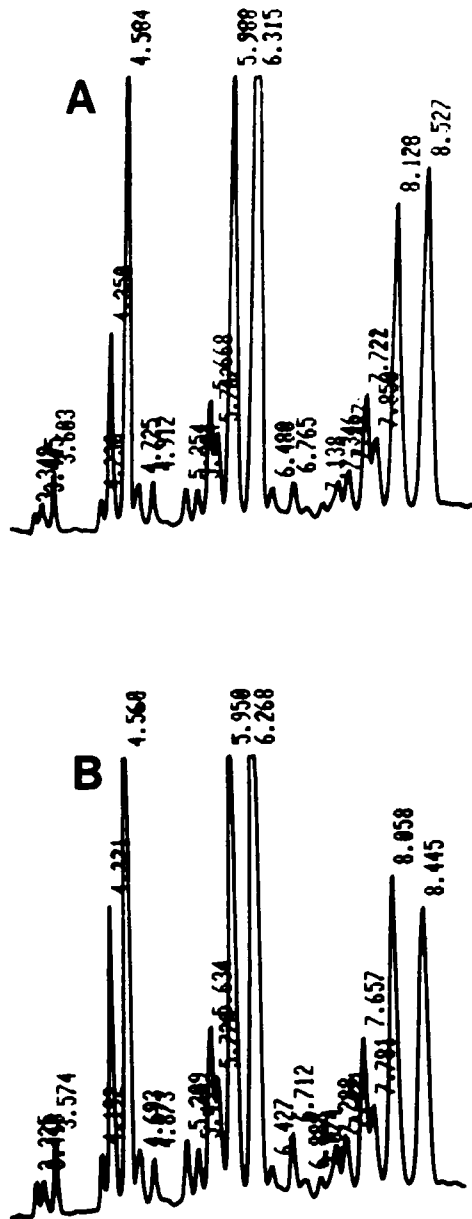


Fig. 11. Effect of static heating time (Days) on triglyceride profile of cottonseed oil liquid shortening. A-unheated; B-heated to 14 days.

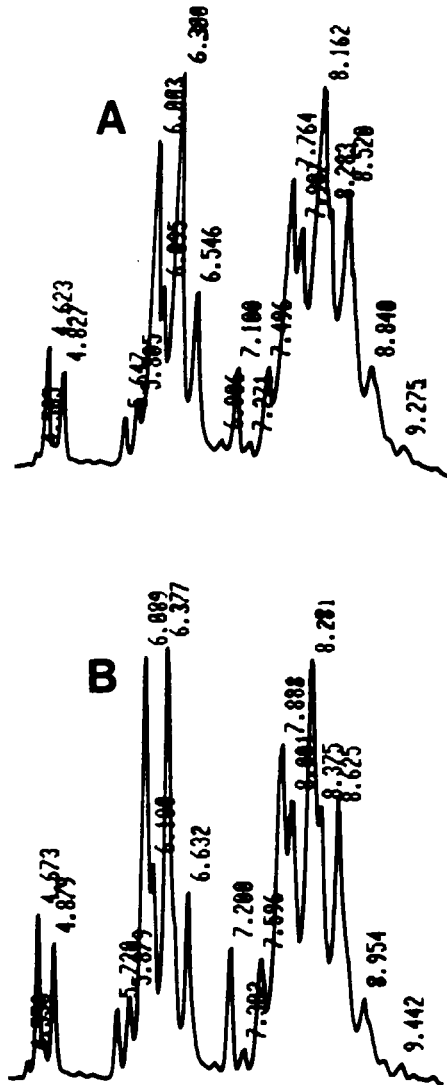


Fig. 12. Effect of static heating time (Days) on triglyceride profile of soybean oil liquid shortening. A-unheated; B-heated to 14 days.



APPENDIX

G

Plots of Linear Regressions

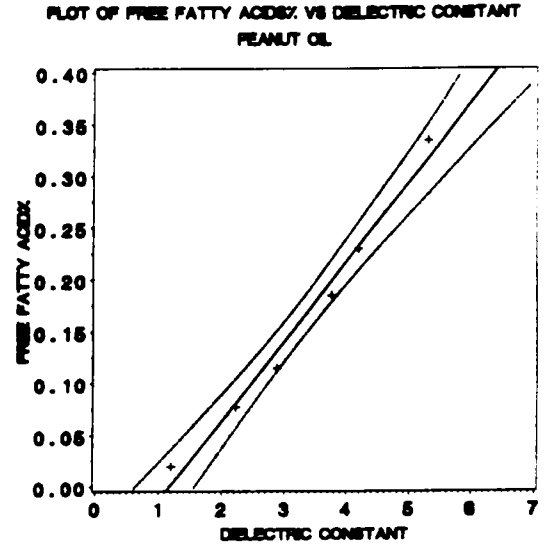
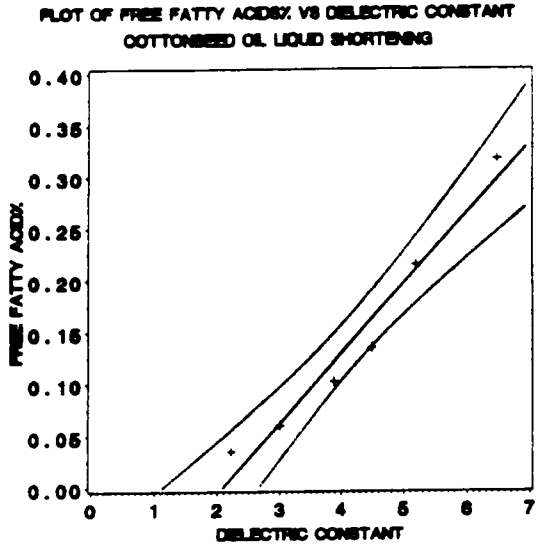
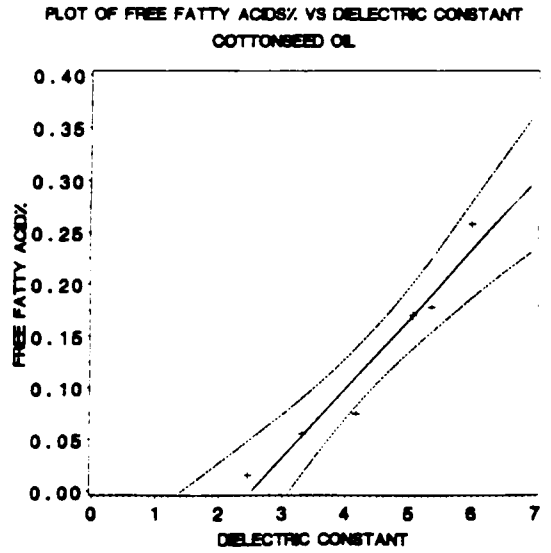
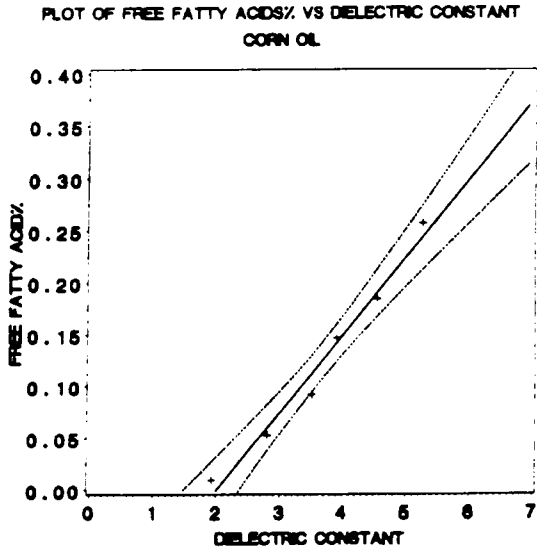


Fig. 13. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

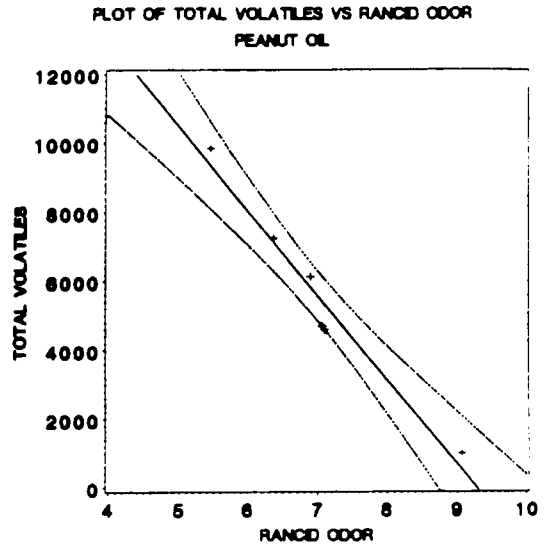
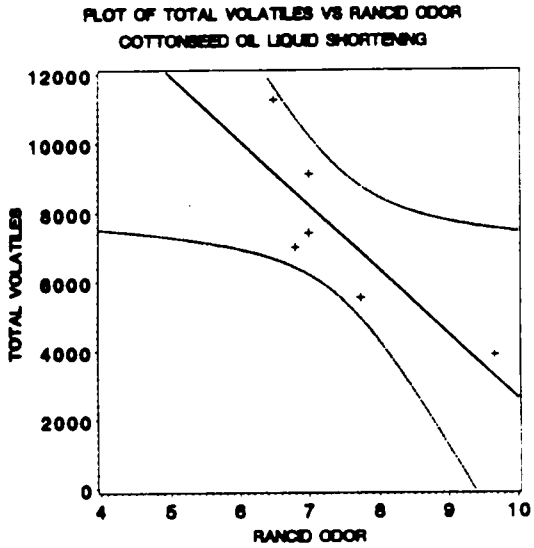
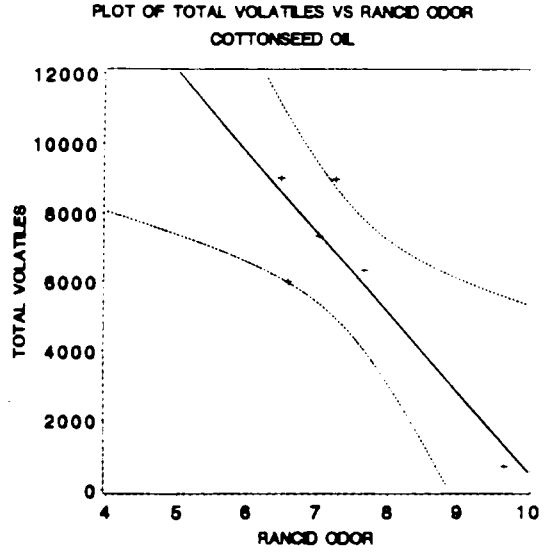
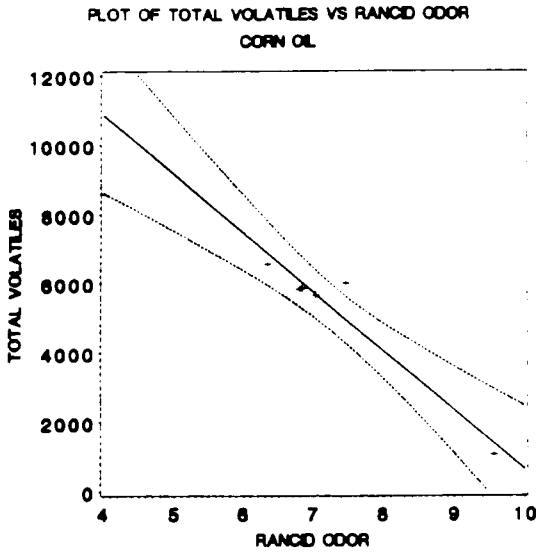


Fig. 14. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

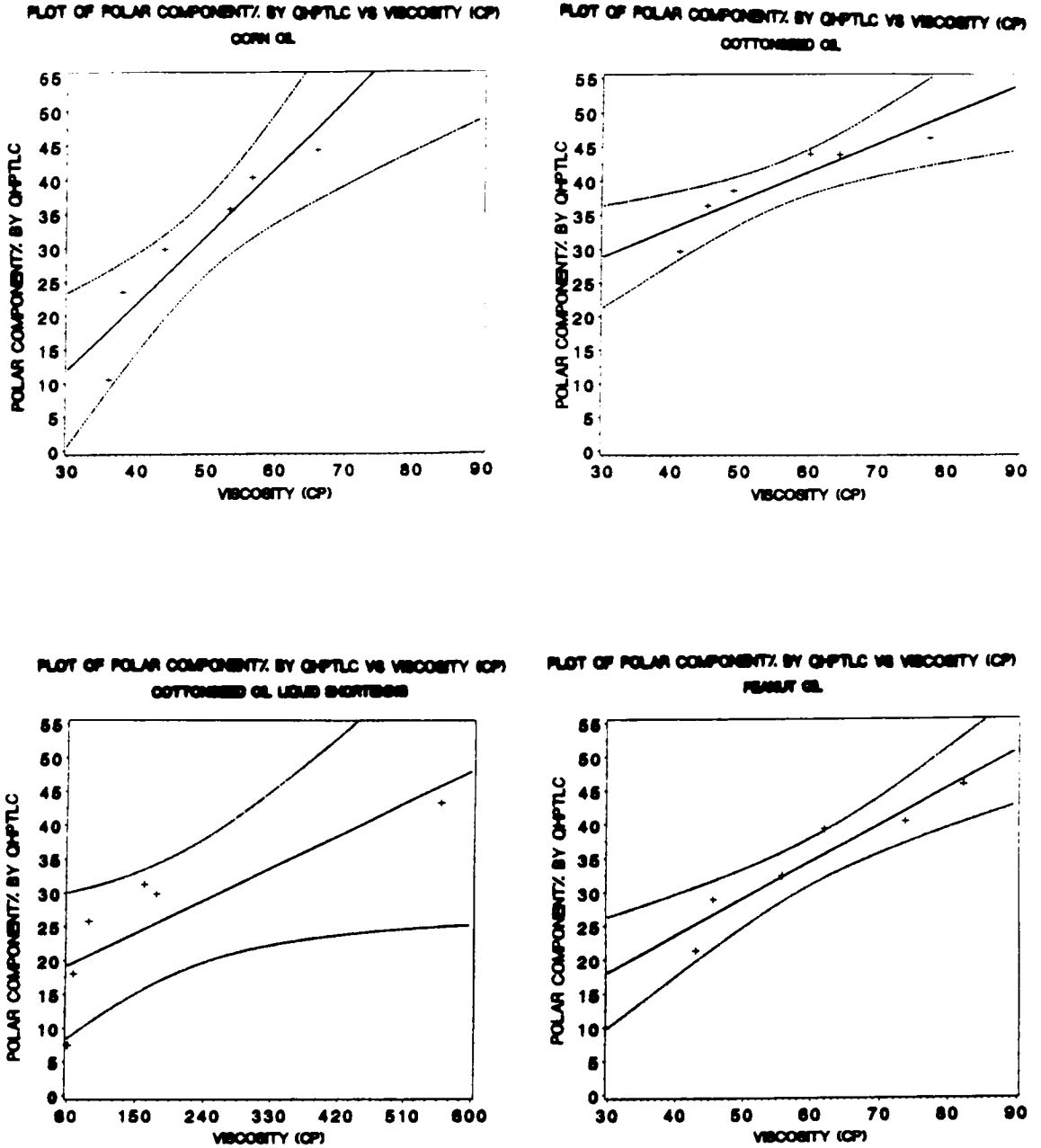


Fig. 15. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

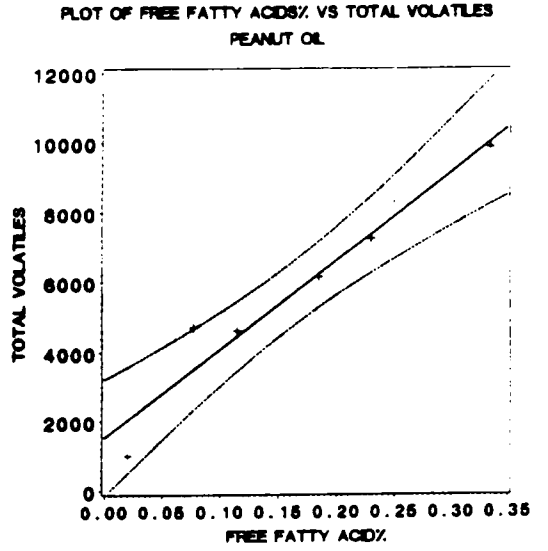
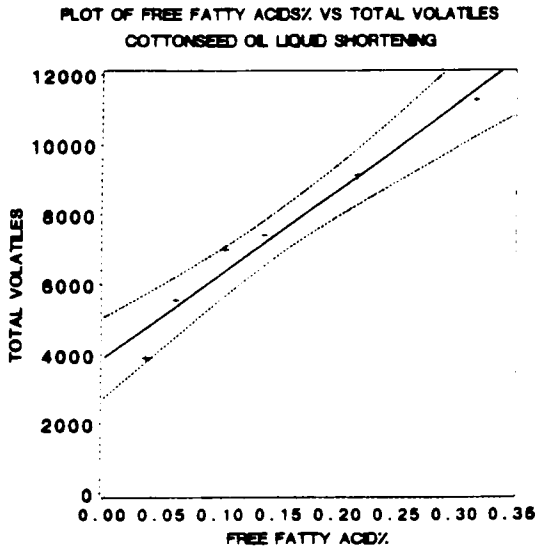
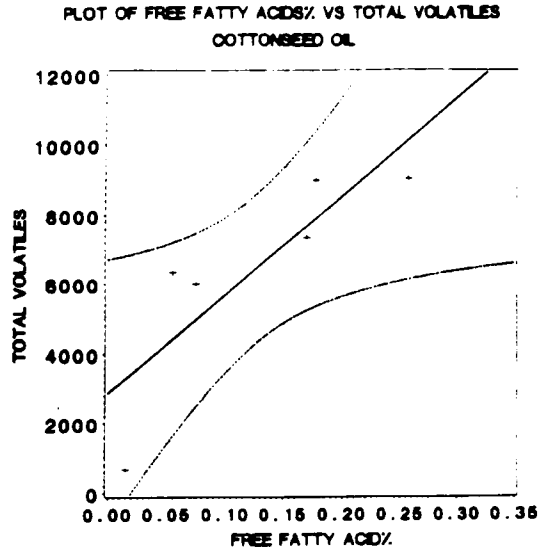
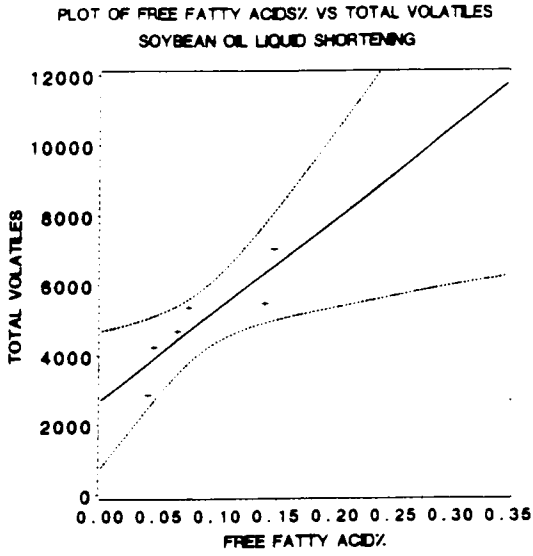


Fig. 16. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

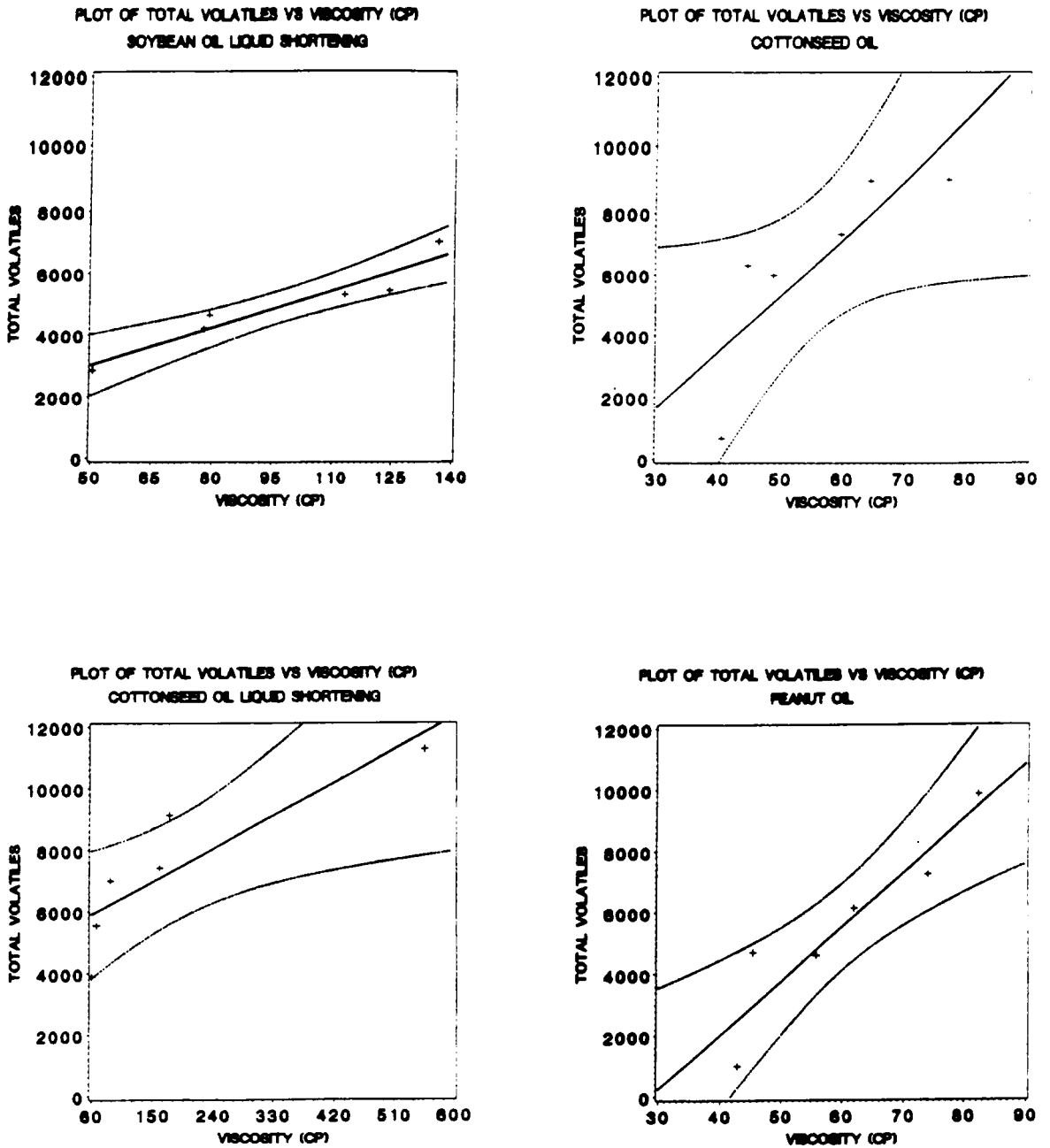


Fig. 17. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

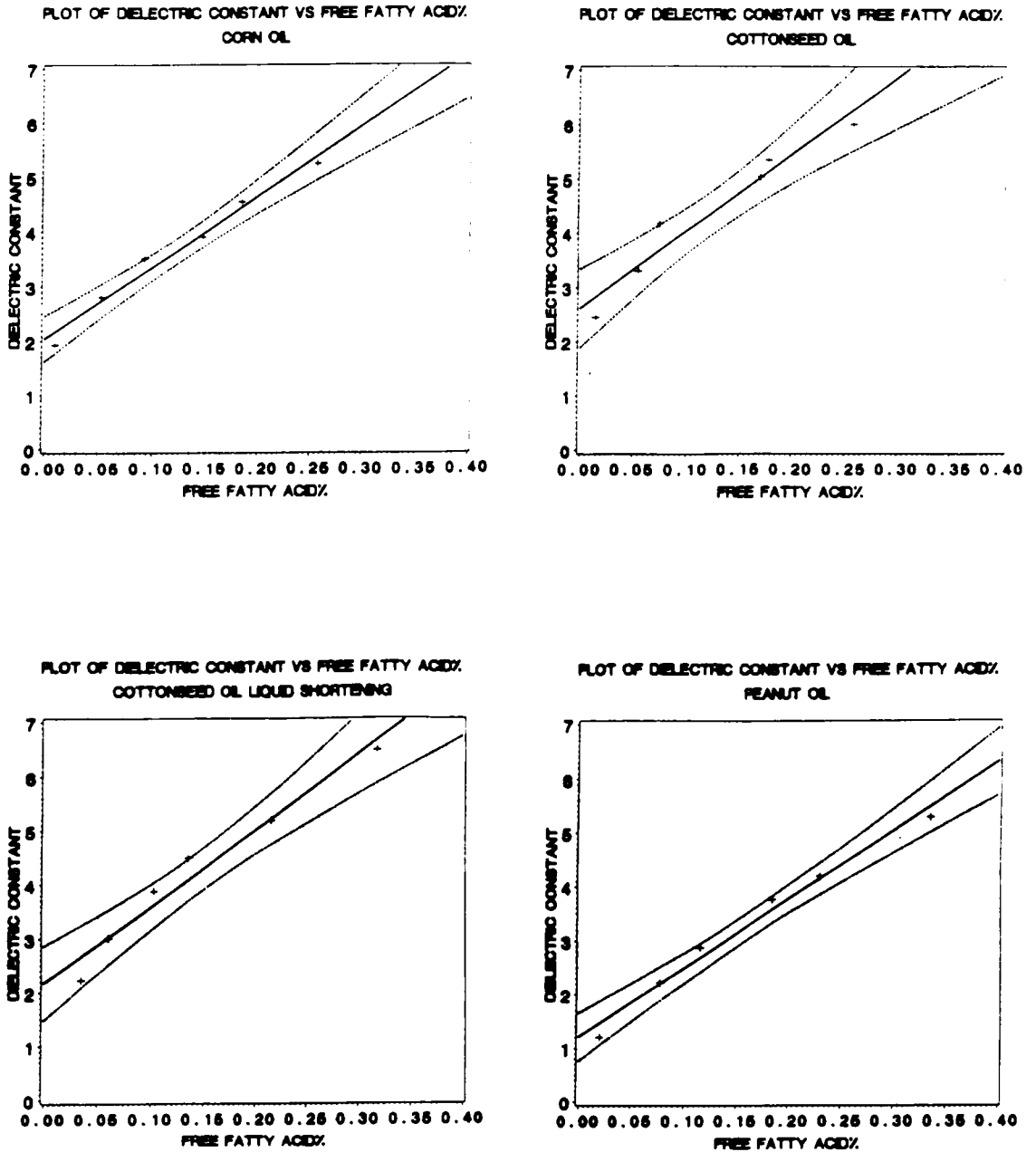


Fig. 18. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

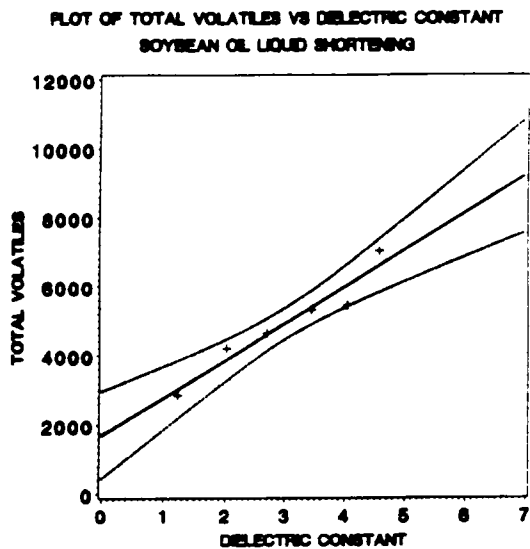
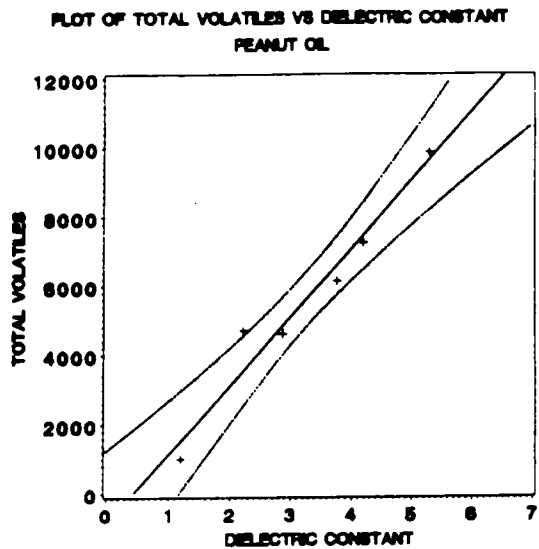
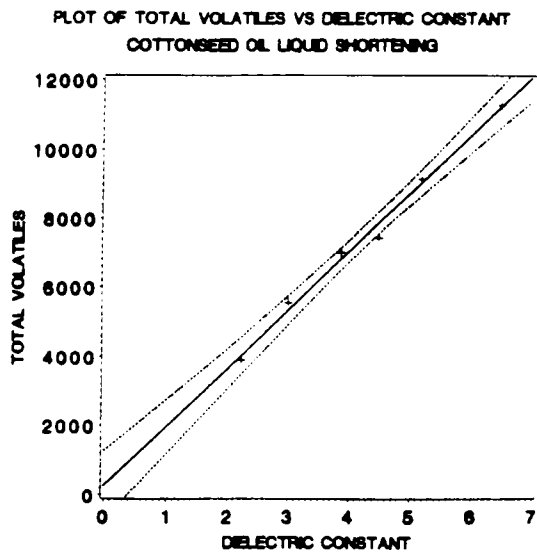
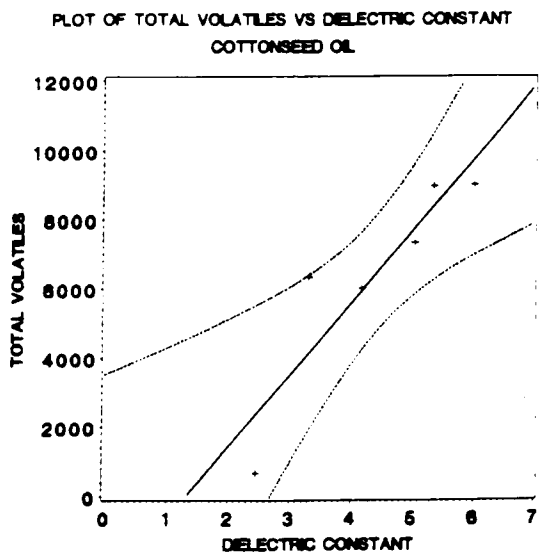


Fig. 19. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.



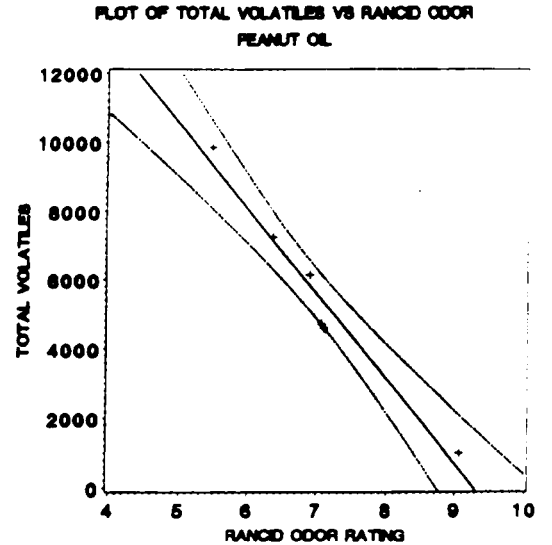
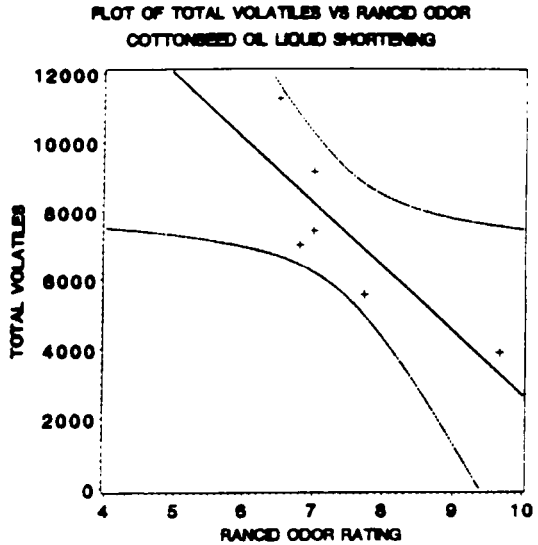
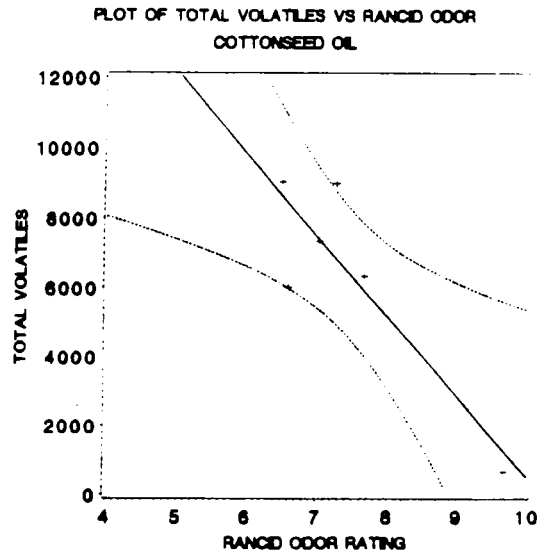
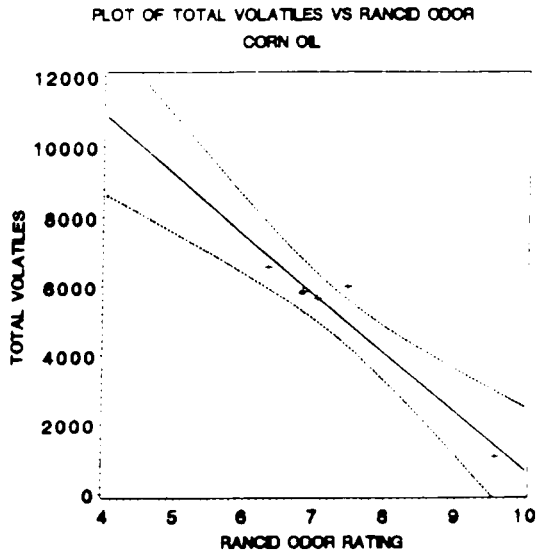
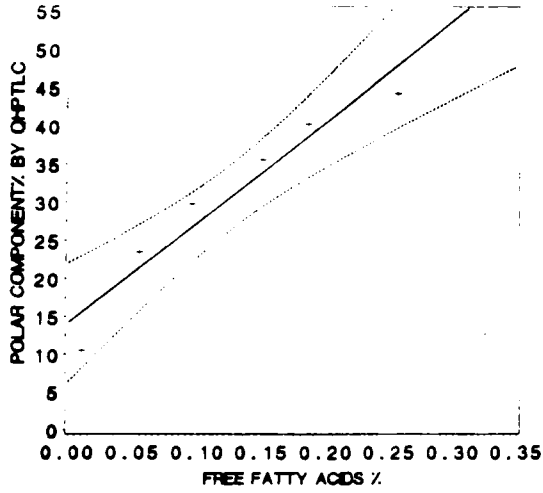
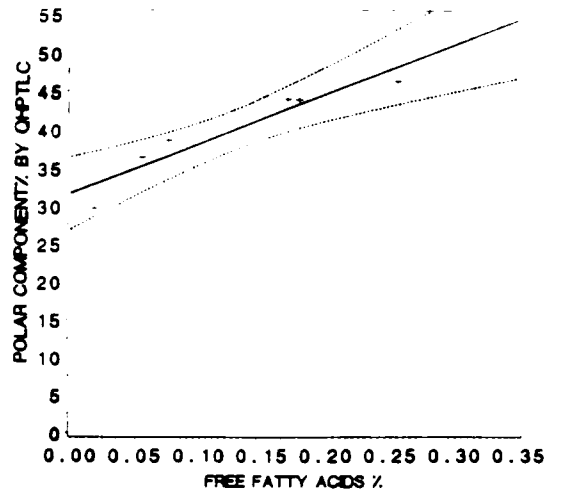


Fig. 20. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

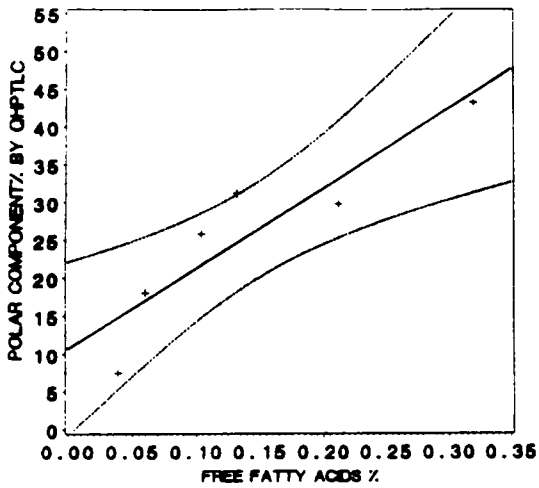
PLOT OF POLAR COMPONENT, BY QHPTLC VS FREE FATTY ACIDS%,  
CORN OIL



PLOT OF POLAR COMPONENT, BY QHPTLC VS FREE FATTY ACIDS%,  
COTTONSEED OIL



PLOT OF POLAR COMPONENT, BY QHPTLC VS FREE FATTY ACIDS%,  
COTTONSEED OIL LIQUID SHORTENING



PLOT OF POLAR COMPONENT, BY QHPTLC VS FREE FATTY ACIDS%,  
PEANUT OIL

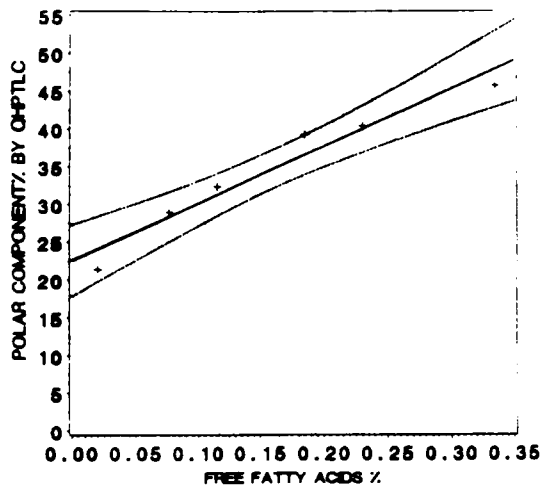


Fig. 21. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

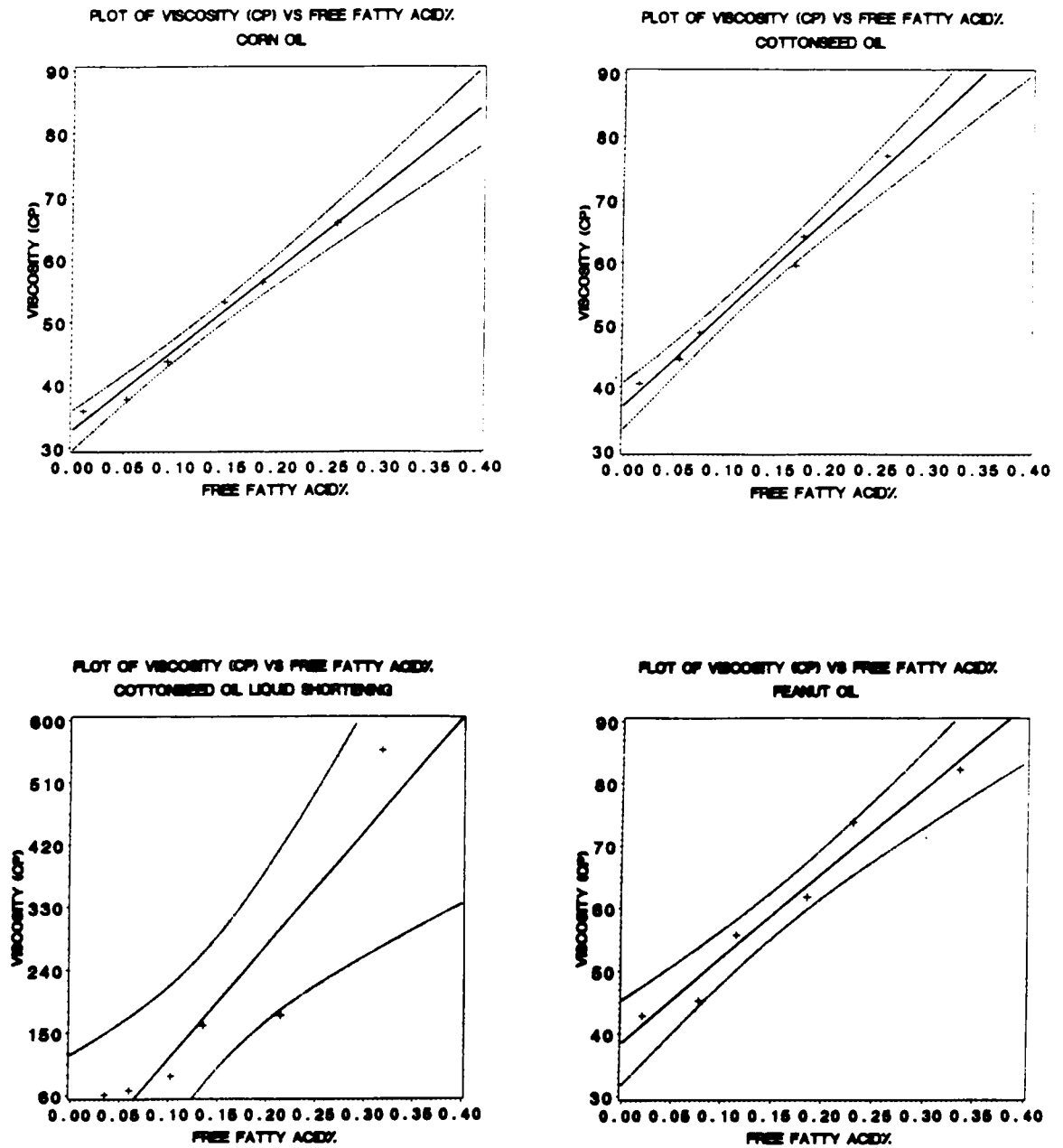
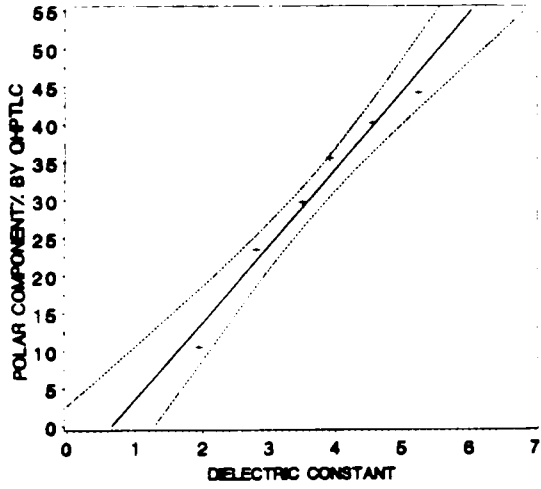
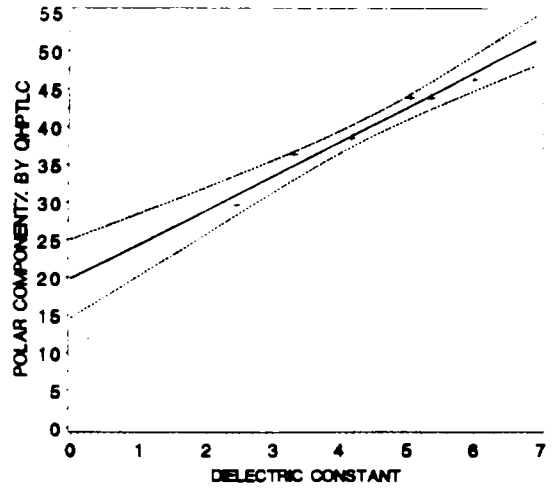


Fig. 22. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

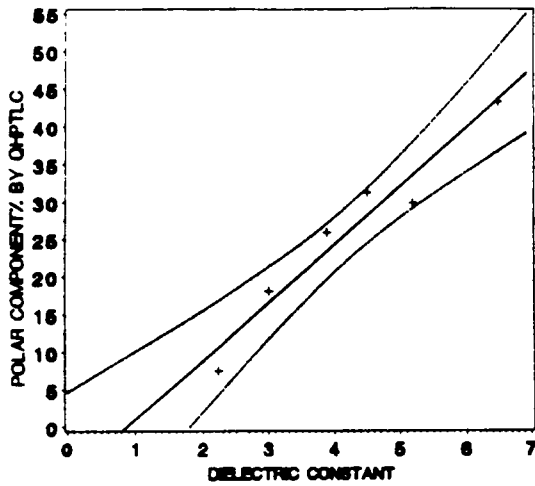
PLOT OF POLAR COMPONENT, BY OHPTLC VS DIELECTRIC CONSTANT  
CORN OIL



PLOT OF POLAR COMPONENT, BY OHPTLC VS DIELECTRIC CONSTANT  
COTTONSEED OIL



PLOT OF POLAR COMPONENT, BY OHPTLC VS DIELECTRIC CONSTANT  
COTTONSEED OIL LIQUID SHORTENING



PLOT OF POLAR COMPONENT, BY OHPTLC VS DIELECTRIC CONSTANT  
PEANUT OIL

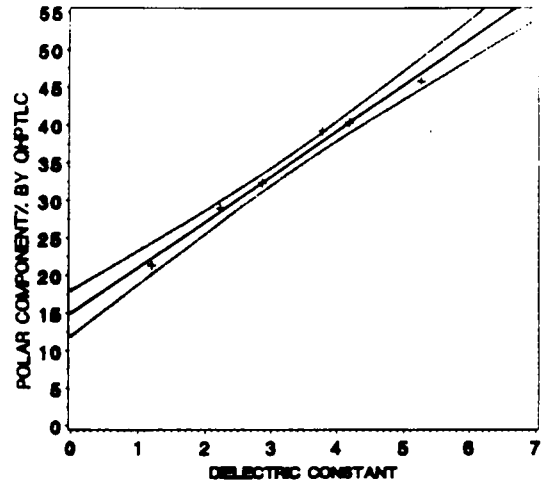


Fig. 23. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

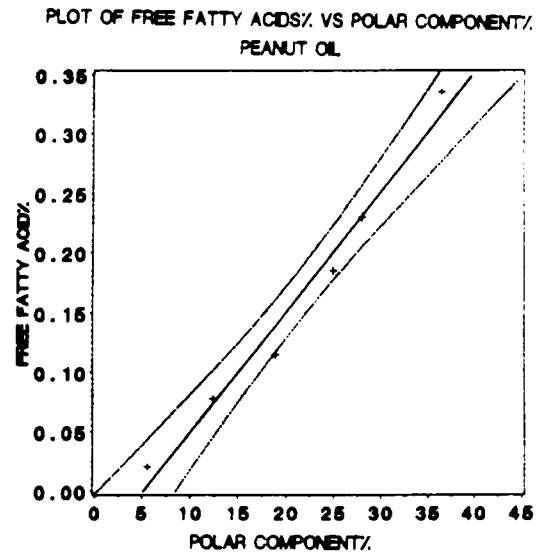
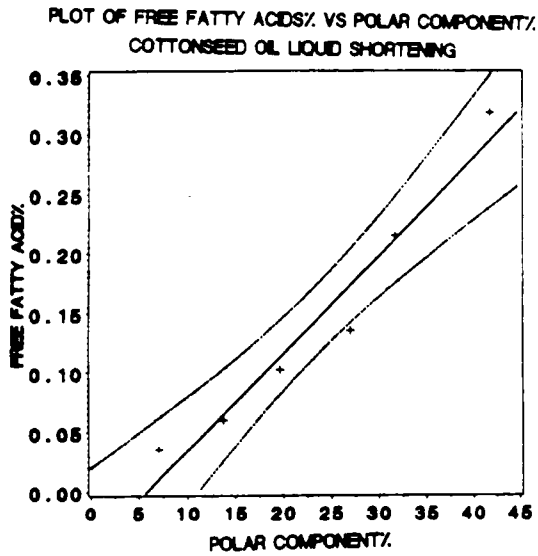
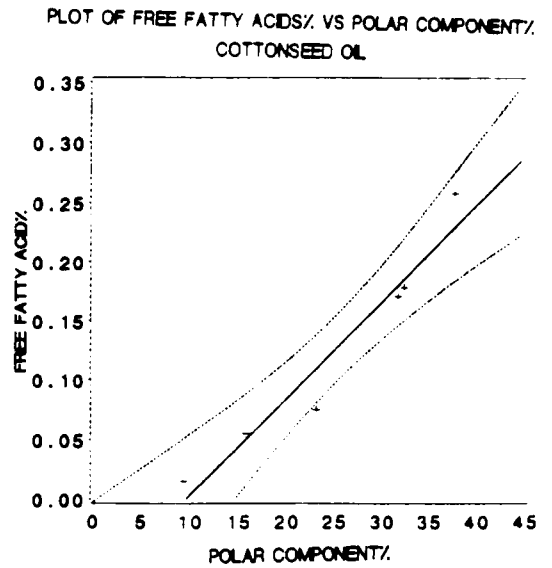
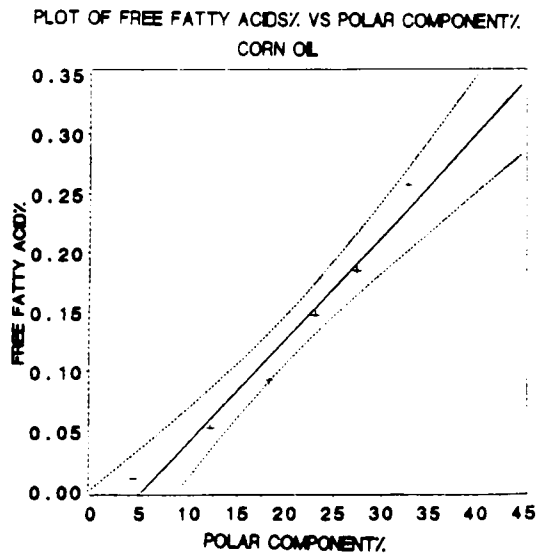


Fig. 24. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

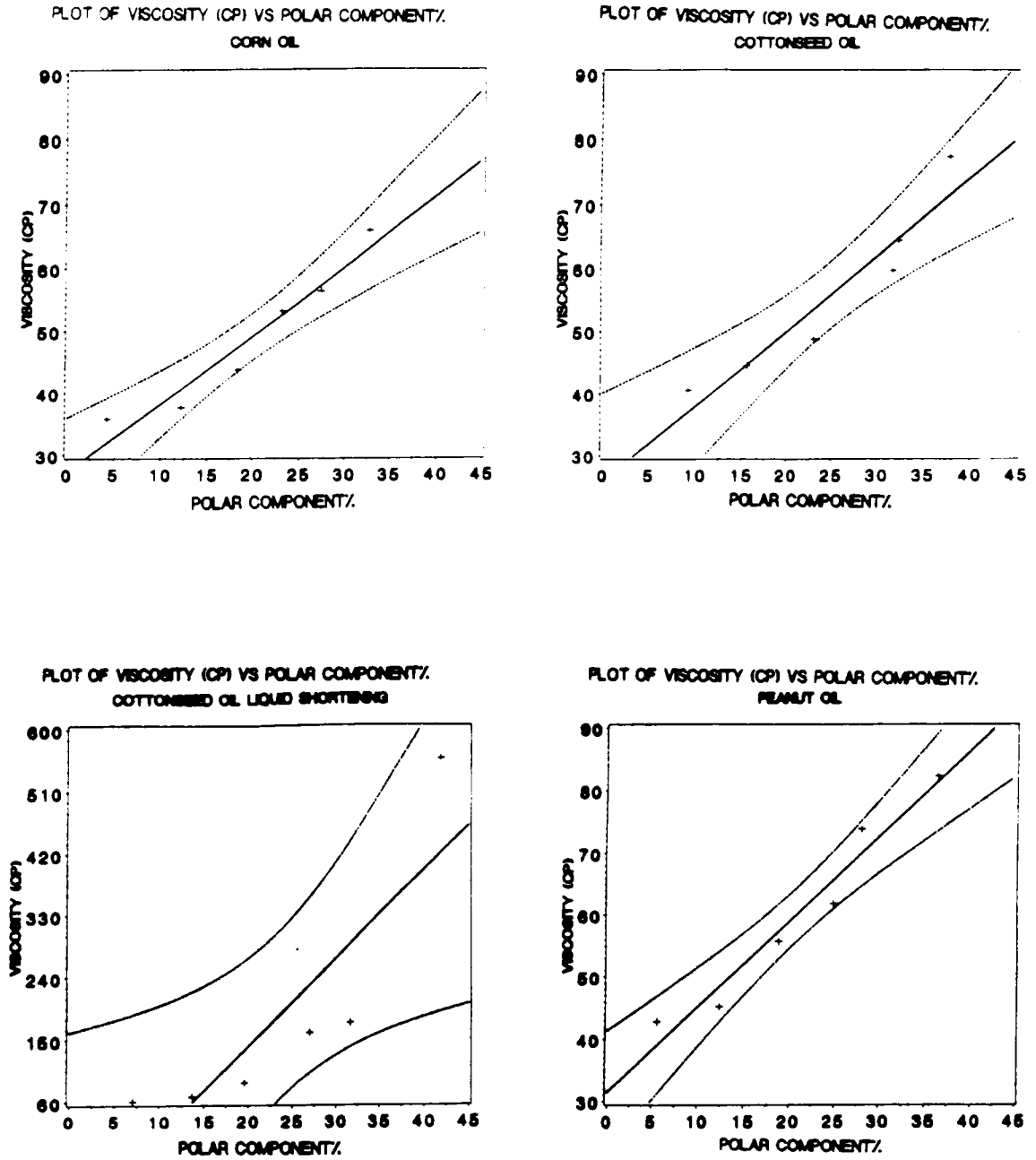


Fig. 25. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

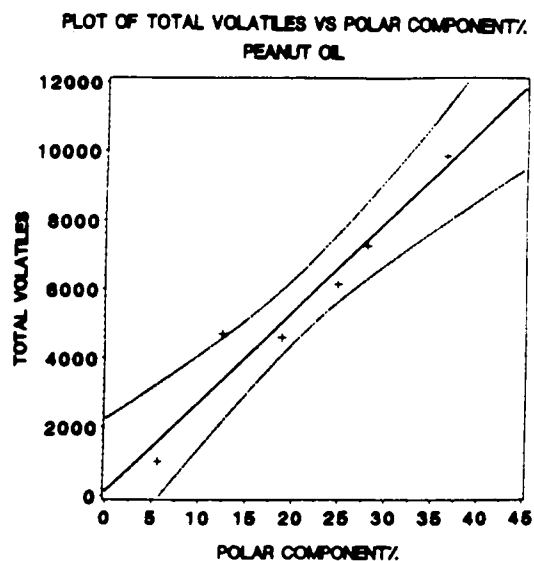
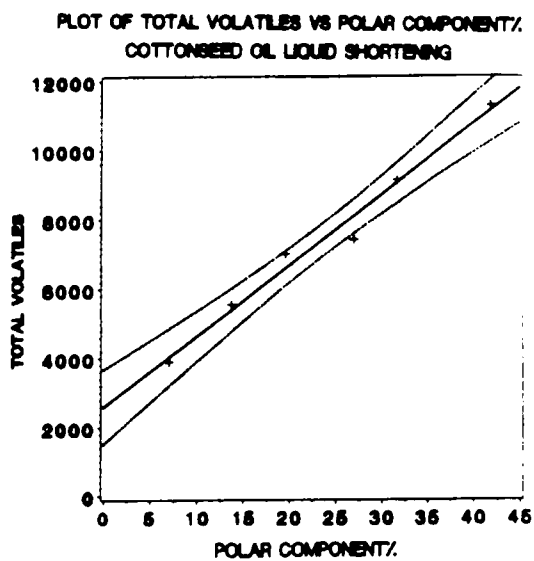
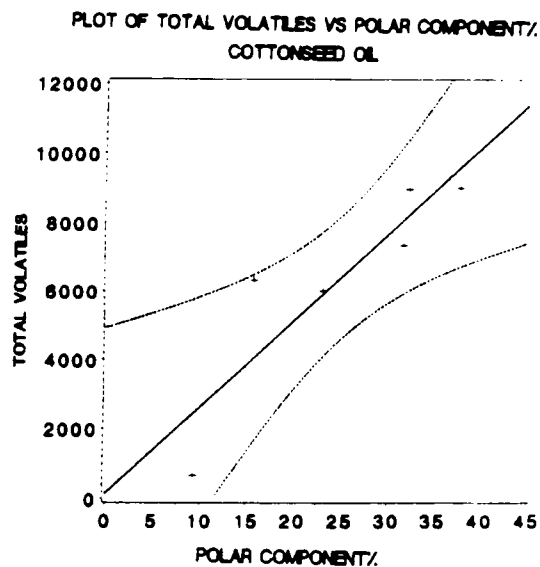
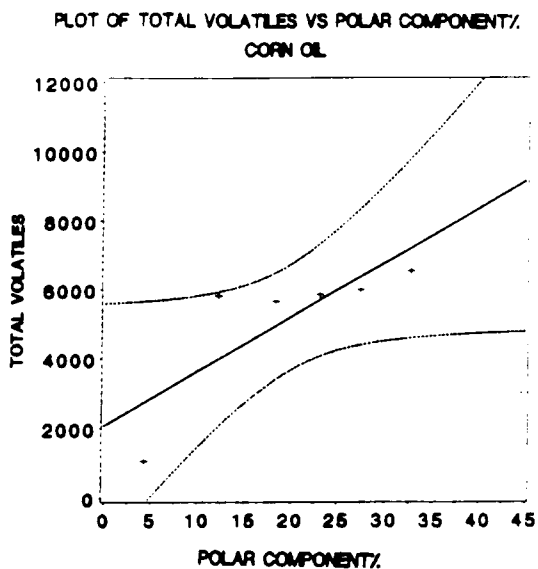
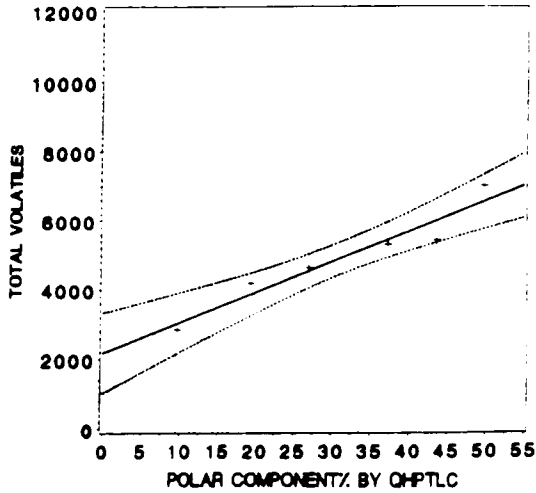
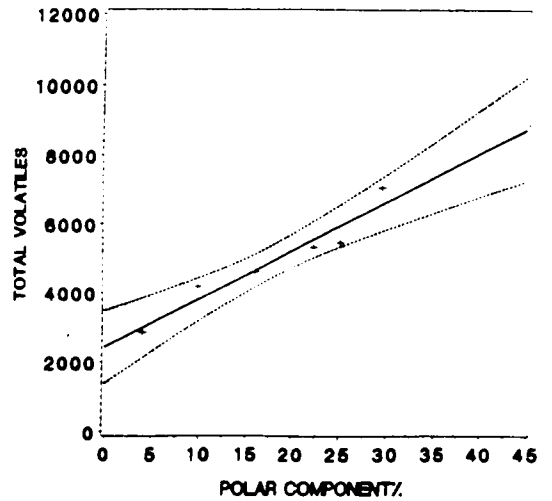


Fig. 26. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

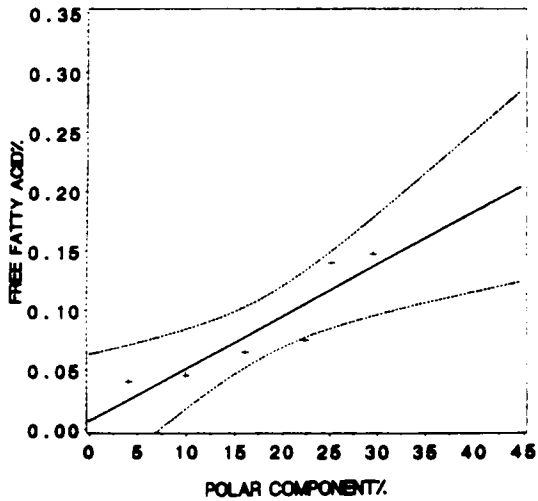
PLOT OF TOTAL VOLATILES VS POLAR COMPONENT% BY QHPTLC  
SOYBEAN OIL LIQUID SHORTENING



PLOT OF TOTAL VOLATILES VS POLAR COMPONENT%  
SOYBEAN OIL LIQUID SHORTENING



PLOT OF FREE FATTY ACIDS% VS POLAR COMPONENT%  
SOYBEAN OIL LIQUID SHORTENING



PLOT OF FREE FATTY ACIDS% VS DIELECTRIC CONSTANT  
SOYBEAN OIL LIQUID SHORTENING

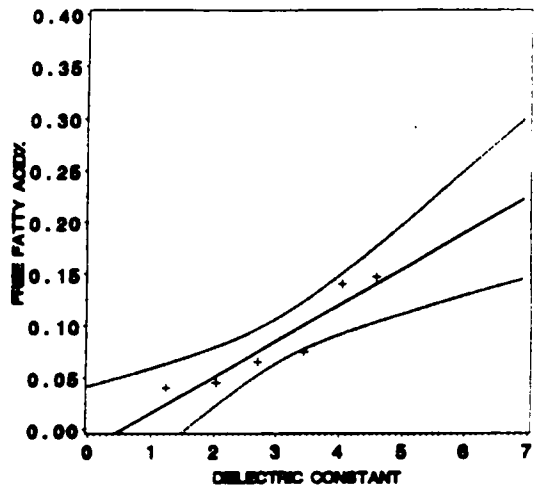


Fig. 27. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.



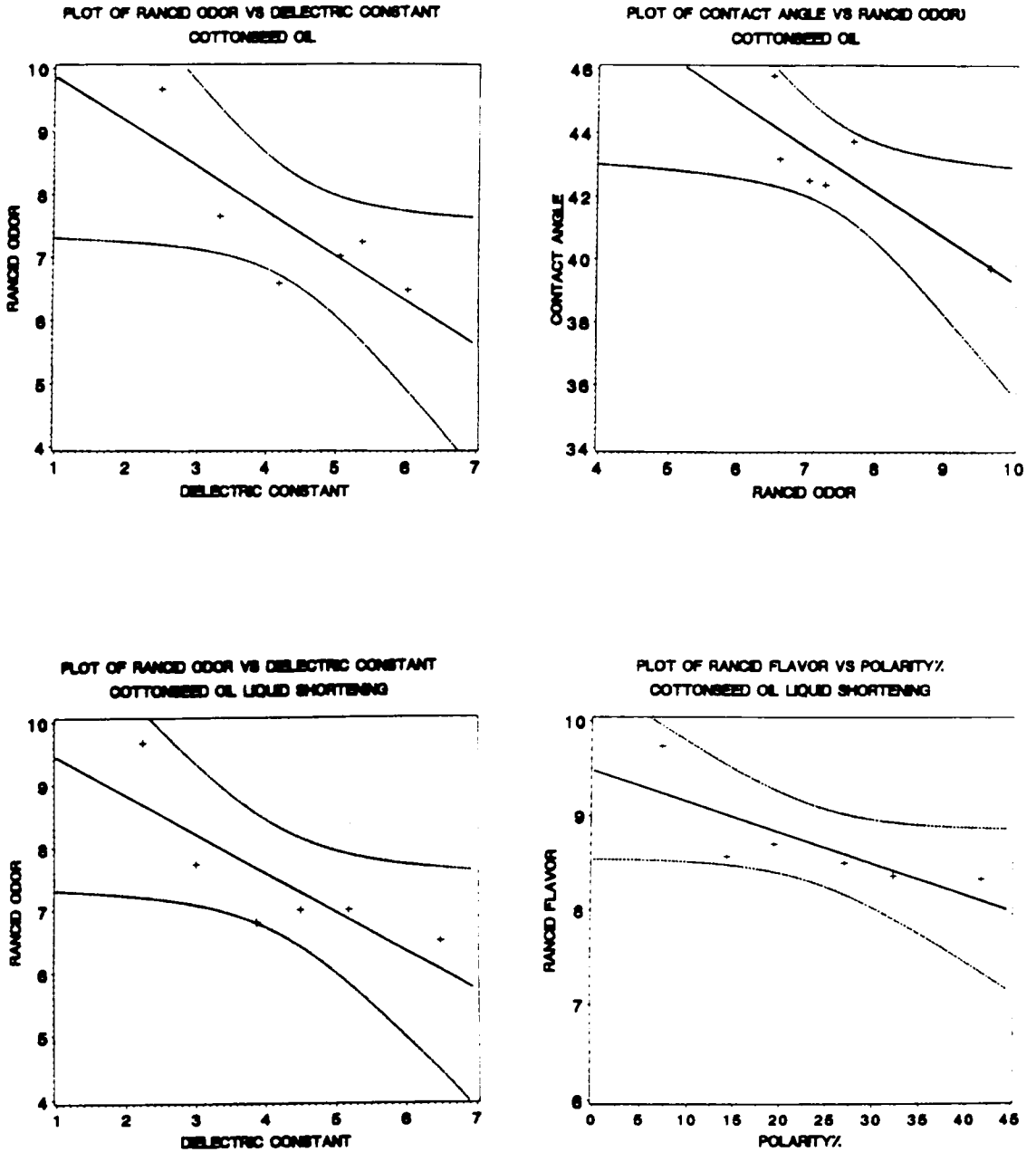


Fig. 28. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

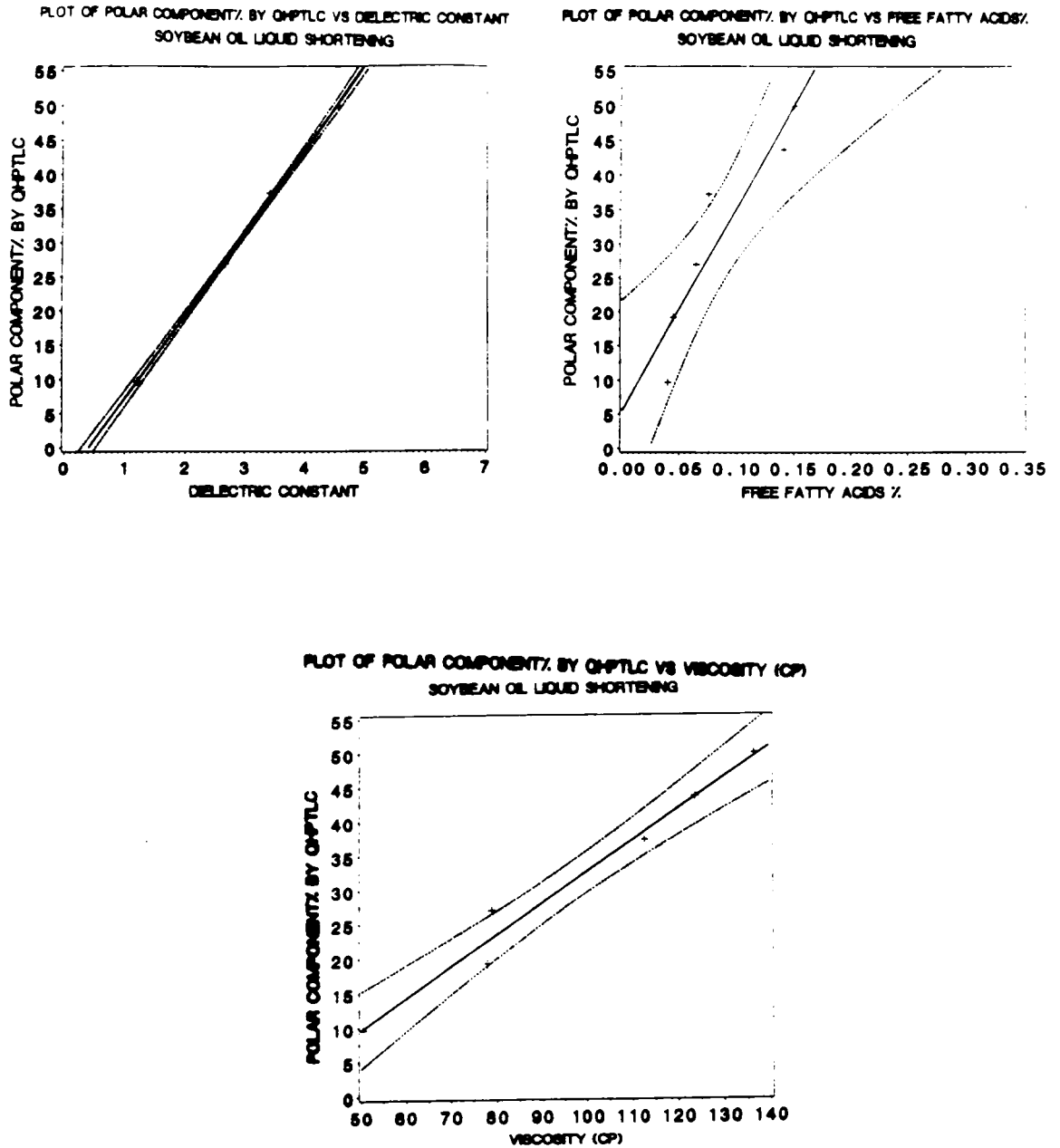


Fig. 29. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

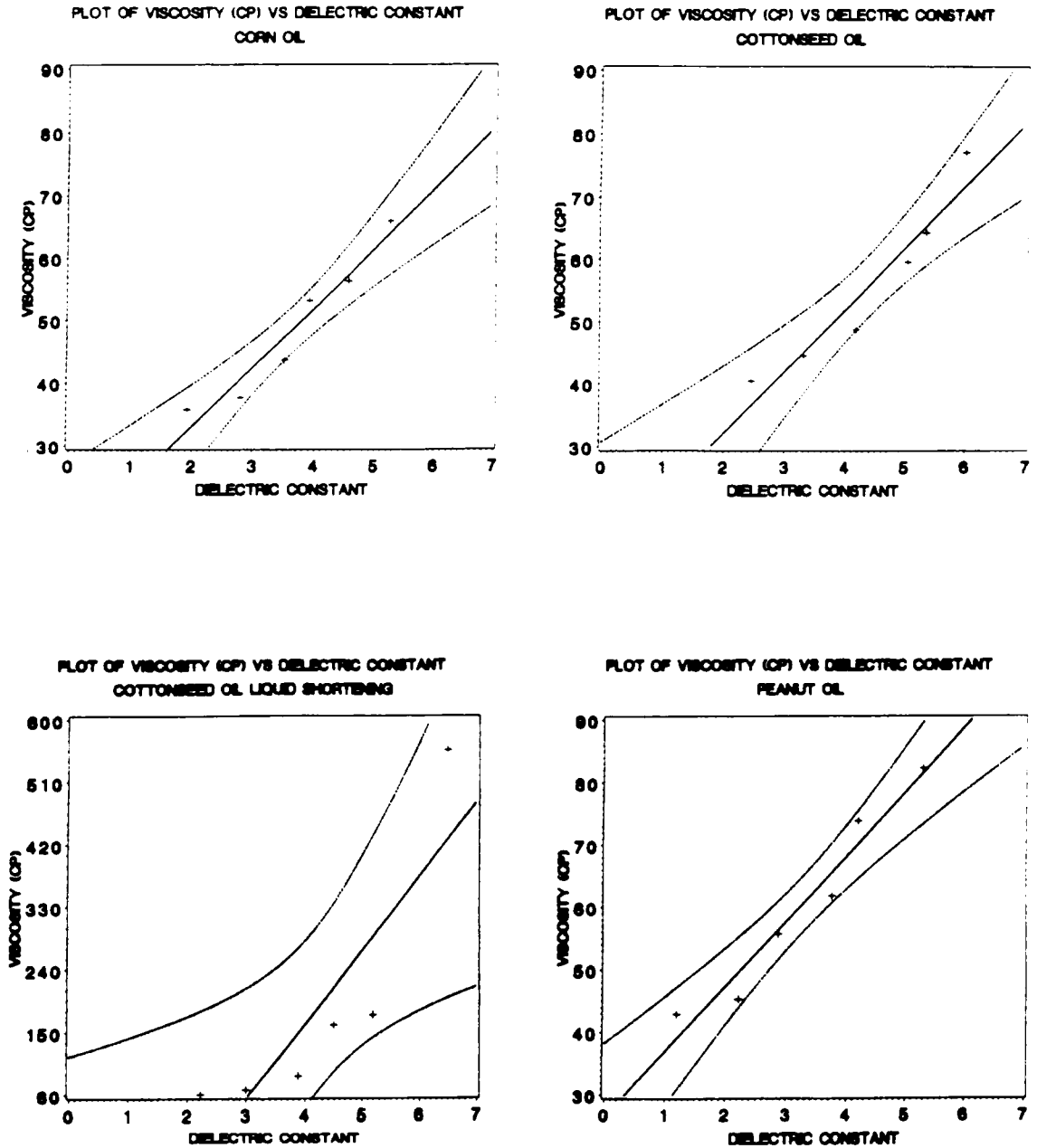


Fig. 30. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

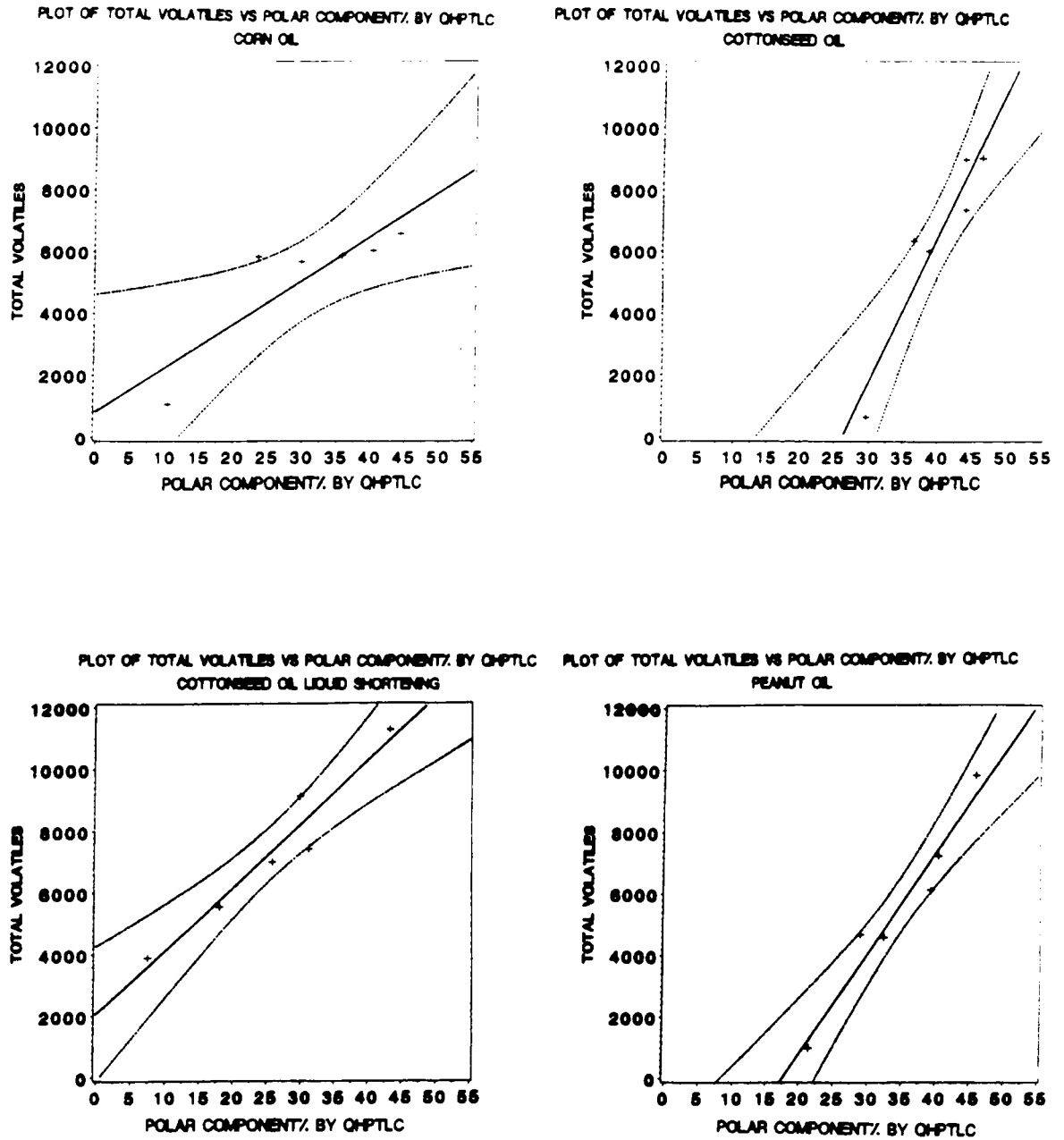


Fig. 31. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

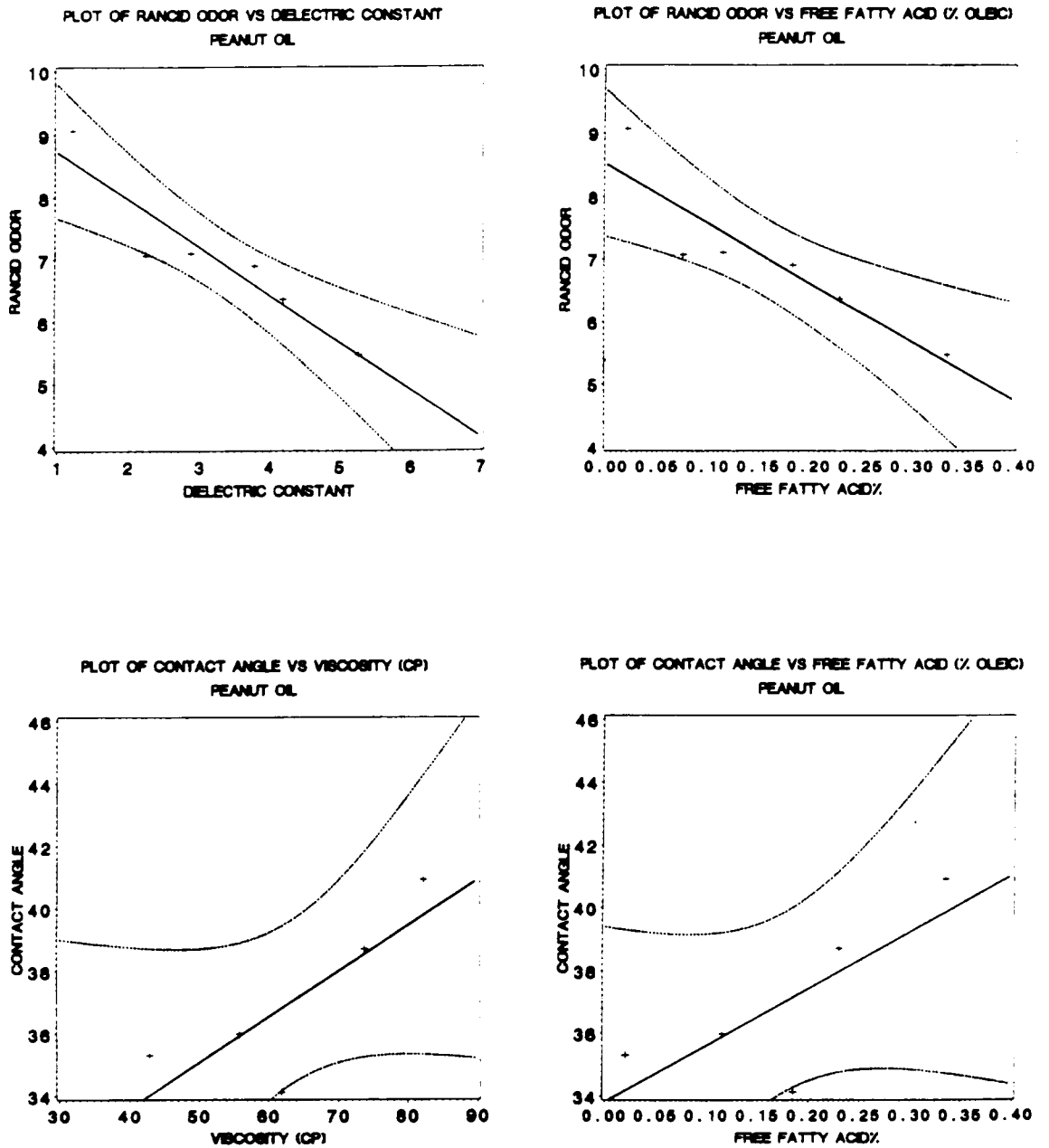
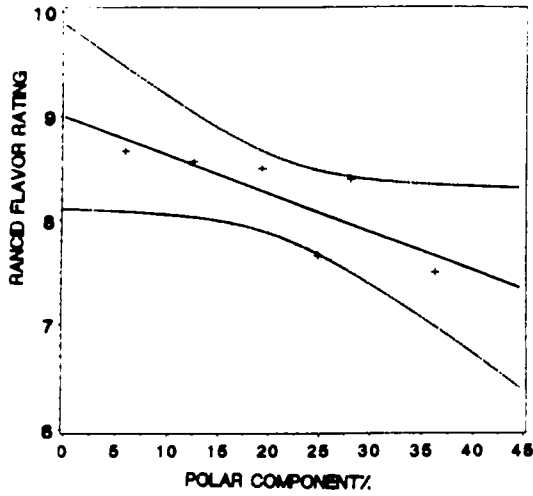
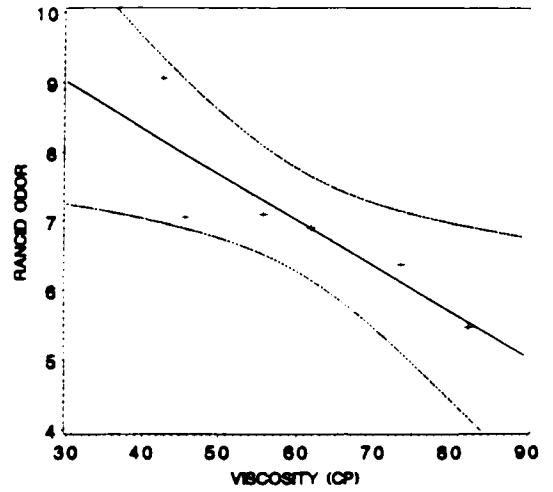


Fig. 32. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

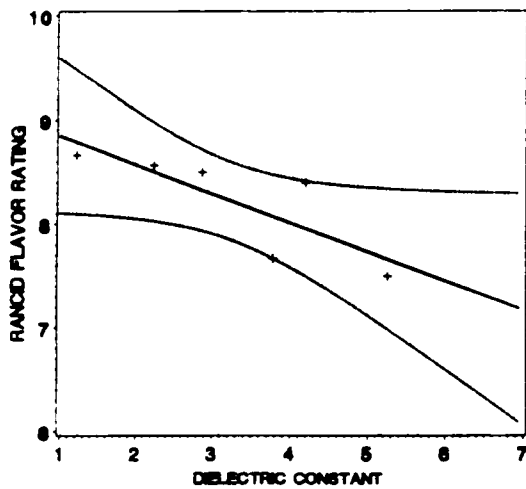
PLOT OF RANCID FLAVOR RATING VS POLAR COMPONENT%, PEANUT OIL



PLOT OF RANCID ODOR VS VISCOSITY (CP) PEANUT OIL



PLOT OF RANCID FLAVOR VS DIELECTRIC CONSTANT PEANUT OIL



PLOT OF RANCID FLAVOR VS FREE FATTY ACID (% OLSC) PEANUT OIL

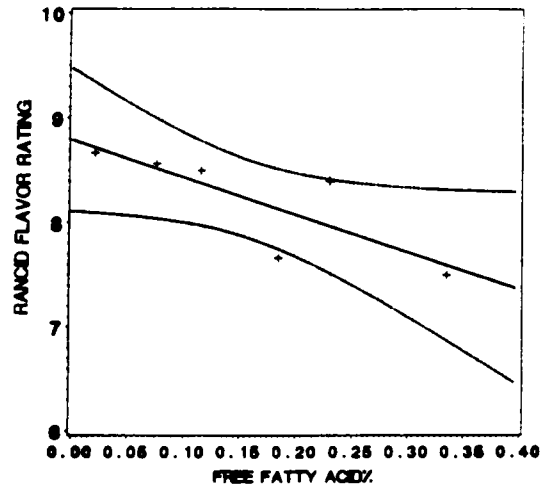


Fig. 33. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

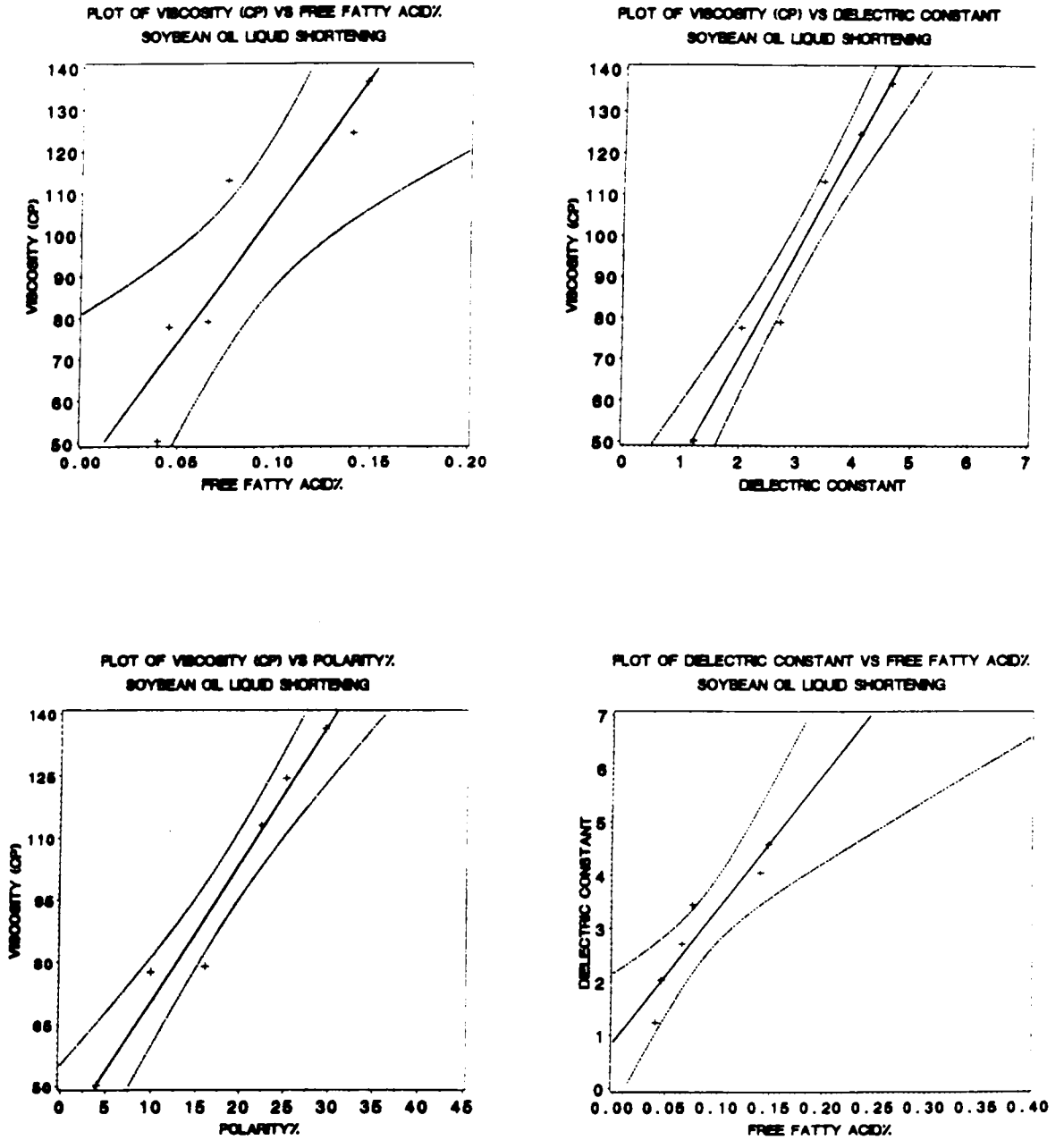


Fig. 34. Plots of Linear Regressions Relating Two Analyses Which Were Conducted Under Static Heating Conditions for Different Frying Shortenings.

APPENDIX

H

Typical Chromatograms of Frying Oil Triglycerides  
as Influenced by the Commercial Frying  
Conditions



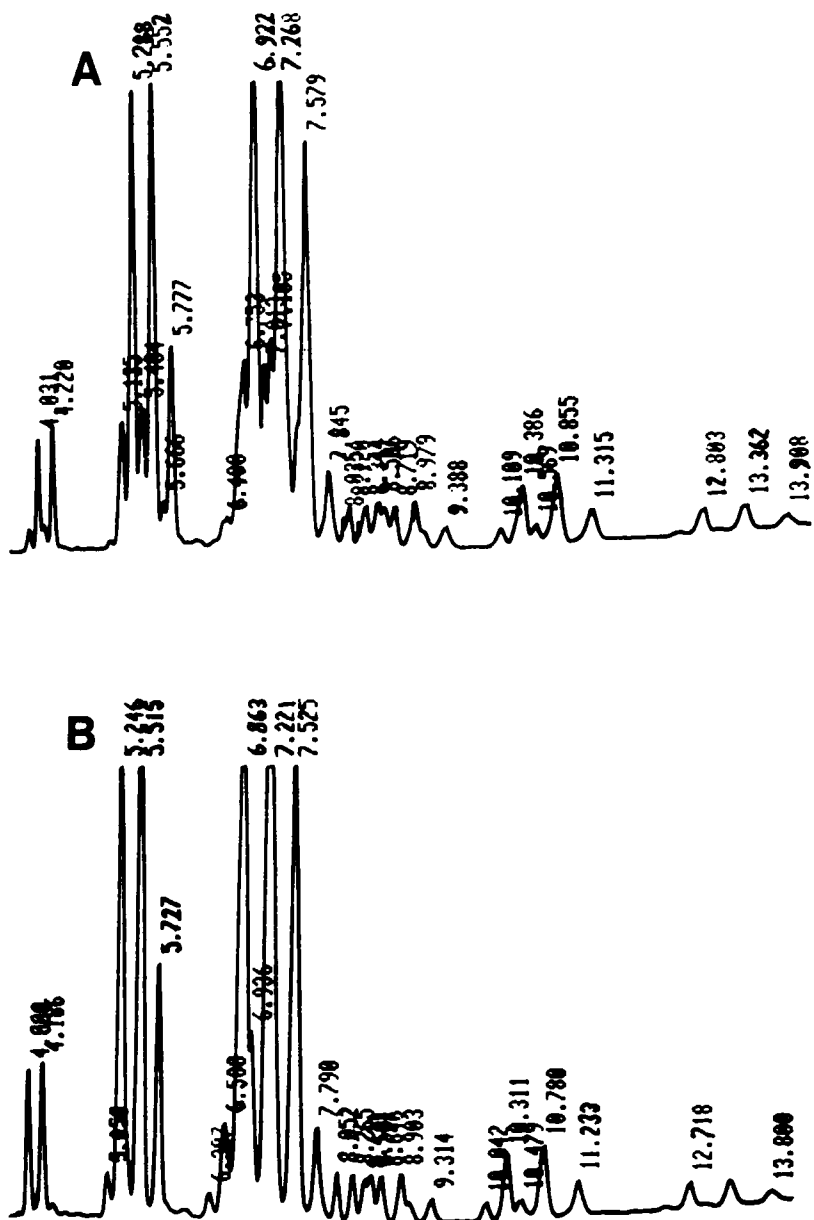


Fig. 35. Effect of commercial frying time (Days) of chicken nugget on triglyceride profile in peanut oil. A-unheated; B-heated to 7 days.

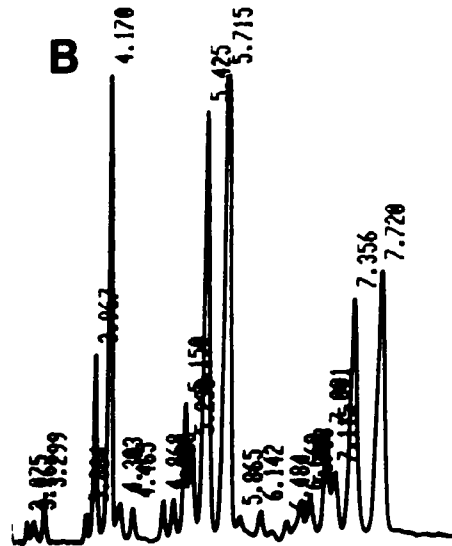


Fig. 36. Effect of commercial frying time (Days) of chicken nugget on triglyceride profile in cottonseed oil liquid shortening. A-unheated; B-heated to 7 days.

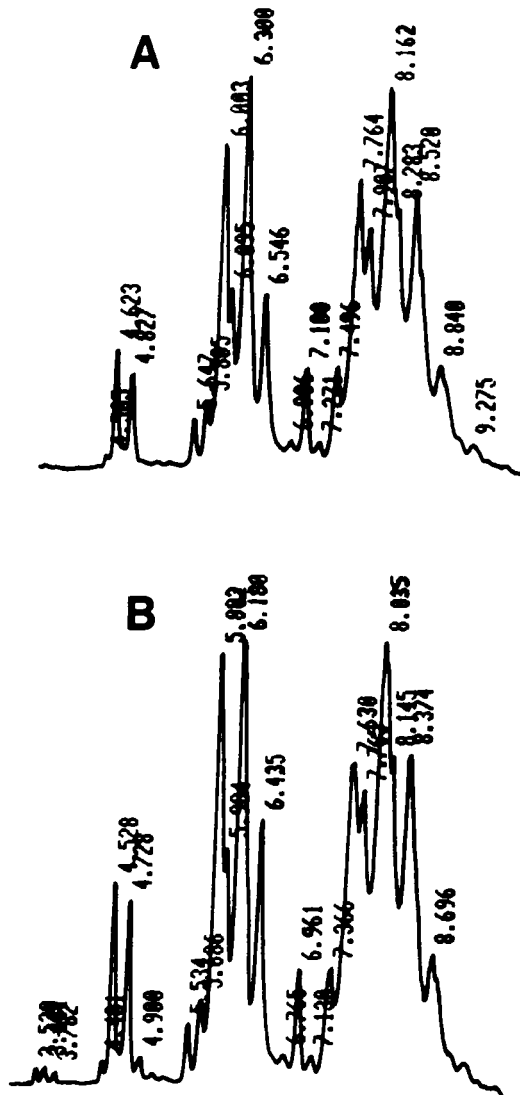


Fig. 37. Effect of commercial frying time (Days) of chicken nugget on triglyceride profile in soybean oil liquid shortening. A-unheated; B-heated to 7 days.

## APPENDIX

### I

Polar Component% by Quantitative High Performance  
Thin Layer Chromatography (QHPTLC).

## SOLVENT SYSTEM OPTIMIZATION.

The selection of an appropriate solvent system to separate nonpolar lipids from the polar fraction into two defined bands representative of the actual polarity in the shortening was the objective of this preliminary study.

The solvents tested for this purpose, with and without acetic acid, were petroleum ether, methanol, and diethyl ether (Table 2) as presented in chapter III. Twenty-two solvent combinations were tested. However, systems #14 (99:1 pet. ether; diethyl ether) and #21 (100:2 pet. ether; acetic acid) both provided reasonable resolutions, except that the polar band (lower) exhibited some diffusion. System #22 (100% pet. ether) resulted in no separation of the polar and nonpolar fractions. Poor resolutions, band broadenings and high  $R_f$  values of the polar fractions were given by the remaining solvent systems, namely #1-13 and #15-20. Since no satisfactory results were obtained from these systems, new solvents and solvent combinations were attempted using eight solvents (Table 3) as presented in chapter III. These solvents were acetone, chloroform, benzene, xylenes, acetonitrile, ethanol, diethyl ether, petroleum ether, used with or without the addition of acetic acid.

Solvent systems #1-27- except #6, #8, #20 and #22- gave unacceptable resolutions, band broadenings and high  $R_f$  values of the polar fraction. Both systems #6 (100% benzene) and #8

(100% xylenes) produced two sharp bands: the first from the baseline was polar and the second was nonpolar. This was confirmed by spraying the same plates with polar and nonpolar fractions separated by an open column chromatographic method. Improved resolution of the two bands was subsequently obtained by the addition of 50% petroleum ether in system #20 and #24. Solvent #24 (50:50 xylenes: pet. ether) was slightly better than #20 (50:50 benzene: pet ether) and was selected for further modification, even though the upper nonpolar band was insufficiently wide to provide a desirable polarity ratio. Consequently, two new ratios were attempted, #25 (60:40) and #26 (40:60) of (xylenes; pet. ether) for both systems. System #25 provided improved resolution; hence several systems were evaluated by increasing the xylenes ratio in #27, #28, #29. However, #28 had shown a slight diffusion of the nonpolar band at 95:5 of xylenes: pet. ether. Eventually solvent system #29 (85:15 xylenes; pet. ether) was selected with no further change in its polarity (Fig. 10). From the results obtained, it was observed that petroleum ether was very nonpolar and did not have sufficient affinity for the nonpolar fraction of the shortening and a separation did not occur. When xylenes was added, up to 85%, the polarity of the system was increased enough to produce the best resolution. The  $R_f$  value of the polar band was almost zero using this solvent environment.

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