THE SOLVENT EXTRACTION OF OIL FROM ACORNS

by

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ABSTRACT OF THESIS

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The possibility of using acorns for food has often been discussed in the literature,[18,21,27] but except in times of scarcity of other foods, acorns have been utilized in any quantity only by wild animals or semi-domestic pigs.[22] There are some references[14,18] to the possible uses of acorn oil, but there is no record of any large scale work with this material. This investigation was undertaken to handle a quantity of acorns, 847 pounds; (1) to disclose what particular problems there are in processing this material for acorn oil; (2) to demonstrate a practical method of obtaining acorn oil; and (3) to relate solvent extraction theory to the experimental extraction of this oil.

There are three general methods for obtaining oil from oleagineous seeds; expression, expelling and solvent extraction. Solvent extraction was selected as the most promising method for obtaining oil from acorns since it can be applied to any oil-bearing material and is particularly suited to the removal of oil from materials of low oil content. The oil content of acorns is reported to vary from 2.5 per cent[25] to 16.0 per cent.[23]

Acorns. One of the problems of this investigation was to determine what type of material could be expected

on offering a price for acorns. Inquiries showed that three cents a pound would be a sufficient inducement to produce a supply of acorns adequate for experimental purposes. The Boy Scounts of Gainesville, Florida and the Anderson Hardware Company of Anderson, South Carolina were the suppliers. There were 361 pounds of acorns sent from Florida and 485.5 pounds from South Carolina. Acorns were ordered from Arkansas, Virginia and New York State, but none were received from these localities.

It is the author's opinion, based on his attempts to get acorns and on his personal observation, that acorns are seldom as abundant as popularly supposed and that harvesting for commercial utilization will be difficult.

The nuts which were supplied were of poor quality. The best lot obtained, the Scrub Oak acorns from Florida, Quercus catesbaei, consisted of 75 per cent sound nuts and 25 per cent of acorns which were either wormy or sprouted. The South Carolina acorns were only 41 per cent sound nuts. The quantity of oil which may be produced from sprouted or wormy nuts depends on the extent of growth or of worm damage. Most of the wormy nuts which were examined contained no meat at all. Fully sprouted nuts contain less than one per cent of oil.[16]

Before an acorn oil mill could be established on a commercial basis an extensive educational campaign would have to be conducted in order to acquaint prospective acorn harvesters with the type of material required for oil extraction. It is necessary that the nuts be gathered immediately after falling and stored in dry cribs.

Preparation of Acorns for Extraction. Most of the acorns were prepared for extraction by removing the shells in a winnower and fanner, built by the J. H. Lehmen Company of New York for Rockwood and Company and used to remove the shells from cocoa beans. It combines a roll for cracking and crushing the nuts with a revolving screen for separation of different sized particles and an air blast for blowing the light hull fragments away from the heavier nut meats. The acorns were shelled at a rate of about 200 pounds per hour. Smaller quantities of acorns were shelled by crushing in a Sturtevant No. TR 32 Two Roller Mill, followed by separation of shells and meats in a Raymond No. 41344 Laboratory Mechanical Separator with all the whizzers removed. The operations carried out in preparing the acorns for extraction are given in Figure 1, Preparation of Acorns for Extraction.

There are a number of methods of preparing seeds for oil extraction. The most satisfactory of these, according to the experience of soybean oil producers, is to prepare a thin flake, the thinner the better. These flakes are readily penetrated by solvent but hold their shape after oil removal so that drainage of miscella from extracted solids is rapid. Rockwood and Company[3,4] have had good success in extracting crushed expeller cake, sized to be retained by an eighth inch mesh screen and passed by a half inch mesh screen. Expelling is a hot pressing operation which produces a porous cake, probably due to the formation of steam within the material passing through the expeller. Solvent penetrates the porous expeller cake fragments easily and these fragments, like the soybean flakes, do not break up on removal of oil. Grinding produces a large per cent of fine flour, evident after oil removal, which cannot be handled by any of the commercial extractors described in the literature as being in current use.[4,8,10,11,12]

Acorn expeller cake was prepared by using an Anderson Duo Expeller on the acorn meats from the winnower and fanner. This expeller was used at Rockwood's plant. The expeller cake was crushed and the material retained by an eighth inch screen and passed

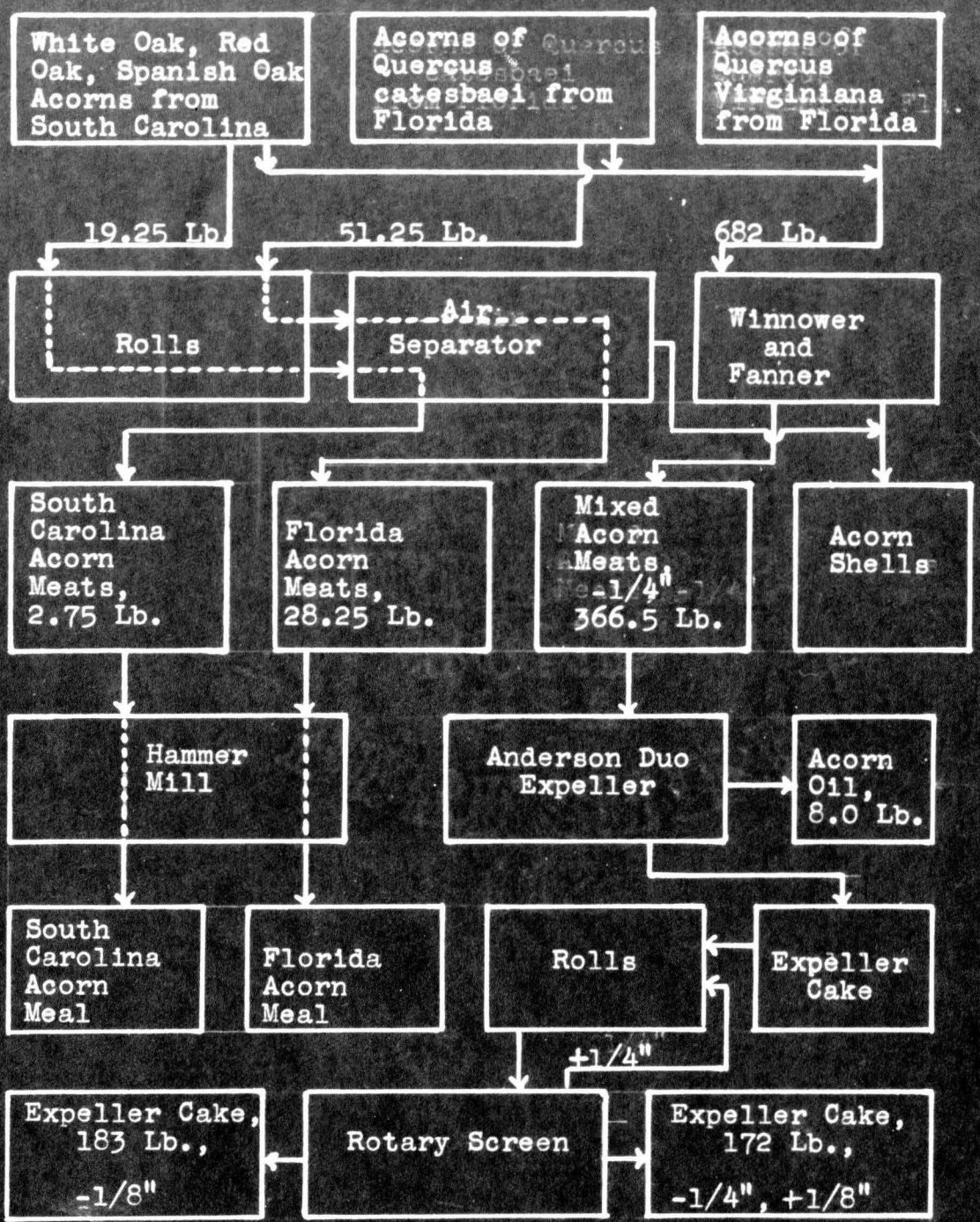


Figure 1.

Preparation of Acorns for Extraction.

by a quarter inch screen was extracted. Since at least 300 pounds of acorn meat were required for one expeller run most of the acorns were used unsorted for this experiment. Carefully selected acorns contain up to 40.0 per cent of n-butanol extractable material whereas the unsorted material fed to the expeller contained only 13.3 per cent of such material. The results of the expeller run show that acorn meats can be handled by an Anderson Duo Expeller. A good many runs would be required to establish optimum expelling conditions. During the first part of the 65 minute run the acorn meat was fed as it was. The first cake was crumbly and not firm. Water was added to the meat during the second part of the run and a much stronger and harder expeller cake was formed.

Methods of Extraction and Choice of Solvent. Two extractors were available for processing acorns on a pilot plant scale. The first of these consisted of a mixing tank, (a one gallon porcelain ball mill jar), for solvent and meal, followed by a Tolhurst No. T-7893 centrifuge to separate extracted solids and miscella. Extraction followed Elgin's[5] pseudo-countercurrent extraction scheme. This scheme is presented in Figure 2, Flow Diagram of a Three Stage

Centrifuge Extraction. Hunter[13] shows that such a scheme gives a deviation of 3 to 10 per cent, depending on the stage, from a truly countercurrent extraction when five cycles are used with four stages. He demonstrates that this deviation becomes less than two per cent for all stages with ten cycles. Three and four cycles were employed with two and three stages in these centrifuge extractions. A considerable deviation from a truly countercurrent process is therefore to be expected.

The second extractor was of the continuous chain type. It consisted of a fourteen foot length of two inch standard galvanized steel pipe bent so that a short section was vertical with the horizontal plane and a long inclined section beyond the bend made an angle of about 25° with this plane. A 28 3/4 foot continuous chain made up of No. 45 malleable detachable links with No. 45 C-1 flights every fourth link ran in at the top of the vertical section, up the inclined section and out at the top of this latter section. The extraction section, that part of the incline from the bottom of the bend to the solvent inlet, was 6 1/2 feet long. The solid material being extracted was carried up the incline by the chain against a counterflow of solvent. There was a 3 1/2 foot drain-

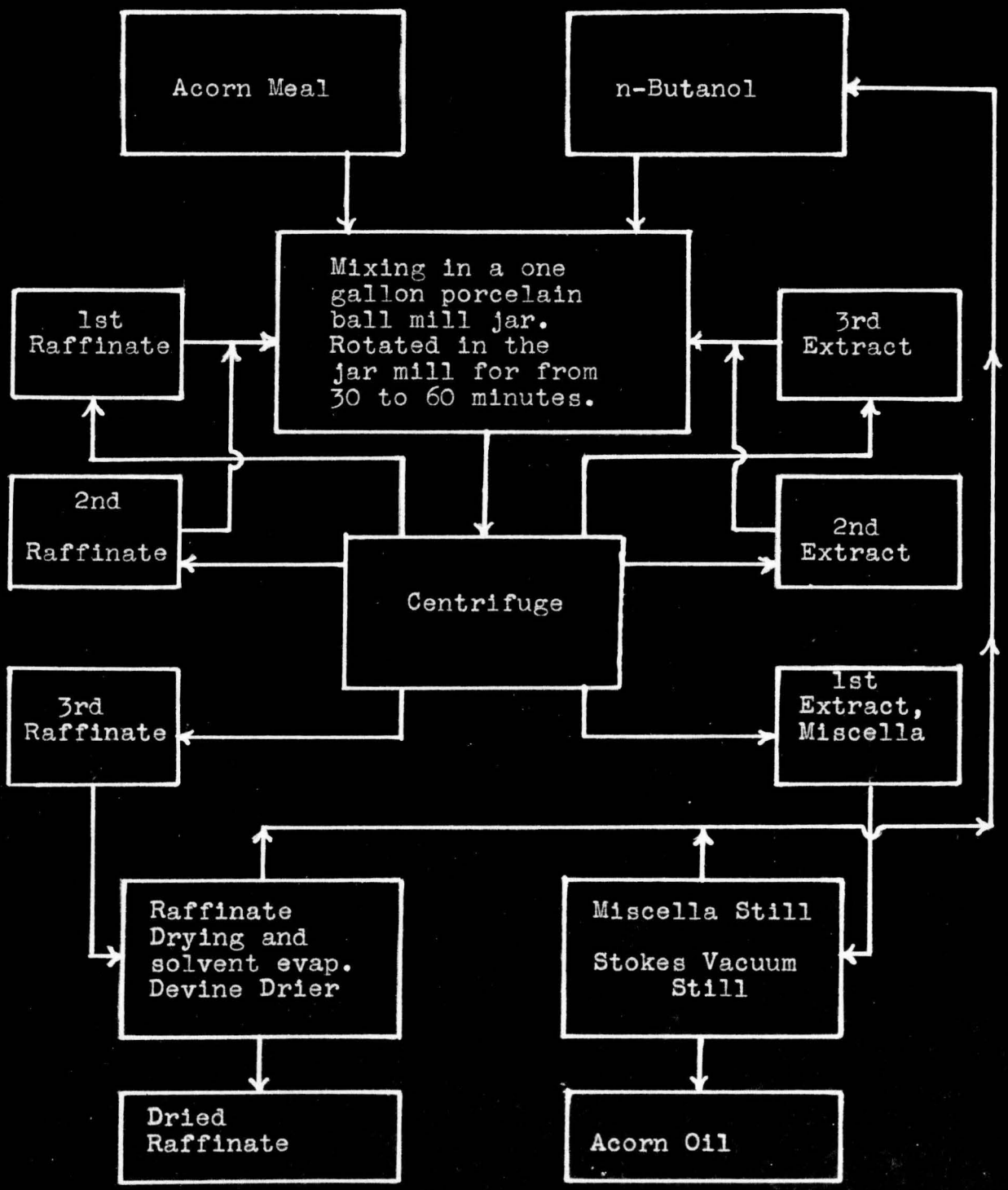


Figure 2.

Flow Diagram of a Three Stage Centrifuge Extraction.

age section above the solvent inlet. The chain carried the solids through this drainage section before running out of the pipe and dropping the drained solids onto a discharge apron. The miscella flowed out of the vertical section at the solvent inlet level. This continuous extractor is shown in the diagram, Figure 3, The Continuous Extractor, and in the photograph, Figure 4.

Neither of these extractors was solvent tight so that no volatile nor dangerous solvent could be used with them. The usual commercial oil solvents are low boiling hydrocarbons[15,19,20] and their chlorinated derivatives.[4,7,26] These solvents are both volatile and dangerous - the hydrocarbons because of their flammability - the chlorinated hydrocarbons because of their toxicity. n-Butanol was selected as solvent because:- it is not very volatile, vapor pressure of 4.3 mm. of mercury at 20°C. and of 18.6 mm. at 40.0°C.; it was not dangerous to use, its flash point is 35°C.; preliminary experiments indicated that it was a satisfactory solvent for acorn oil.

Centrifuge Extractions. The feed in the centrifuge extractions consisted of acorn meal of which 83.2 per cent passed through a standard 35 mesh sieve

and two per cent was retained by an 8 mesh sieve. The extractions were carried out under the conditions given in Table I, Experimental Conditions for Centrifuge Extractions, to give the results of Table II, Results of Centrifuge Extractions.

Continuous Extractions. The continuous extractor was operated according to the conditions given in Table III, Experimental Conditions for the Continuous Extractions, to give the results of Table IV, Results of Continuous Extractions. It was difficult to obtain consistent results with this extractor. This was due to the tendency for solid material to hold up in the pipe and to progress irregularly through the system. This difficulty was particularly noticeable with acorn meal which packed against the sides of the pipe so that the chain moved through a square duct composed of solvent-wet meal. This was an unstable condition. It was found possible to reach a rather unstable equilibrium by cleaning the system well before a run; filling it with solvent and establishing the solvent feed rate; and then feeding in the solids at the selected rate for the run for a period of about an hour. Under these conditions the fairly consistent results of Table IV were obtained.

The extraction operations are presented in Figure 5, Extraction of Acorn Oil.

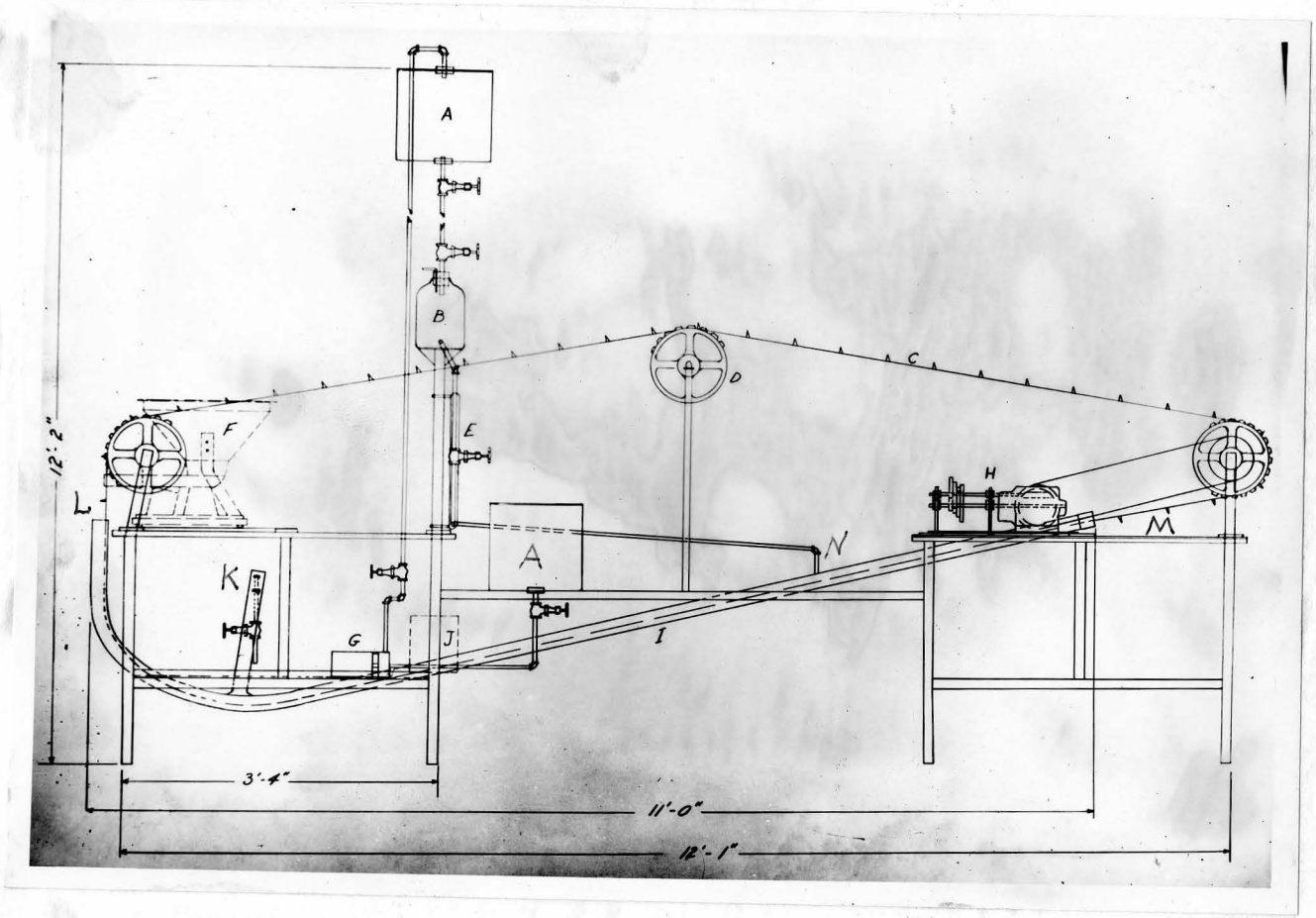


Figure 3.

The Continuous Extractor.

A. Solvent Reservoir
B. Calibrated Bottle
C. Continuous Chain
D. Sprocket Wheel
E. Rotameter
F. Vibratory Feeder
G. Pump
H. Motor and Reducing Drive
I. Two Inch Std. Pipe
J. Speed Regulator for Feeder
K. Miscella Outlet
L. Feed Inlet
M. Solids Discharge
N. Solvent Inlet

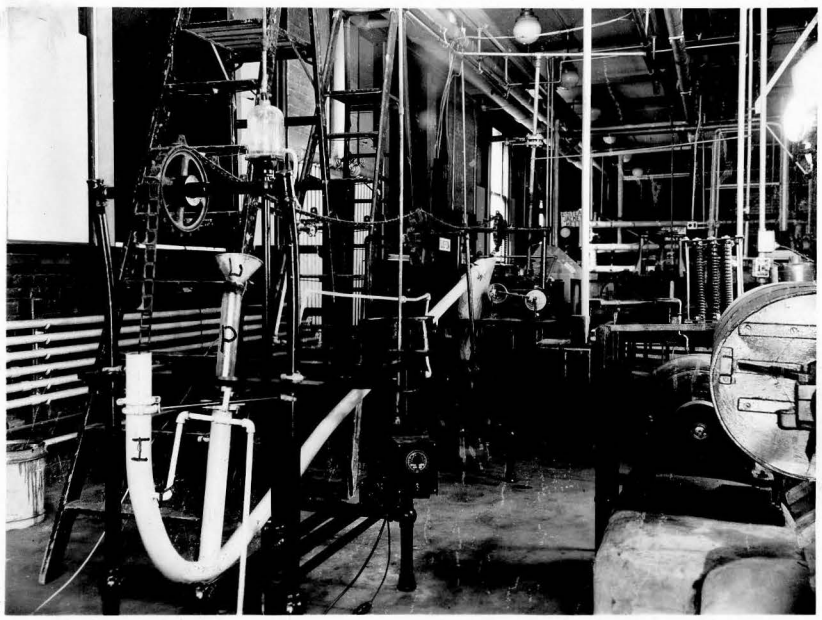


Figure 4.

The Continuous Extractor from the Miscella Discharge End.

I. Two inch std. pipe
L. Solid feed inlet
K. Miscella outlet
P. Two inch pyrex pipe

Table I.

Experimental Conditions for Centrifuge Extractions.

Experiment Number	1	2	3
System	inert, acorn oil, n-butanol	inert, acorn oil, n-butanol	inert, acorn oil, n-butanol
No. of Stages	3	2	2
No. of Cycles	3	3	3
Temp., °C.	25	25	25
Extraction time, Hours.	1.0	1.0	0.5
Feed:-			
Weight, Lbs.	2.0	2.0	4.0
Solute, acorn Oil, Per cent	40.0	36.0	38.6
Inert, Per cent	60.0	64.0	61.4
Solvent:-			
Weight, Lbs.	4.0	4.0	4.0
Solvent, Per cent	100.0	100.0	100.0
Overflow, Lbs.	3.81	3.28	4.07
Weight ratio, Solvent to feed	2.0	2.0	1.0

Table II

Results of Centrifuge Extractions.

Experiment number	1	2	3
Solute in miscella, Experimental, Per cent	18.63	18.4	28.6
Miscella, Weight, Lbs.	3.44	3.5	3.94
Oil in miscella, Lbs. oil extracted	0.64	0.65	1.13
Oil in feed, Lbs.	0.80	0.72	1.54
Oil experimentally extracted, Per cent	80.0	89.5	73.0
Solute in miscella, Theoretical, Per cent	22.10	19.3	36.0
Oil theoretically extracted, Per cent	95.0	93.7	92.2
Difference between per cent experimentally extracted and per cent theoretically extracted.	15.0	4.2	19.2
Difference, Per cent, (Difference divided by per cent oil theoretically extracted).	15.8	4.5	20.8

Solvent and Oil Recovery. The miscella was distilled under a vacuum of 25 inches of mercury in an F. J. Stokes Company No. 84260 Vacuum Pan with condenser and vacuum receiver. The results of solvent and oil recovery are given in Figure 6, Solvent and Oil Recovery. The still was equipped so that steam could be blown through the charge and traces of solvent were removed from the acorn oil by steaming out under vacuum. After this operation a two inch gate valve at the bottom of the still was opened and the residue was discharged. This residue divided into two layers, an upper acorn oil layer, and a lower water layer. The acorn oil was freed of impurities such as iron tannate and suspended solids by washing with water until the wash water was uncolored.

Approximately three gallons of crude acorn oil were produced by these pilot plant operations, about half of this oil resulted from the experiments reported in this paper and the rest came from acorn meal and expeller cake processed in preliminary experiments.

A sample of this crude acorn oil from Quercus catesbaei had the following characteristics:-

Specific gravity at 25°C., 0.907, index of refraction at 25°C., 1.4677, acid number 5.6, saponification number 190 and iodine number (Wijs') 106.

Table III.

Experimental Conditions for the Continuous Extractions.

Experiment	Series	Feed	Length of run, Minutes	Solvent fed, Lbs.	Solid fed, Lbs.	Raffinate removed, Lbs.	Miscella out, Lbs.
I	I	crushed cocoa expeller cake, 11.5 per cent cocoa butter	120	8.0	4.0	8.4	4.4
2			120	8.0	4.0	6.3	6.3
3			30	4.0	2.0	2.7	2.4
4	II	acorn meal, 38.5 per cent acorn oil	60	4.0	2.0	3.6	1.5
5			210	16.2	7.0	14.7	8.4
6			120	10.5	4.0	6.7	6.3
7			120	8.6	4.0	7.6	3.3
8			120	5.3	4.0	7.7	3.1
9	III	crushed acorn expeller cake, 17.8 per cent acorn oil	20	2.4	0.9	1.3	1.8
10			120	7.2	4.6	6.4	5.8
11			90	4.0	4.0	3.4	4.1

TABLE IV

Results of the Continuous Extractions.

Experiment	Oil extracted, Lbs.	Oil in feed, Lbs.	Oil experimentally extracted, Per cent	Liquid into system, Lbs.	Oil extracted, theoretical, Per cent	Ratio, solvent to feed
1	0.19	0.46	41.3	8.46	52.0	2.0
2	0.21	0.46	45.6	8.46	74.5	2.0
3	0.09	0.23	39.2	4.23	57.8	2.0
4	0.13	0.77	16.9	4.77	31.5	2.0
5	1.13	2.70	41.8	18.90	44.5	2.3
6	0.55	1.54	35.8	12.04	52.2	2.6
7	0.32	1.54	20.8	10.14	32.5	2.2
8	0.35	1.54	22.8	6.84	45.4	1.3
9	0.10	0.16	62.3	2.56	70.3	2.7
10	0.55	0.82	67.0	8.02	72.3	1.5
11	0.51	0.71	71.8	4.71	87.0	1.0

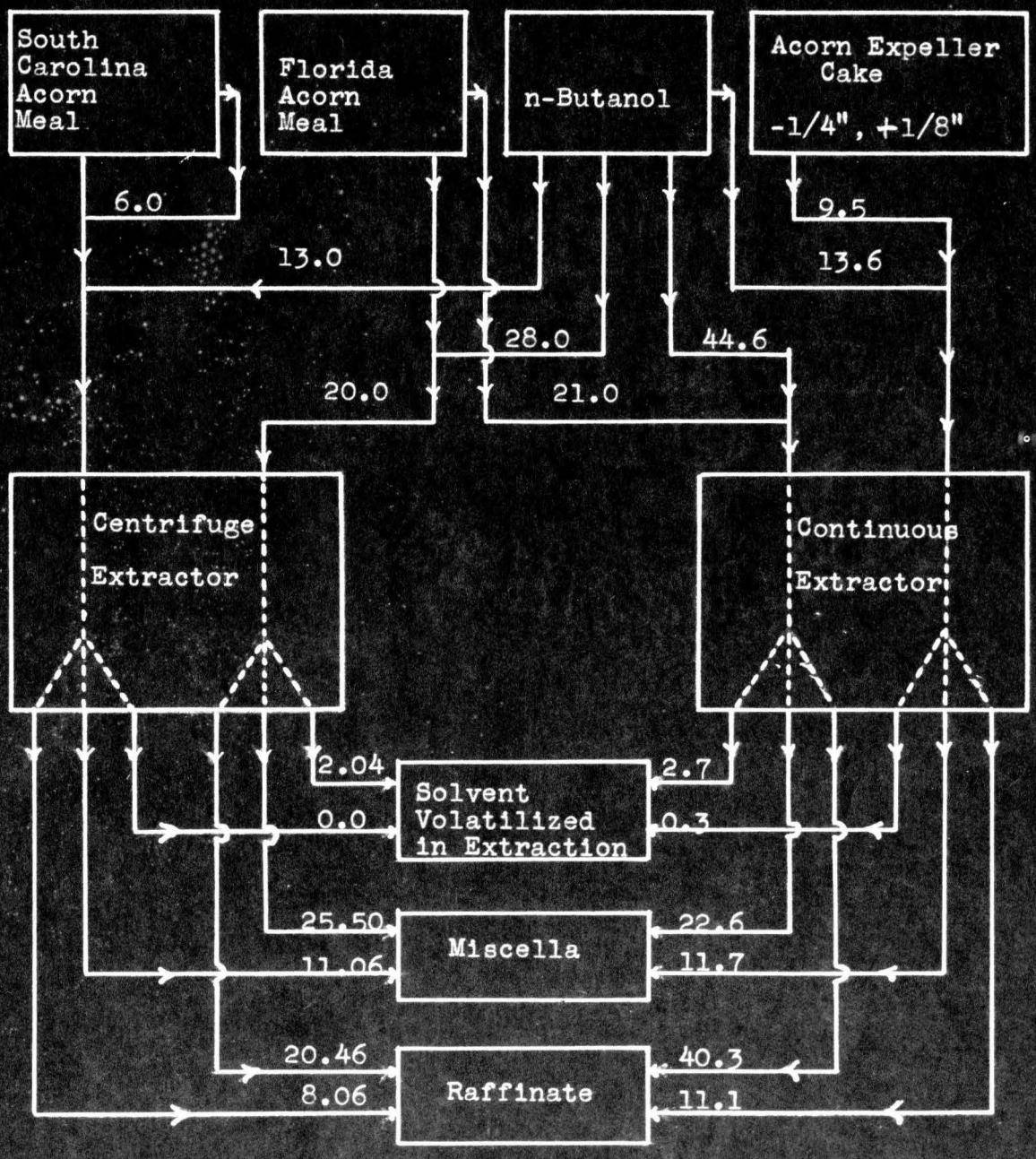


Figure 5.

Extraction of Acorn Oil

(All figures are pounds)

It was refined to give an oil of:-

Specific gravity at 25°C., 0.903, index of refraction at 25°C., 1.4684, acid number 2.6, saponification number 188 and iodine number (Wijs') 107.

In addition to the oil prepared by the pilot plant extractions there were the eight pounds of oil collected from the expelling operation with the V. D. Anderson Duo Expeller at Rockwood and Company. This expelled oil was filtered to free it from a small amount of solid material to give an oil of:-

Specific gravity at 25°C. of 0.907, index of refraction at 25°C. of 1.4669, acid number of 29.5, saponification number 192 and iodine number (Wijs') 105.

Relation of Solvent Extraction Theory to the Experimental Extraction of Acorn Oil. Solvent extraction theory has been developed and discussed by Evans,[7] Hawley,[9] Baker,[1,2] Ravenscroft,[1,24] Elgin,[6] and Kinney.[17] Theoretical calculations, following this theory, were made for the pilot plant extractions of acorn oil and cocoa butter with the results given in Tables II and IV. The calculations for the centrifuge extractions were for extractions of the experimental number of stages carried out under the experimental conditions. The number of stages in the continuous extractor was unknown until some of the experiments had

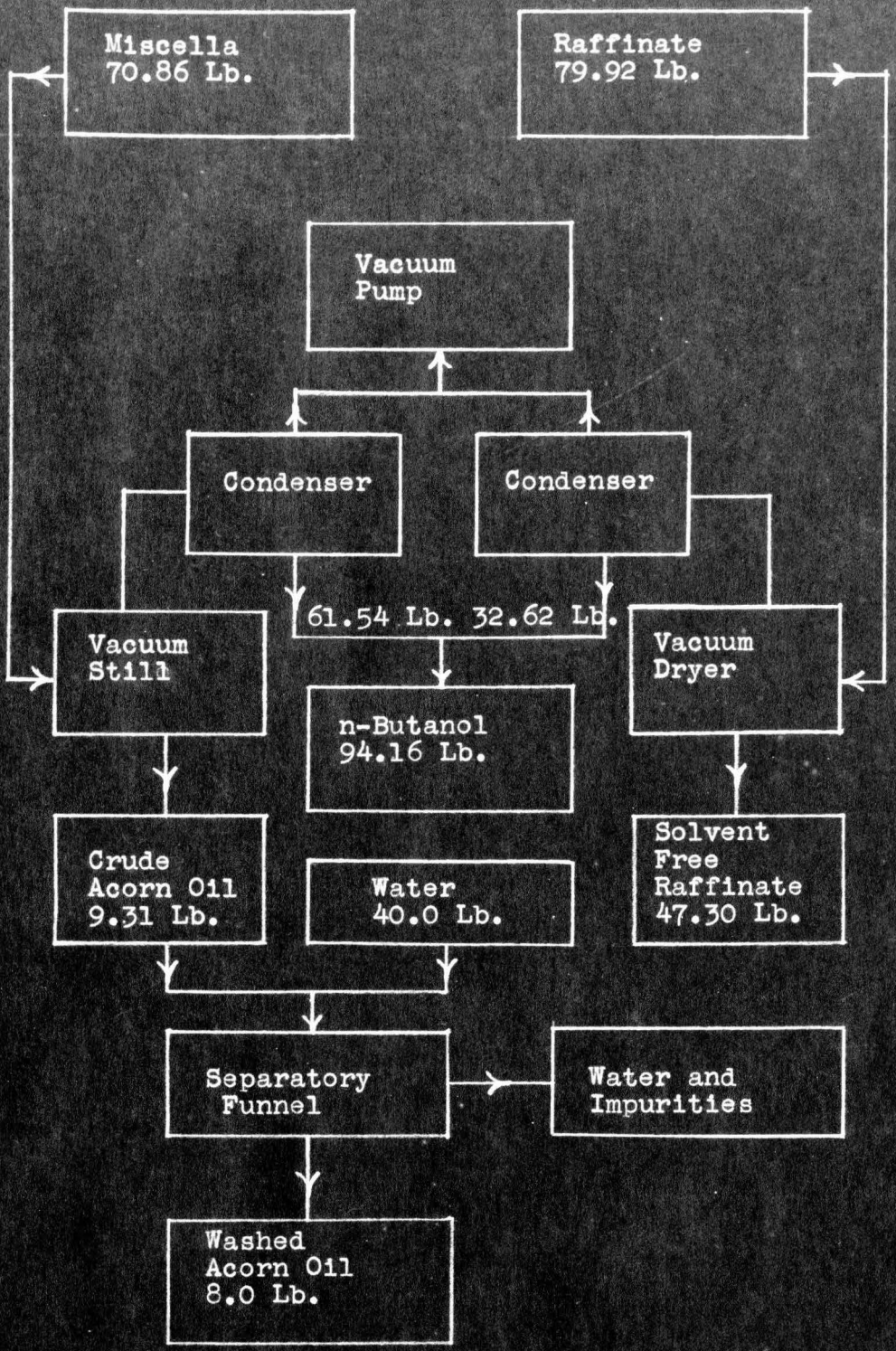


Figure 6.

Solvent and Oil Recovery

been completed. It was found that there was slightly less than one theoretical stage in this extractor. The calculations for the continuous extractor are based on its containing one theoretical stage.

Increasing the solvent-solid ratio in the continuous extractions increased the extraction in the case of acorn meal--as would be expected. Increasing this ratio led to decreased extraction in the case of the acorn expeller cake extractions--contrary to expectation. The effect of increasing solvent-solid ratios is plotted in Figure 7. Solvent-solid ratio is not a sufficient criterion for the prediction of the results of an oil extraction. Solvent-extraction theory, however, can be used successfully to predict the results of such extractions. Experimental vs. theoretical extraction is plotted in Figure 8 and this figure shows that this theory is capable of predicting the results of such extractions as those of acorn oil from acorn meal and acorn expeller cake as well as the extraction of cocoa butter from cocoa bean expeller cake. A comparison of the results for extraction of cocoa butter and acorn oil shows that, if the same extractor is operated under similar conditions on these three materials, there are the same number of theoretical stages in that extractor, whether the feed is acorn meal, acorn expeller cake, or cocoa bean expeller cake.

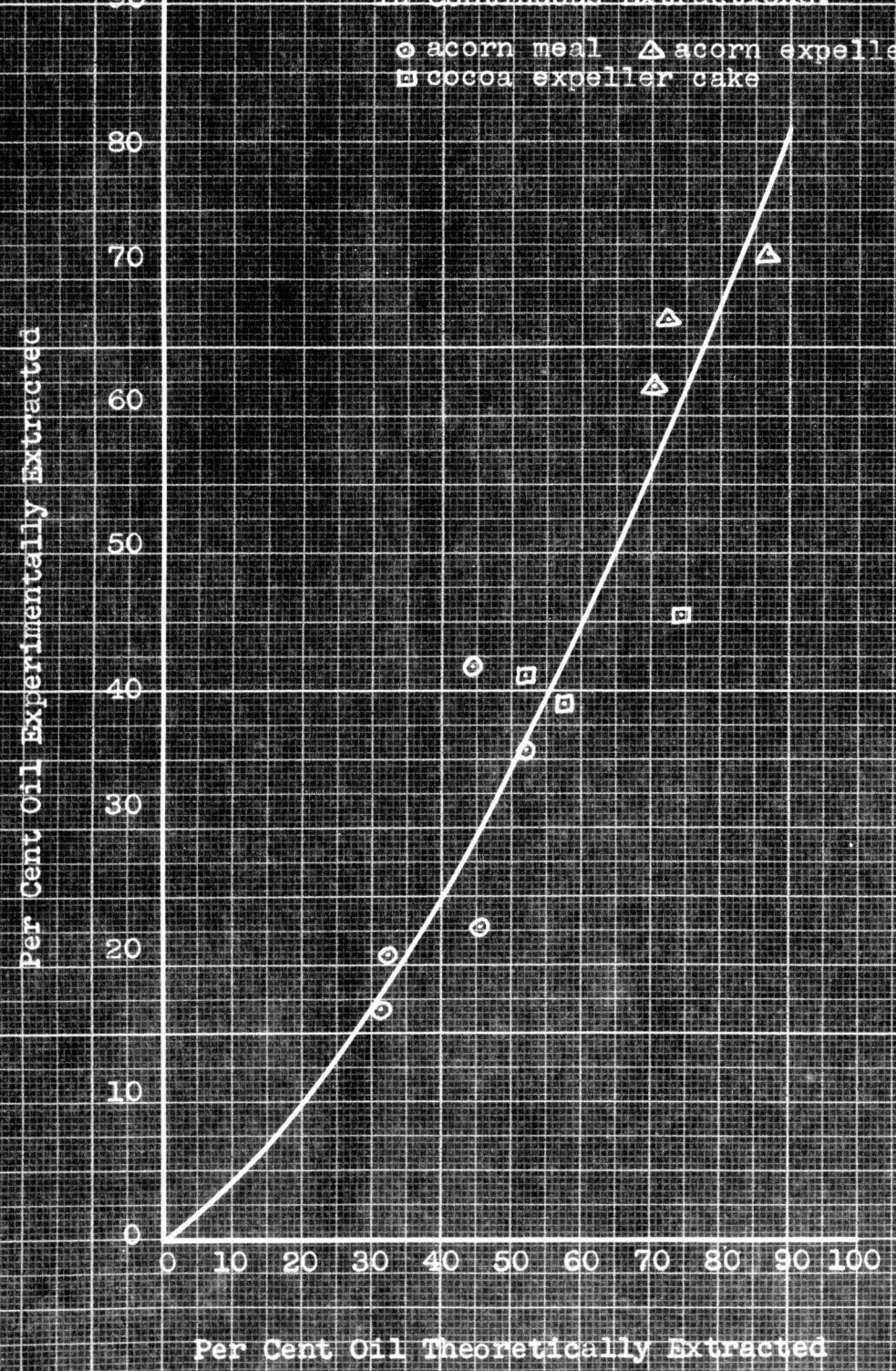
Figure 7.

Effect of Increasing Solvent-Solid Ratios on the Per Cent of Oil Extracted from the Feed.

⊙ acorn meal △ acorn expeller cake

Figure 8.

Experimental vs. Theoretical Extraction in Continuous Extractions.



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