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CROSSLINKING OF COTTON CELLULOSE BY LIGNIN-ISOCYANATE COPOLYMER

by

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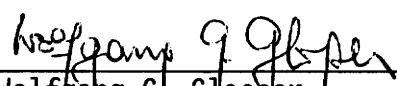
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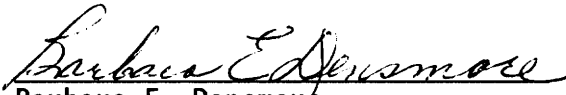
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(ABSTRACT)

Ethoxyl capped hydroxypropyl lignin (HP-EL) was used as a finishing agent for cotton fabric to impart the property of crease recovery to the fabric. The synthesis of the finish involved two steps:

Synthesis of phenyl N(3 - cyanato 4 methyl phenyl) carbamate by reacting phenol and Toluene diisocyanate (TDI) in a 1:1 mole ratio.

Reaction of the above prepolymer with the modified lignin (HP- EL) using a mole ratio of 1:20 to form a lignin carbamate.

The lignin carbamate finish was crosslinked to cotton cellulose fabric through carbamoyl linkages when the fabric was cured. There is empirical evidence for the chemical crosslinking of cellulose with lignin. The effects of the finish on the fabric physical properties like durable press rating, dimensional stability, tensile strength, abrasion resistance, warp and weft recovery angle were investigated. Analysis of the results showed improvement in the durable press rating and excellent retention of tensile strength and abrasion resistance. The results of warp and weft recovery angle tests which relate to durable press properties, suggest the need to further investigate the possibility of using a different chain-extended lignin to achieve improved results.

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Introduction

Cotton fabric is preferred by consumers for its comfort properties but in use, care and wear, it has not met the current consumer demands. The increasing competitiveness of synthetic fibers for "easy care" has threatened cotton's market-share. Although synthetic fibers have proven superior to natural fibers in end uses where high strength is required, they have not been proven superior in terms of comfort.

Lignin and cellulose are closely associated in nature. In wood and bast fibers, lignin acts as a hydrophobic binder and as a strength reinforcing agent. The world forests grow at a yearly rate of 7-9 billion cubic meters of biomass (Lindberg et al., 1989). About 50 million tons of lignin are formed as a by-product during the production of 140 million tons of cellulose and pulp from the biomass. With the separation of the cellulose fibers from the pulping liquor, the lignin fraction is disposed of as waste or used as an energy source for the recovery of inorganic pulping chemicals (Lindberg et al., 1989). Lin's study shows that over 750,000 tons of lignin are used per year (other than being used as a fuel) mostly as lignin sulfonates for aqueous dispersions (Lin, 1983). Hence lignin is considered one of the largest low value (in terms of utility and cost) industrial by-products (Glasser et al., 1984). The easy availability of a low cost natural polymeric material as a by-product has inspired researchers to investigate new uses for lignin as a full scale industrially used product.

Isolation of lignin in its unchanged state as it occurs in plants has been found to be impossible (Pearl, 1967). Pulping process adopted to separate lignin from cellulose fibers results in considerable degradation of lignin. Hence a majority of research on lignin is based on degraded or modified lignin. Though precise characterization of lignin still remains elusive due to its complex structure and insolubility, it has been accepted as a three dimensional structure formed by phenylpropane units through various linkages (Goring, 1989). Well defined chemical groupings have been identified in the complex structure of lignin. Extensive work has also been done on chemically modifying lignin to utilize it as a component in resins, epoxies and plastics.

Formaldehyde based finishing agents are widely used in the textile industry for durable press finishing of cotton fabrics. Interest in finding nonformaldehyde finishes have been increasing after carcinogenic effects of formaldehyde were known. Though many nonformaldehyde finishes have been proposed, none have become widely accepted largely due to high cost (Welch & Andrews, 1988). This strongly suggests the possibility of using lignin a low cost, under utilized natural polymer. Advances made in the synthesis of lignin-carbohydrate copolymers (Demaret, 1986) and incorporation of lignin in polyurethane foams (Hsu & Glasser, 1975) lend credibility for this research.

In this study, a chemically modified lignin is examined as a finishing agent for cotton fabrics.

Lignin review

Lignin is a naturally occurring complex biopolymer comprised of phenylpropane repeating units. In vascular plants, 25%-30% of dry plant weight is accounted for by lignin and it is associated with cellulose (Sjostrom, 1981). The study of lignin began with the formulation of its chemical structure. The multifunctionality and insolubility of the material have made the characterization of lignin very difficult. Most of the studies have been carried out by degradation or derivatization of lignin (Sarkanen & Ludwig, 1971). Advances in instrumentation and understanding of fundamental concepts in fields like polymer chemistry and biochemistry have led to a better understanding of the structure of lignin. There is no evidence of crystallinity in lignin and so can be considered amorphous (Sarkanen & Ludwig, 1971).

Various structures have been proposed and they include Braun's, Erdtman's and Alder's formulations to explain the structure of lignin. In 1968, Freudentberg proposed a structure which consisted of guaiacylpropane subunits with 25% of p-hydroxy phenylpropane and 5% of syringylpropane units. A schematic representation of Freudentberg's model is shown in figure 1. With the aid of computer techniques, a statistical structure for lignin (for both hardwood and softwood) has been developed by Glasser (Glasser & Glasser, 1974) and is shown in figure 2.

Lignin is chemically an aromatic polyether and the structure is formed by free radical polymerization of certain plant phenols (Ball, 1966). Coniferin (a basic chemical constituent of lignin molecule), a relatively stable compound has been crystallized from the sap of coniferous wood. In plants the enzyme glucosidase helps in splitting off the glucose molecule from the coniferin molecule to release coniferyl alcohol. The enzyme

peroxidase, removes hydrogen from coniferyl alcohol and produces a coniferyl radical that can exist in various mesomeric (resonance) forms. When the coniferyl radicals are oxidized they combine to form dimers which further polymerize to lignin or lignin-like products.

Figure 3 represents the phenylpropane building unit of lignin. The usual features of lignin that are utilized for modification and polymerization reactions are: phenolic hydroxy group; aliphatic hydroxy group and the unsubstituted 3- or 5- position on the C9 unit (Klashorst, 1989). The presence of aliphatic and aromatic hydroxyl groups and aldehyde groups offer useful reactive sites. Hence, lignin can be halogenated, nitrated, alkylated, hydrogenated and sulfonated and further reacted with isocyanate to form carbamate or urethane linkages. Model compounds which have a similar structure as the phenylpropane unit are normally used to investigate the chemistry of lignin. Some of the model compounds used are shown in the figure 4 (Wu & Glasser, 1984).

Though lignin occurs as a natural polymer with attractive structural features and many types of reactive functional groups, the lignin obtained from pulping is not very suitable for polymerization reactions. The harsh reaction conditions of pulping make lignin less attractive for such reactions (Huttermann et al., 1989). The pulping process converts lignin into a rather inert material. As compared to the original lignin occurring in plants the lignin obtained from pulping process has fewer phenolic groups and the number of alkyl aryl ether bonds are also considerably reduced. Chemically modified lignins have been studied extensively and have proven to make lignin more useful for further reactions. Christian et al. attempted to increase the number of reactive positions of lignin by reacting lignin with ethylene oxide and propylene oxide (cited in Hsu & Glasser, 1975). Hsu and Glasser extended the phenolic hydroxyl groups of lignin to form long chains of aliphatic alcohols, carboxylic acids or esters for selective hydroxyl

group reactivity (Hsu & Glasser, 1975). The reaction of lignin with maleic anhydride via a vinyl type copolymerization, simultaneously adding carboxyl groups to activate the phenolic hydroxyl groups for further oxyalkylation, has been successful (Hsu & Glasser, 1975). The reactions involved are shown in figures 5 and 6. Copolymers made from derivatives obtained from the reaction of lignin with propylene oxide improved the viscoelastic properties of lignin. Generally hydroxypropylation of lignin is carried out in a batch reactor under alkaline conditions at 180C for about 90 minutes. Details of the method are reported elsewhere (Wu & Glasser, 1984). Propoxylated lignins have secondary hydroxy groups as their primary reactive sites, whereas hydroxy ethyl lignin derivatives have primary hydroxyl groups (Glasser et al., 1984). A schematic representation of hydroxypropylated lignin is shown in figure 7 (Demaret, 1986).

The phenolic nature of lignin and its reactivity with formaldehyde have resulted in partial replacement of phenol in phenol formaldehyde resins (Nada et al., 1987; Lora et al., 1989). In one such study 35% of the resin solids in a phenol formaldehyde resin was successfully replaced with organosolv hardwood lignin (Cook & Sellers, 1989). Replacement of a petroleum based products with lignin, a biobased material opens new avenues for utilizing lignin.

Phenolic hydroxy groups of lignins have been utilized in the preparation of lignin epoxies by reaction with epichlorohydrin; esterification reaction with bis-chlorides; and polymerization with aziridines (Klashorst, 1989). When lignins are incorporated in urethane resins the aliphatic hydroxy groups in lignin are used and in many applications are oxyalkylated to afford more aliphatic alcohols (Hsu & Glasser, 1975). Other areas where lignin has found applications include nonformaldehyde glue for particle boards, wood adhesives, water soluble polymer systems, surfactants and dye dispersants (Klashorst, 1989). Successful work on copolymerizing lignin with carbohydrates

(Demaret, 1986; Koshijima et al., 1989) and the possibility of tailoring lignin to the end use suggest using lignin as a durable press finish for cotton, where crosslinking lignin with cotton would provide crease recovery property to fabric.

Modified or derivatized lignin, has been used for engineering plastics and other similar polymeric materials like polyurethanes (Hsu & Glasser, 1975). Incorporation of lignin into linear polyurethane foam requires that lignin be of uniform functionality. It has already been shown that oxyalkylation of lignin improves the flexibility and renders it as a product with uniform functionality (Wu & Glasser, 1984). Polyols have been synthesized from propylene oxide and lignin to yield a lignin polyol has been successful (Wu & Glasser, 1984). Extension of the propoxyl arm in hydroxypropylated lignin was investigated by De Oliveira. The number and the length of the arms have been found to have a strong influence on the properties of the end product (De Oliveira & Glasser, 1989). Reaction of isocyanates with the hydroxyl groups of lignin laid the foundation for the use of lignin in engineered plastics. Reaction of isocyanates with lignin simultaneously with or without the presence of poly glycols like polyethylene glycol or polybutadiene glycol have been studied (Rials & Glasser, 1984). In such studies the focus was on the effect of the change in properties in relation to the content of lignin in the end product (Rials & Glasser, 1984). It is thus documented that isocyanates can be reacted with the hydroxyl groups in lignin. The present study of utilizing lignin (with a blocked diisocyanate) to crosslink cotton by forming a network of urethane linkages with cotton fibers has close relation to lignin-based polyurethanes studied by Glasser et al. (Kelley et al., 1989).

The modified lignin used in this study is a chain extended hydroxypropyl lignin with partial ethylene oxide capping. The modification was done at the Brooks Center at Virginia Tech. Lignin was reacted with propylene oxide to extend the propylene oxide

arm and then reacted with ethylene oxide to cap some hydroxypropyl hydroxy groups arm. The capping reaction with ethylene oxide forms primary hydroxyl groups which are more reactive than the secondary hydroxyl groups. The chain extension acts as the soft segment and the resulting product is a dark viscous solution with a glass transition temperature below room temperature.

Various lignin compounds have been examined under different techniques for identification. The infrared (IR) spectra of kraft lignin and hydroxyethylpropyl lignin are shown in figures 8 and 9 respectively (Cardamone,1990). The common bands include the broad absorptions in the region of 3550 cm^{-1} - 3300 cm^{-1} indicating the presence of nonhydrogen bonded hydroxyl groups; bands in the region of 2930 cm^{-1} - 2870 cm^{-1} and at 1465 cm^{-1} indicate the presence of aliphatic structures consistent with C-H bending from methoxy groups and C-II side chains (Rials & Glasser, 1984); the regions 1750 cm^{-1} - 1650 cm^{-1} represents a variety of carbonyl, carboxyl and ester vibrations for lignins and hydroxyalkyl lignins (Glasser et al., 1984). Absorptions due to C=O stretching of aliphatic carbonyls can be noticed at 1653 cm^{-1} - 1718 cm^{-1} ; complex absorptions at 1400 cm^{-1} - 950 cm^{-1} have been assigned to oxygen containing groups (Durie et al., 1960).

1.2 Cellulose Review

Like lignin, cellulose is an abundant and renewable natural organic polymeric material. Peters found that a third of all vegetable matter is made up of cellulose (Peters, 1963). Though cellulose occurs in many different forms in most cases it does not occur in its pure form. In cotton fiber, cellulose content is over 90% and is one of the purest forms. Cotton also contains noncellulosic materials such as fats, waxes and

pectin, but they are mainly confined to the outer layers of the fiber and residual protoplasm in the lumen. Cellulose is described as a linear condensation polymer consisting of D-anhydroglucopyranose units usually referred to as anhydroglucose units or glucose units) joined together by β -1,4 glycosidic bonds (Nevell & Zeronian, 1985). Protruding from these chains are hydroxyl groups which are capable of forming intermolecular and intramolecular hydrogen bonds (Morton & Hearle, 1962). The intramolecular bonding occurs between the OH(3) and the pyranose ring. The intermolecular bonding between the OH(3) in one chain and the OH(6) in the adjacent chain. The formation of these bonds result in the cellulose molecules forming a sheet or planar structure which in turn form a three dimensional layered structure. These structures are held together by weak Van der Waal's forces. Parallel and ordered arrangement of these structures form crystallite strands which form the basic structure of elementary cellulose microfibril (Casey, 1980). The nature of the microfibril is that it is almost completely crystalline and is interrupted by dislocations due to mechanical processing, natural imperfections or twisted molecular crystallite strands or chain bonding disorders. These regions where they are disordered are referred to as amorphous regions and are on the surface of the microfibril. The disruption of hydrogen bonds gives rise to hydroxyl groups for reaction and it occurs only in the amorphous regions. The crystalline regions which are closely packed with microfibrils and any internal amorphous regions are inaccessible for reaction (Lewin, 1977).

The empirical formula of cellulose is $C_6H_{10}O_5$, which is configured structurally as an anhydro β - D glucopyranose unit. The chemical structure of cellulose is shown in figure 10. This shows the conventional and most frequently used way to represent cellulose. Studies done on cotton using X-ray diffraction, NMR and analysis of bond lengths led to the development of the chair conformation structure of cotton. The chair

conformation is shown in the figure 11 which shows that the $-CH_2OH$ and the $-OH$ groups as well as the glycosidic bonds, are all equatorial with respect to the mean planes of the rings. Though representing cellulose in the chair conformation structure has been adopted by some, the conventional representation is accurate based on the structure restrictions from intramolecular hydrogen bonding.

Cotton has one primary hydroxyl group (at position 6 in figure 10) and two secondary hydroxyl groups (at position 2 & 3 in figure 10). In general the reactivity of primary hydroxyl groups is higher than the secondary hydroxyl groups (Nevell & Zeronian, 1985). The $OII(2)$, $OII(3)$ and $OII(6)$ hydroxyl groups are ionizable and occur on opposite sides of the chain in each repeating glucosidic residue. Because of the inductive effects of neighboring substituents the acidity and tendency for (II^1) dissociation is $OII(2) > OII(3) > OII(6)$. Hence $OII(2)$ is most readily etherified. After substitution of $OII(2)$, the acidity of $OII(3)$ is usually increased and thus has a higher reactivity (Sjostrom, 1981). Thus $OII(6)$ is least likely to be substituted. But since it is a primary hydroxyl it will preferentially engage in esterification in acidic substituents and in general is least sterically hindered. Since the reaction depends on ionizability of an H from OII 's, $OII(2)$ is the most favorable followed by $OII(3)$, then $OII(6)$ depending on the prevailing factors.

Many techniques for measuring accessibility of hydroxyl groups in the amorphous regions to chemical agents and thus relating it to the crystallinity of the substrate have been explored in the past. Accessibility to hydroxyl groups depends on the kind of agent system having its own individual size, swelling and penetrating abilities (Nevell & Zeronian, 1985). Hence relating accessibility to crystallinity using a specific system can only be true for that specific system and cannot be generalized (Nevell & Zeronian, 1985). Rowland and Cousins determined that reduction in crystalline regions by the

attack of periodate can be used to estimate the percentage of crystalline regions in cotton (cited in Nevell & Zeronian, 1985). The study concluded that 20% of anhydroglucose units in cotton fibers lie in the amorphous regions. It is expected that the lignin finish will penetrate these regions and form crosslinks to give the durable press property to the fabric.

The formation and presence of crosslinks help in the recovery from crease formation. Vail determined that the level of crosslinking depends considerably on the process (Vail, 1985). Formaldehyde based resins have become very popular as durable press finishes and are presently being used to a large extent in the industry. Though most these finishes are chemically linked to cotton it is well known that formaldehyde is released due to hydrolysis. Stability to hydrolysis varies enormously depending on the variety of crosslinking resin used. The release also depends on external conditions like temperature, humidity and time (Hilden, 1986). Kamath and co-workers determined that the fabric pH played a major role in the hydrolysis of the crosslinks (Kamath et al., 1985). The effects of durable press finishing cotton fabric with DMDHEU on the physical properties of the fabric are shown in figure 12 (Rowland et al., 1983). It is clear from the plots that the increase in durable press rating is obtained at the expense of property value loss.

1.3 Cotton Finishing Review

Cotton cellulosic fabrics are very susceptible to crease formation (Marsh, 1963) due to their inability to recover from applied stress (Mark et al., 1971). Counteracting this serious defect of crease formation can be considered as one of the major achieve-

ments in the history of textile finishing (Marsh, 1963). The terms crease resistance and crease recovery are often used indiscriminately. While crease resistance depends on the rigidity, crease recovery depends on elasticity of the material (Marsh, 1962). A number of studies have concentrated on improving the property of recovery from creasing which is normally referred to as durable press property. Crosslinking of cellulose molecules for the durable press property increases the highly valued properties of crease recovery and dimensional stability. Such crosslinks prevent the movement of the chains and thus impart to the fabric the property of recovering from deformation and return to its original shape in which the cross links were formed. The changes in the fabric properties are dependent on the extent of the cross links and the condition of the fiber at the time the cross links were introduced. Some of the disadvantages of the process are the fibers becoming brittle, loss in extensibility, abrasion resistance, tensile strength and tearing strength. The loss in strength has been explained by reduction in proportion of chain molecules in the fiber which can be fully extended to resist applied stress (Nevell & Zeronian, 1985). Such losses in the physical properties of finished goods have been accepted as unavoidable side effects of finishing treatments (Abhyankar et al., 1987).

Application of starch to improve the crease resistance of cotton marked the beginning of the work done in the area of finishing of cotton fabrics. It has been recognized that crosslinking of cellulose is the best method to achieve the property of crease recovery and is being practiced in the finishing industry.

About two hundred years ago starch was used as a sizing agent to reduce abrasion during weaving and as a thickener in block printing. Starch also served to give a freshly ironed effect free from wrinkles but did not help in "crease recovery" of the fiber after deformation beyond a yield point (Marsh, 1963). Work done by Munier and Guyot in crosslinking viscose using formaldehyde was probably the earliest work on crosslinking

cellulose (cited in Andrews et al., 1986). Later developments included the use of melamine formaldehyde and urea formaldehyde which gave less strength loss than formaldehyde. In 1944, Cameron and Morton suggested crosslinking of cellulose with methyloxylamides (cited in Andrews et al., 1986). Steele and Giddings showed that crosslinking was the primary reaction for "crease resist" properties (Steele & Giddings, 1956). From then on development of crosslinking agents have progressed over the period of years, from early urea-formaldehyde, melamine-formaldehyde and phenol formaldehyde agents for wash and wear finishes to the modern methylolated cyclic ureas for durable press finishes. About 80% of the durable press finished fabrics today are finished with dimethyloldihydroxyethyleneurea (DMDHEU) (Andrews et al., 1986).

Precondensates of urea formaldehyde and phenol formaldehyde were considered the most appropriate to use for crease proofing or permanent press finishing processes. In this process the fabric was fully cured in the fabric stage at the factory. But problems like seam puckering, difficulty in the formation and retention of creases (where it was essential) were encountered when the fabrics were tailored (it was not possible to crease the fabric after crease proofing), and this led to the development of the durable press finishes. In this process the fabric was partially cured in the fabric stage and curing was completed after the garment was made.

Many studies (Goodson, 1984; Hilden, 1986; Reinhardt & Andrews, 1986; 1987) indicate that in spite of being converted into a resin like dimethyloldihydroxyethylene urea (DMDHEU) formaldehyde is released from the fabric during processing, storage (Andrews et al., 1988) and usage. Some of the factors which have been found to influence the release of formaldehyde are fiber composition, structure, impurities, the presence of dyes or other treatments; the finishing agent, particularly its chemical reactivity and stability; the catalyst, its activity and acidity; the concentrations of agent and cata-

lyst applied; the processing conditions such as time and temperature of curing and method of application; the presence of additives, the amount of free formaldehyde in the finishing system; after treatments and washing to remove unreacted material and by-products. Most of these factors have also been found to be inter reactive and cannot be considered in isolation (Reinhardt & Andrews, 1987). "Formaldehyde release" has been defined by the American Association of Textile Chemists and Colorists for the test method 112-1984 as

that formaldehyde evolved from the textile under the accelerated storage conditions of this test. It includes the free (unbound or occluded) formaldehyde, formaldehyde from unreacted resin, as well as that from finish degradation as a result of this test (AATCC technical manual, 1989).

The application of the finish is a standard procedure where the cotton fabric is padded (with a wet pick up of 80% -90%) with monomeric urea formaldehyde or melamine formaldehyde together with a suitable acid forming catalyst and other desired additives (like a wetting agent). After impregnation the cloth is dried for a few minutes (normally for 5-7 minutes at 65-70C). Curing takes place at higher temperatures of around 160C for 2-3 minutes. Low formaldehyde application methods such as foam finishing are also being practiced (Turner, 1980). Such methods reduce the free formaldehyde content in the fabric (Lewin, 1983). Addition of formaldehyde scavengers like diethyleneglycol and carbohydrazide have also been investigated (Perry, 1980).

Durable press finishes based on formaldehyde contain CH_2OH groups which form acetal linkages with the adjacent cellulose chains and hence form a crosslink. Since urea molecules have two reactive groups, methylol ureas can react with cellulose to give many reaction products (Mark et al., 1971). As shown in figure 13 and 14 the crosslinking can be brought about by two methods:

(1) Bi-functional crosslinking compounds condensing between the cellulose molecules

(2) Self-condensation of the resin before crosslinking with the cellulose molecules.

In durable press finished fabrics formaldehyde can be present in either "free" or "bound" state (Hilden, 1986). Formaldehyde chemically crosslinked with cellulose molecules are referred as "bound" and formaldehyde present without any chemical linkage to cellulose is termed "free". Figure 15 shows the various forms of formaldehyde which may be present in the resin solution and fabrics treated with the resin solution (Kim & Smith, 1985).

Free formaldehyde which can be present in the finishing liquor can eventually get transferred on to the fabric when finished. Elimination of the free formaldehyde in the finishing bath is almost impossible due to its state of equilibrium (Hilden, 1986). Partial hydrolysis occurs while the bath is made and also when it is left to stand before being used. Stability to hydrolysis varies with individual type of resins. Generally the rate of hydrolysis is very high below a pH 4 and above pH 8; and is lowest at pH 5 - pH 7. Uncrosslinked resin present on the fabric is another source of free formaldehyde in the fabric. The cleavage of the linkage between the crosslinking resin and cellulose is promoted under humid and warm conditions (Hilden, 1986). Loss of free formaldehyde is also strongly influenced by relative humidity (Reinhardt & Andrews, 1986).

The reactions involving the release of formaldehyde are shown in figure 16. Formaldehyde release from a durable press finished fabric can either be attributed to the cleavage of acetal linkages between the durable press finish and cellulose (C-O scission - bound formaldehyde) and/or the hydrolysis of N-CH₂OH groups (C-N scission - free formaldehyde). Temperature and pH of the fabric influence reactions (1) and (2) while

reaction (3) is influenced by the temperature dependent partition of formaldehyde between the cellulose and environment (water or air) (Kamath et al, 1985).

Extensive research has been done on determining the health hazards of exposure to formaldehyde. Details of the studies are covered in detail by Turoski (Turoski, 1985). A few studies indicate strong causal relationship between carcinogenic effects and exposure to formaldehyde. Consumer protection agencies and health organizations have been voicing their concerns over the problem of exposure to formaldehyde in working environments. As a result Government regulations stipulate the levels of formaldehyde in areas where it is being used. The threshold limit value (TLV), which is the maximum permissible limit of formaldehyde in the atmosphere varies from country to country. Due to the financial hardships in complying with U.S. Government regulations which permit only 1-3 ppm of formaldehyde in the atmosphere (Hilden, 1986) and in anticipation of the worsening of the situation the industry has been striving hard to find alternatives for formaldehyde.

Development of a nonformaldehyde finish would eliminate the problem of formaldehyde release from cotton textiles and apparel. A number of formaldehyde free crosslinking agents for cotton have been proposed (Welch & Andrews, 1988). Disadvantages like high cost, inadequate reactivity, fabric yellowing, excessive fabric tendering and toxicity have prevented many alternatives from replacing the formaldehyde based resins which are commercially used (Welch & Andrews, 1988). Welch showed that glyoxal was one of the few readily available nonnitrogenous cellulose crosslinking agent exhibiting high reaction rates required for durable press finishing of cotton. He also found that using aluminum sulfate and an alpha hydroxy acid effectively catalyzed the crosslinking reaction under mild curing conditions (Welch, 1983). Using diethyleneglycol as a coreactant for glyoxal in a 1:1 mole ratio and curing at 115C re-

sulted in a durable press rating of 4.0 with a 46.48% retention of breaking strength which is comparable to DMDIIEU (Welch, 1983). Dihydroxyimidazolidinones, dihydroxyethylene ureas performed well as nonformaldehyde finishes but did not become popular due to its cost and effectiveness (Frick & Harper, 1982). An adduct of methyl carbamate and gluteraldehyde applied by a special method gave results close to other formaldehyde free agents (Frick & Harper, 1982).

Most proposed finishing agents for cotton involve the formation of ester or acetal crosslinkages with the hydroxyl groups of cellulose. Lack of effective non-tendering esterification catalysts and the wide spread view that ester linkages are easily hydrolyzed by alkaline detergents resulted in very little progress of ester type cross linking agents. Promising results from using polycarboxylic acids has created new interest in the area of durable finishing of cotton. Polycarboxylic acids were found to be very effective as durable press finishing agents in the presence of alkali metal salts of phosphorus containing inorganic acids (Welch & Andrews, 1988). The durable press performance was comparable to that of DMDIIEU and better than DMDHEU in terms of strength retention. The property of being recurable was an added advantage with such finishes. Formation of durable creases was possible by moistening the treated fabric followed by ironing-in the desired crease at high temperature (Welch & Andrews, 1988). The formation of new creases at high temperature is made possible by the mobility of ester linkages, which is due to the transesterification by adjacent hydroxyl groups of cellulose. Once again the practicality of this is influenced by the cost of the suitable polycarboxylic acid.

Phenyl isocyanates have previously been used to crosslink cotton fabrics. Ellzey's study indicate improvement in breaking strength and elongation and decrease in wrinkle

resistance (Ellzey et al., 1962). Verburg and Snowden observed similar results when acetate fabrics were crosslinked with tolylene diisocyanate (Verburg & Snowden, 1967).

1.4 Objectives and Justification

From the discussions above the need for a low cost, nonformaldehyde durable press finishing agent for cotton fabrics is evident. The easy availability of lignin as a by-product of pulping and its network structure makes it a potential material for use as a durable press finishing agent. Though lignin is a thermoset polymer as it occurs in nature, modification reactions with alkoxide chain extension produces a lignin derivative with glass transition temperature declining to values as low as -40C. The precedent for this study is the successful incorporation of such derivatives in block copolymers where these derivatives alter the viscoelastic properties (Demaret, 1986).

The reaction of isocyanates with lignin and cellulose suggests the possibility of an investigation to chemically link lignin and cellulose. A diisocyanate with one isocyanate group attached to lignin while the other is attached to cotton will chemically link lignin to cotton. It is envisioned that due to the formation of crosslinks between lignin and cotton, the cotton fabric would acquire the property of crease recovery. The success of this investigation will result in a formaldehyde free durable press finish and also find a very important use for lignin in the textile industry.

The synthesis of the finish involves two steps. The first step is the synthesis of a prepolymer, phenyl N-(3-cyanato-4-methyl phenyl) carbamate (I). Tolylene diisocyanate was reacted with phenol using a molar ratio of 1:1 to cap only the -NCO group in the

para position, to yield the precursor product to lignin polyurethane (LPU) which will be applied to the cloth. The choice of tolylene diisocyanate is supported by the fact that the reactivities of the two isocyanate groups are different due to the steric hindrance of the methylene group, ortho to the 2- position isocyanate group. Selective capping of the isocyanate group in the para or 4- position is therefore favored. The fact that heat liberation of phenol regenerates the 4- position isocyanate group at a temperature of 130C, which is below 160C, the curing temperature of the fabric, justifies the use of phenol. The second step is the synthesis of the copolymer to be used as the finishing agent. HP-EL lignin is reacted with the prepolymer using a molar ratio of 1:20 (lignin to I), in the presence of a catalyst dibutyltin dilaurate. The stoichiometry was decided after determining the capacity of the modified lignin to react with the available isocyanate groups in the reaction mixture. Beyond a molar ratio of 1:20 the presence of unreacted isocyanate groups were confirmed. The solvent for the reaction is methylene chloride which dissolves both (I) and LPU. Syntheses of (I) and LPU were carried out at room temperature, to prevent the capping of the isocyanate group in the ortho position in the first step and to prevent the decapping of the isocyanate group in the second step. The reaction were allowed to proceed for 72 hours in air.

Methylene chloride was used as the medium of application of the finish to the fabric since the finish is not soluble in water. Padding, by a one dip and one nip was done using a 12 inch wide laboratory scale padding equipment. Since methylene chloride evaporates quite fast the fabric was pinned to metal frames in a dry condition and it also eliminated the process of drying in the oven before curing. Curing of the fabric was done at 160C for 5 minutes using a metal pin frame to hold the fabric without wrinkles.

Three levels of % add-on (as determined by the percentage increase in weight of the fabric) were chosen and the fabrics were durable press finished accordingly. The padding

bath was prepared on the basis of the wet pick-up of the fabric. The performance of the finish was evaluated by comparing the finished and unfinished fabrics for durable press rating, dimensional stability, tensile strength, abrasion resistance and recovery angle.

The hypotheses to be tested by this study are:

- * Lignin can be used to crosslink cotton.
- * Formation and presence of a polyurethane network by this crosslinking in cotton fibers will confer durable press properties to the fabric.
- * Some of the adverse effects of formaldehyde based finishes on the physical properties of cotton will be reduced by using the lignin finish.
- * Excessive crosslinking at high levels of add-on will deteriorate the physical properties of cotton.

2.0 Materials and methods

2.1 Materials

Diisocyanate	2,4 Tolylene diisocyanate (TDI) supplied by Mobay Corporation (Pure grade without 2,6 TDI isomer)
Phenol	Pure grade phenol supplied by Sigma Chemical Company
Catalyst	Dibutyltin-dilaurate, supplied by Union Carbide
Organosolv Lignin	Lignin from steam exploded yellow poplar obtained from Bio-regional Energy Associates Ltd., Floyd, VA.
HP-EL Lignin	Lignin from above - propoxylated and ethoxylated, was synthesized and supplied by Brooks Center Dept. of Wood Science and Forest Products at VPI & SU.
400M Print cloth	80 X 80 mercerized and bleached cotton print cloth with a fabric weight of 3.2 oz/sq. yd. supplied by Test Fabrics Inc., New Jersey.

2.2 Characterization of HP-EL

Steam exploded lignin obtained from poplar fibers was reacted with propylene oxide with KOH as a catalyst. This reaction forms the propylene oxide arms on the lignin molecules. After the completion of this reaction ethylene oxide was reacted to partially cap the propylene oxide arms. The resultant product was a dark viscous fluid. The following details were obtained from Brooks Center, the supplier of the material.

Product details

Lignin content = 36.3%

Homopolymer content = 50%

OH number = 230

% OH = 6.97%

Molecular weight \approx 5000 grams/mole

Moles of HP-EL = Weight of HP-EL / 5000

2.3 Methods

2.3.1 Synthesis of blocked-isocyanate

Phenyl N-(3-cyanato-4-methylphenyl) carbamate (I) was synthesized from 2,4 tolylene diisocyanate and phenol. This was a precursor product to lignin carbamate, the end product which was applied to cotton fabric for crosslinking.

Equimolar quantities of phenol and tolylene diisocyanate were reacted at room temperature to cap only the para- or 4- position isocyanate group. A three necked flask with attachments for addition of phenol and for maintaining an inert atmosphere were cleaned thoroughly, oven dried and finally flame dried to eliminate any trace of water. The apparatus used and the reaction are schematically shown in figure 17 and 18 respectively. 60 gm (0.34 moles) of TDI in 25 mL of anhydrous toluene was poured into the three necked flask provided with a magnetic stir bar. 32.42 gm (0.33 moles) of pure grade phenol was dissolved in 25 mL of anhydrous tolylene. The phenol solution was added to it in bulk to the TDI solution which was kept stirred in the flask. Inert atmosphere was maintained in the flask by constant flow of dry nitrogen. The mixture was allowed to react at room temperature for 72 hours, after which a white crystalline solid precipitated from the solution. The product was filtered and recrystallized, by dissolving in hot methylene chloride followed by precipitation from hexane. The product was dried in vacuum and was assured by infrared spectroscopy. Elemental analysis of carbon, hydrogen, oxygen and nitrogen percentages for the molecular formula $C_{15}H_{12}N_2O_3$ confirmed the product.

2.3.2 Synthesis of lignin-isocyanate copolymer

The precursor product (I), a monocapped-aromat, was reacted with the lignin to form a lignin carbamate (IIP-EL PU). The molar ratio of lignin to the blocked isocyanate required to react with the available hydroxyl groups in lignin was determined by conducting a series of HP-EL PU syntheses at room temperature with different molar ratios of IIP-EL to (I). Dibutyltin-dilaurate was used as a catalyst. These reactions were allowed to stir at room temperature for 72 hours. The presence of unreacted isocyanate groups in the reaction mixture was determined by infrared spectroscopy by the presence of the absorption band at 2261 cm^{-1} in figure 18. Beyond a molar ratio of 1:20 (IIP-EL : I) the presence of unreacted isocyanate groups was confirmed by the 2260 cm^{-1} NCO absorption band. Hence for the complete reaction of available -OH groups on lignin by the NCO groups of (I), the molar ratio of 1:20 was chosen.

Both IIP-EL and I were soluble in methylene chloride. 20 gm (0.004 moles) of IIP-EL (molecular weight of $\approx 5000\text{ gm/mole}$) was dissolved in 100 mL of methylene chloride and 21.4 gm (0.08 moles) of (I) was dissolved in 100 mL of methylene chloride separately. Both the solutions were mixed together with two drops of dibutyltin dilaurate acting as a catalyst. The mixture was allowed to react at room temperature for 72 hours. The completion of the reaction was determined by infrared spectroscopy, for the disappearance of the isocyanate band at 2261 cm^{-1} and the appearance of the -NH band at 3300 cm^{-1} as in figure 18.

2.3.3 Determination of hydroxyl content in lignin (HP-EL)

The total hydroxyl content of lignin (HP-EL) being used was determined by the back titration method (Morohoshi, 1986)

A predetermined quantity of dry lignin and vanillin alcohol were weighed into a vial. The above was done in triplicate and two blanks without lignin were prepared. 0.5 mL of dimethylformamide was added to each of the vials to dissolve the contents. Pyridine/acetic anhydride reagent (1:0.3 by volume), about four times the weight of the sample was added to each of the vials and were sealed tight. All the samples were allowed to stand overnight at 50C. Each sample was separately rinsed into a 250 mL beaker with acetone. The cap was also put into the beaker. After the addition of 100 mL of distilled water the beakers were covered with parafilm. The mixture was allowed to stand for one hour.

When opened for titration the parafilm and the sides of the beaker were rinsed into the beaker. The mixture was titrated with 0.1 sodium hydroxide (standardized) to pH 9, using a Fisher Accumet, Model 630 pH meter.

Calculation

The following equation was used to calculate the OH% in the sample.

$$\% \text{ OH} = \frac{[(t/w \times W_s)] - t_s (N \text{ NaOH}) (0.017) (100)}{\text{Sample weight in grams}}$$

t - mL of NaOH for blank

t_s - mL NaOH for sample

w - mg pyridine/acetic anhydride in blank

W_s - mg pyridine/acetic anhydride in sample

Average value of t/w obtained from the blanks was used for sample calculations.

2.3.4 Determination of isocyanate content

The isocyanate content in blocked TDI was measured by a known procedure (Sorenson & Campbell, 1961). A slight modification of the procedure was required to dissolve the blocked isocyanate. Freshly prepared (I) was easily soluble in toluene. (I) stored over a period of time becomes less soluble in toluene. In such cases methylene chloride was used as solvent instead of toluene. A known quantity of (I)(0.5 gm) was transferred into a dry erlenmeyer flask. Methylene chloride (25 mL) was added to dissolve the contents. A solution of 25 mL of 0.1 N butylamine in chlorobenzene was added to the flask and stirred for 15 minutes. The butylamine solution reacts with all the -NCO groups in (I). After the addition of 100 mL of isopropyl alcohol and 4-6 drops of bromophenol blue indicator solution, the excess of butalymine in the mixture was titrated with standardized 0.1 N HCl to a yellow-green end point. A blank test with all the reagents without the sample was carried out.

The percentage isocyanate content was determined by the following equation where the factor 4.202 represents 4.202 gm - NCO per equivalent.

$$\% \text{ NCO} = \frac{(B - V) \times N \times 4.202}{\text{Weight of sample (g)}}$$

B = mL 0.1 HCl required for blank

V = mL 0.1 HCl required for sample

N = Normality of HCl solution

2.3.5 Analysis for nitrogen content

Nitrogen content(%N Kjeldahl) of the lignin-urethane samples was done by Galbraith laboratories. The author determined the nitrogen content (%N Kjeldahl)of cloth samples using the equipment from the Human Nutrition and Foods Laboratory.

2.3.6 Finishing of the fabric

A laboratory scale model (Atlas Laboratory wringer and padder - 12 inches wide, Model LW-1) of a padding mangle was used to pad the finish on the fabric. The fabric was padded with one dip and one nip with a load of 5 lb on the top roller. The wet pick-up percentage was determined from the increase in weight of the fabric after wetting by one dip and nip through the padder. The bath concentrations were calculated from a wet pick- up of 64 % obtained by padding the fabric with a 4% solution of HP-EL PU/methylene chloride solution. Theoretical concentrations of the solutions required for

an intended add-on percentage of 1%, 3% and 6% based on a 64% wet pick-up as shown in Table 1. The 1% theoretical add-on solution resulted in an actual weight gain of 2.2%. The concentration of further solutions were adjusted by either the addition of concentrated solution of HP-EL PU/methylene chloride to increase the concentration of the bath or by addition of methylene chloride alone to dilute the bath. The required concentration of the bath was determined by finishing trial samples and calculating their add-on percentage by weight gain.

Percentage wet-pick up is conventionally done with all components in the padding bath except the resin finish. When this was done the wet pick-up was 84%. This led to the theoretical calculation shown in Table 2.

Although Tables 1 and 2 illustrate how the theoretical bath is formulated, the finishing baths used in this research was empirically adjusted. The solution for an intended add-on of 1%, 3% and 6% approximately contained 7.70g, 11.60g and 23.19g of HP-EL PU respectively, for 500mL of the bath in each case. The actual add-on percentages were 2.2%, 3.1% and 5.7% (on the basis of weight gain) as shown in Table 3.

The samples were passed through the padder and pinned onto pin frames before being cured at 160C for five minutes. The cured samples were marked for dimensional stability and samples were cut out for Kjeldahl analysis to determine the nitrogen content. Other tests described in this chapter were carried out on the finished fabric after being washed five times.

2.3.7 Nitrogen content by Kjeldahl procedure

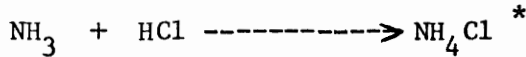
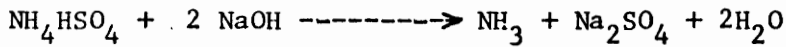
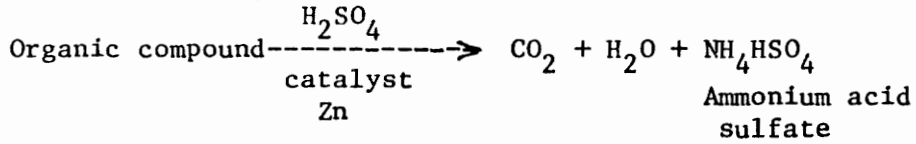
Kjeldahl method is a recognized procedure to measure nitrogen content in a sample. A known weight of the sample was transferred into a digestion tube. About 10 grams of sodium sulfate/cupric sulfate mixture and glass beads were added to each tube. A known volume of concentrated sulfuric acid (25 mL) was poured into each tube. The sample was digested by placing the tube on the block digester heated to 245°C. The samples were digested until a light green color was obtained. The tube was cooled and 225 mL of distilled water is added.

Granular zinc, 1 tsp., was added to each Kjeldahl flask. After the addition of 70-80 mL of 50% sodium hydroxide solution through the insides of the flask the flask was swirled vigorously and more sodium hydroxide was added if the solution did not turn blue in color. The Kjeldahl flask with its contents was connected to the distillation apparatus. The mixture was distilled and the distillate received in a 500mL erlenmeyer flask containing 25 mL of 4% boric acid; 25 mL of water and 4 drops of methyl red-methylene blue indicator. Distillation was done to collect 200 mL.

The distillate was titrated against 0.1N HCl till a pink color was obtained. All samples was titrated until the color matched the end point of the blank.

Nitrogen content

The following set of reactions describe the Kjeldahl digestion procedures for % nitrogen determination.



* One equivalent of HCl reacts quantitatively with one equivalent of N as ammonium borate.

Theoretical calculation:

$$0.1 \text{ eq/L (normality of acid)} \times \frac{14.0 \text{ g of N}}{1 \text{ eq.}} = 1.4 \text{ g N/ L of acid}$$

$$\text{Total N} = (\text{L HCl} - \text{L blank}) \times \frac{1.4 \text{ g N}}{\text{L of acid}} = \text{grams of N}$$

$$\% \text{ N content in fabric} = \% \text{ N in g/g of fabric} = \frac{\text{Total N(g)}}{\text{sample wt.}} \times 100$$

Empirical calculation:

$$\begin{aligned} \text{A-0-1U :-} \quad (\text{L HCl} - \text{L blank}) &= 0.00043 \text{ L} \\ \text{Fabric weight} &= 1.0333 \text{ g} \end{aligned}$$

$$\frac{(0.00043 \text{ L}) (0.1 \text{ eq./L}) (14 \text{ g/eq})}{1.0333} \times 100 = 0.058 \% \text{ N}$$

2.3.8 Sampling of the fabric

A plain woven cotton print cloth designated as 400M by Test Fabrics Inc., was used for the study. The size of the sample was restricted by the width of the padder which was 12 inches wide. With the limitation of the width of the padder, availability of the finish and the fabric the following scheme was drawn. The scheme allowed ten

samples for each group to be tested - unwashed, washed, intended add-on levels of 1%, 3% and 6%. The method is schematically shown in Figure 20. The scheme indicates sampling of one set, and rest of the sets were similar along the length of the fabric. The fabric was 44 inches wide and samples were cut from two columns designated as A and B. The numbering of the samples were as follows: The first number indicates the column from which the sample came from (A or B); the second indicates the percentage of intended add-on (0, 1, 3 or 6); the third number indicates the set (1 to 5) and the last letter indicates the treatment (U - unfinished and unwashed; W - unfinished but washed; S - finished and washed). Hence totally 50 samples were tested.

2.3.9. Dimensional stability

The test for dimensional stability (shrinkage of length and width) was carried out in accordance with the AATCC test method 135-1987. All the samples, were marked for a 10" square, 2" from each edge. About one gram of the finished samples was cut from each piece for Kjeldahl analysis. The remaining pieces were stitched at the seams to prevent fraying of the yarns. All the samples except the unfinished-unwashed samples were washed five times with 90 grams of AATCC standard detergent (for each wash). A top loading Maytag washer was used for washing. Washing was done in normal cycle lasting for 12 minutes with the wash water at $41\pm 3^{\circ}\text{C}$. The size of the load was 1.8 kg and the samples were tumble dried (permanent press setting). The samples were removed from the dryer as soon as the samples were dry and were conditioned for minimum of 6 hours before the dimensional changes were determined as the change in length and width. The conditioned samples were weighed after the third and fifth washes to determine the change in weight.

2.3.10 Durable press rating

The appearance of the unfinished and finished fabrics after five washes were evaluated in accordance with the AATCC test method 124-1984. The washing and drying procedures are described in the section above. Each sample was rated independently by three observers, by comparing the test specimen to AATCC three dimensional durable press replicas. The average rating for each specimen was used for comparisons.

2.3.11 Abrasion resistance (flex)

The resistance of the unfinished and finished samples to flexing and abrasion was determined using the Universal wear tester in accordance with the ASTM test method D 3885-80. All the samples except the unwashed-unfinished samples were washed five times before being tested. A head load of 1 pound and a flex tension weight of 4 pounds were used for testing. The number of cycles required for the samples to fail was used to compare the abrasion resistance of the samples.

2.3.12 Wrinkle recovery of fabric: recovery angle method

The wrinkle recovery of the fabric was determined, using the Monsanto wrinkle recovery tester in accordance with the AATCC test method 66-84. Twenty conditioned samples, 15 by 40 mm, ten with their longer dimension parallel to the warp and ten with their longer dimension parallel to the filling were used for each of the unwashed, washed and finished sets. The specimens were loaded with 500 g for $5\text{min} \pm 5\text{sec}$. and allowed to recover for 5 minutes before the recovery angle was measured. The wrinkle recovery of

the fabric is reported as (W + F) which is the sum of the average recovery angles of the warp and weft.

2.3.13 Tensile strength (Grab method)

Tensile strength in terms of breaking load was determined by ASTM method D 1682 - 64. Samples 6 in. X 4 in. were cut with the longer dimensional parallel to the weft. A line was drawn parallel to the weft, 1 1/2 inches from the edge. A constant rate of extension machine (Instron tensile tension tester) with one inch jaw faces were used. The instrument was calibrated and the samples were tested with the gage length of 3 inches. A 50 kg load cell was used. The speed of the crosshead and the chart were 1.57 inches/min. and 12 in./min. respectively. The breaking load and elongation were calculated from the graph.

2.3.14 Color analysis

The durable press finish synthesized from lignin was brown in color. The presence of the finish on the fabric could be assured visually. The change in the color of the fabric between washes was measured by colorimetry, using model LS-1500 Hunter Lab Scan Spectrocolorimeter in accordance with the ASTM procedure E313-73. L,a,b values which are color values measured in three axes and the yellowness index values were read from the instrument. Ten observations were made for each sample. The overall color change and difference in chromaticity were calculated as follows (Hunter Associates Laboratory, Inc, 1983).

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

$$\Delta C = [(\Delta a)^2 + (\Delta b)^2]^{1/2}$$

where

$$\Delta L = 'L'(sample) - 'L'(standard)$$

(if + L, sample is lighter than standard if - L, sample is darker than standard)

$$\Delta a = 'a'(sample) - 'a'(standard)$$

(if + a, sample is redder than standard if - a, sample is greener than standard)

$$\Delta b = 'b'(sample) - 'b'(standard)$$

(if + b, sample is yellower than standard if - b, sample is bluer than standard)

If there is no color change at all then the ΔE and ΔC values will be close to zero. In the industry, standards are set to determine the acceptable levels of color change. In the present study low values (below 5) of ΔE and ΔC will be considered acceptable. The above color change level was derived after examining the methodology and results reported by Galbraith et al. (1986). On the assumption that the change in color values will indicate loss of finish from the fabric, these values can be used to evaluate the stability of the finish to washing.

The differences in color between unwashed samples, samples washed 3 and 5 times were determined.

3.0 Results and discussion

3.1 Synthesis of blocked isocyanate

To determine a procedure of synthesizing the blocked isocyanate the reaction of tolylene diisocyanate (TDI) and phenol were done in carbon tetra chloride, dimethylformamide and toluene. Reaction temperatures of 20C and 60C were tried. Since it was understood that higher temperatures reduce the difference in the reactivities of the two isocyanate groups (Sodoun et al., 1987) reactions at room temperature were favored. The reactions carried out using the different solvents yielded the product. The product obtained by reaction done in toluene and the reaction done without any solvent seemed to give better results in terms of yield and purity of the product. The product yield ranged from 80-95% and the melting point of the product ranged from 95-100C. If there was an excess of the solvent it took longer for the product to form which can be explained by the fact that the molecules are too far apart in the solvent to react quickly. Absolute elimination of water was essential for the success of the reaction and this was achieved by oven drying the glassware and later flame drying them before the reaction was started. Dropwise addition or bulk addition of phenol to TDI did not seem to make any difference. If a slight excess of phenol was used then the reaction tended to form a dicapped product with occlusion of TDI within the product. Recrystallization

was done by dissolution in methylene chloride and precipitating in hexane or ether in which the monocapped product is insoluble.

Isocyanate content in the blocked isocyanate was determined by the method described earlier and was found to be 14.6% which is close to the theoretical value of 15.66%. Percentage of nitrogen was analyzed by Galbraith laboratories and was estimated to be 10.34% (theoretical value is 10.43%). The theoretical and the practical values are close, and the differences are assumed due to the analytical procedures used. The infrared spectrum of the blocked isocyanate shown in figure 21 clearly indicate the broad -NH stretching vibration at 3273 cm^{-1} and the -NCO absorption at 2272 cm^{-1} . Hence it is assured that one of the NCO groups is capped by phenol. The yield of the product ranged from 80% - 90%. The melting point of the monocapped product was 95-100C.

3.2 Synthesis of lignin-isocyanate copolymer

The modification reaction of lignin using propylene oxide was described earlier. Chain extension of the propyl arm was carried out by reacting propylene oxide with IPL and later reacted with ethylene oxide to cap the propoxyl arm (Brooks Center, 1990). The product thus obtained is designated as HP-EL and used in this study. This modification forms primary -OH groups for reaction with the blocked isocyanate.

The hydroxyl content determinations on HP-EL lignin by back titration method showed 6% of hydroxyl content. The molecular weight of HP-EL was assumed to be 5000. The assumption was made on the basis that HP-EL comprised of twentyfive C9

units with each unit having a molar weight of 200g/mole. Since a molar ratio of 1:20 is being used for the reaction there would be twenty molecules of the blocked isocyanate attached per molecule of IIP-EL. Hence the molecular weight of the lignin-urethane product would be $[5000 + (268 \times 20)] = 10,360$ g/mole.

The main idea of this study was to chemically link the lignin molecule to the cellulose molecule. Blocked isocyanate was the linking element. As shown in the following table the reaction of IIP-EL with blocked isocyanate was studied by doing a series of reactions with varying molar ratios of HP-EL to blocked isocyanate.

Molar ratio HP-EL : (I)	Weight of HP-EL	Weight of (I)
1 : 15	1.0 g (0.0002 mole)	0.804 g (0.003 mole)
1 : 20	1.0 g (")	1.072 g (0.004 ")
1 : 25	1.0 g (")	1.340 g (0.005 ")
1 : 30	1.0 g (")	1.608 g (0.006 ")
1 : 35	1.0 g (")	1.876 g (0.007 ")
1 : 40	1.0 g (")	2.144 g (0.008 ")

In figure 18, the infrared spectrum of molar ratio of 1:20 shows complete reaction of the hydroxyl groups in lignin with (I).

The reaction was done in methylene chloride and dibutyltin dilaurate used as a catalyst at room temperature for 72 hours. The completion of the reaction was deter-

mined by infrared spectroscopy. A portion of the mixture was rotovaporated and infrared scans were taken on the product. Infrared scans of products obtained from reactions of HP-EL with the blocked isocyanate at different molar ratios clearly indicate the reduction of -OH groups in HP-EL. The infrared spectrum of the product obtained from the reaction of HP-EL with the blocked isocyanate with a molar ratio of 1:20 is shown in figure 22. The reduction of the -OH band at 3400 cm^{-1} - 3500 cm^{-1} ; the presence of -NH band from 3287 cm^{-1} - 3318 cm^{-1} ; and the presence of C=O carbonyl band at 1738 cm^{-1} - 1805 cm^{-1} confirm the reaction of the blocked isocyanate with the lignin. The characteristic peaks of lignin at 2800 cm^{-1} - 2950 cm^{-1} can also be observed. Beyond the molar ratio of 1:20 the 2272 cm^{-1} band was pronounced indicating the presence of unreacted isocyanate groups. It can be concluded that beyond the ratio of 1:20 not all the blocked isocyanate in the mixture reacts with the lignin.

Blocked isocyanate and the products obtained from reactions using different molar ratios were sent to Galbraith laboratories for nitrogen content analysis. Table 4 shows the results obtained. From figure 23 it can be observed that there is no appreciable difference between the theoretical and actual nitrogen content in the products.

3.3 Finishing of the fabric

The fabric was padded with the finish using a laboratory model of a padding mangle. Limitations of its size influenced the sampling method and width the finish using a laboratory model of a padding mangle. Since methylene chloride was used as a solvent drying was done at room temperature by evaporating the methylene chloride. Initial problems of the migration of the solvent and nonuniform coating were solved by

quicken the process of passing the fabric through the padder and drying the fabric in air without contact with any surface. It was noticed that contact while the fabric was drying caused nonuniform patches on the surface.

Evaporation of the solvent from the fabric made it difficult to determine the wet pick up. However, a pre-weighed piece of fabric was padded with a 4% HP-EL PU/methylene chloride solution and quickly transferred to a glass container with a lid. The wet pick-up percentage was determined by the increase in weight of the fabric. It was found to be 64%. The first batch of samples which were finished had a percentage add-on of 2.2 percent and not 1.0 as was intended. The concentrations of the second and third bath were adjusted by trial and error and the percentage gain in weight of finished fabrics were within reasonable limits. The padding bath was kept closed when not being used to minimize the evaporation of the solvent from the bath. The add-on percentages were close to the calculated add-on percentages and the coating was observed to be uniform to the naked eye. As shown in Table 5, calculation of add-on percentages after five washes resulted in add-on percentages of 2.24%, 3.90%, 7.50%. Though the first two add-on percentages are close to the add-on values calculated before washing, there seem to be some anomaly in the third. Percentage add-on calculations from the nitrogen content in the fabric as shown in Table 6 is in good agreement with the add-on values obtained by weight gain. However, since add-on percentage generally refers to the increase in weight due to the finish, the add-on percentages calculated before washing have been used hereafter. Table 7 summarizes the different add-on values which were calculated on the basis of weight gain and nitrogen content in the fabric.

Metal frames were designed to simulate industrial curing techniques where curing is done on pin frames to hold the fabric flat. The samples were mounted on the frames and cured for 5 minutes at 160C. Migration of the solvent with the finish due to han-

dling prevented mounting the fabric when it was wet. Moreover unlike water, methylene chloride does not affect the hydrogen bonds in cotton, and thus has minimal influence on the properties of the fabric. A strong odor of phenol indicated the decapping of the finish. All the samples were analyzed with the colorimeter to determine the color change by the change in the L, a, b values and Yellowness Index (YI).

About one gram of the unwashed samples were cut out for Kjeldahl analysis. After that the seams were stitched the samples were washed according to the AATCC test method 5 times. The weight change and color change after the third and the fifth washes were determined. Though a slight change in the yellowness was visually noticeable the weight change was not considerable.

To establish the chemical cross linking of the lignin to cellulose a sample of the finished fabric, washed five times was extracted with methylene chloride in a soxhlet apparatus for 24 hours. There was no color or weight change of the sample. Since the finish is soluble in the solvent it was expected that if the finish was not chemically linked it would wash away with the solvent. The solvent used was absolutely colorless proving that no finish was removed the fabric. As shown in figure 24 the specular reflectance infrared spectra (Cardamone, 1989) of the fabric before and after extraction with methylene chloride indicated no difference.

3.4 Fabric testing

The durable press ratings were done according to AATCC Test Method 124-1984. In all the three levels of actual add-on an increase of DP rating (after five washes) was

observed as compared to the unfinished fabric but ratings were below 3.0 which is the minimum acceptable standard in the industry.

The color parameters L, a, b and YI (Yellowness Index), were statistically analyzed. Tables 8, 9 and 10 compare the L, a, b and Yellowness index values for finished samples with 2.2 %, 3.1% and 5.7% add-ons respectively. Paired comparisons were made between the samples unwashed, after 3 washings and after 5 washings. The first column indicates the three letter coding used. The first letter denotes the value being compared - L, a, b or YI. The second letter denotes the number of washings U, 3 or 5. The last letter denotes the add-on percentage - 2.2%, 3.1% or 5.7%. The second column indicates the mean value obtained from the ten readings taken for each sample. Paired comparisons between means were done using student's 't' test. The *** marks in the third column indicate statistically significant difference between the pair at the 95 % level.

Figure 25 shows the changes in the L, a, b values between washes in all the three levels of add-ons. From the figure it can be seen that the 'L' value indicating the lightness of the fabric is progressively decreasing (in other words the fabric is becoming darker progressively) as the add-on percentage increases. Similarly 'a' and 'b' values which are color indicators increase progressively. It can be observed that there is no significance difference in the lightness values between washes in all the three cases. Though 'a' and 'b' values appear to be unchanged, they were statistically significant and was reflected in the Yellowness index values. This may be an indication of possible change in the lignin chromophoric structure or slight finish loss not detected by weight change but detected by %N analysis.

Table 11 details the overall or total color difference (ΔE) and chromaticity difference (ΔC). L, a and b values are three elements measured at three different axes by the instrument. (ΔE) is calculated using all the three values and indicates the overall color difference. The chromaticity difference is calculated using the 'a' and 'b' values. From the figure 26 it can be noticed that the maximum overall color change after 5 washes is at the 3.1% level add-on. There is a greater overall color change after 5 wash/dry cycles as compared to 3 wash/dry cycles. the greatest color change is at the 3.1% add-on level.

The nitrogen content per gram of finished fabric for the three levels of add-on is listed in Table 6. Since Galbraith Laboratories reported a 1.4% relative standard deviation in their %N analysis of the synthesized IIP-EL PU, this was used as a correction factor in subsequent computations. From figure 27 it can be seen that the increase in nitrogen content is consistent with the increase in the add-on percentage as was expected. The increasing nitrogen content assures increase in the crosslinks formed. By %N Kjeldahl analysis there is an indication of finish loss after five wash/dry cycles. Finish loss was computed to be 18.6%, 6.3% and 11.4% at respective add-ons of 2.1%, 3.7% and 4.9%. This indicates that more wash/dry cycles should be performed to determine finish durability. Theoretical calculations as shown below Table 6 for determining the quantity of finish in the fabric from the nitrogen content gives a value approximately equal to the add-on percentage calculated by weight gain, except the last add-on level.

Finished and unfinished fabrics were tested for dimensional stability, durable press performance, tensile strength, abrasion resistance (flex), recovery angle (W + F). The mean values obtained are shown in Table 12. All the means were statistically analyzed for significant difference at 95 % level. Table 13 lists the results of the statistical

analysis. The *** marks indicate significant difference between the pair being compared.

The change in durable press ratings are shown in figure 28. It can be noticed that the lignin-urethane finish does contribute towards the increase in durable press performance of the fabric. Direct comparisons to DMDHEU finish cannot be done since lignin finish was applied from an organic solvent medium and not water as it is normally done. More work needs to be done to find if the incorporation of a different kind of lignin would increase the durable press rating significantly. As shown in figure 29 the highest tensile strength is observed at the 2.2% level of add on. The abrasion resistance as shown in figure 30 indicate a significant improvement at add-on levels of 3.1% and 5.7%. The recovery angle (W + F) do not show any improvement due to the finish at any level. There is significant reduction of elongation % at all levels of add-on indicating an increase in brittleness of the fabric due to crosslinking. No correlation was observed between the three levels of add-on for any property. Chemical crosslinking of cotton by this method definitely indicates the possibility of lignin being used as a durable press finish for cotton. But more work needs to be done in this area to increase the DP rating and the angle of recovery.

Two results which attract interest is that there is no significant drop in the tensile strength and abrasion resistance. Generally when the hydroxyl groups in cellulose are modified a drop in strength and abrasion resistance would be expected. This result indicates the possibility of utilizing this approach in areas other than durable press finishing, for instance manufacturing composites with cellulose or industrial fabrics where retention of abrasion resistance and tensile strength would be required under severe conditions.

From the results it was evident that the finish did increase the durable press rating. However the recovery angle (W + F) did not improve considerably. As expected, as the percentage add-on increased the color of the fabric was darker and an increase in stiffness was also observed. In spite of the modification of the hydroxyl groups in cellulose very little loss was observed in terms of strength and abrasion resistance. These results were surprising since considerable loss in physical properties were expected at least in the 5.7% add-on samples. It is possible that the loss in strength due to the chemical modification was compensated for by the strength imparted by the presence of lignin. The retention of strength in spite of the finishing treatment and exposure to heat opens new avenues for its use.

Finish loss was detected through %N analysis but was not detectable from change in weight. The change in color values indicate possible changes in the chromophoric structure of lignin.

4.0 Conclusions

Cellulose and lignin can be crosslinked by using a lignin- isocyanate copolymer. Comparisons with formaldehyde based resins used for durable press finishing of cotton fabrics was not possible due to insolubility of the finish in water. However, the possibility of using lignin as a potential material for durable press finishing of cotton fabrics was confirmed. The following facts summarize the outcome of this research:

Increase in durable press rating as compared to unfinished fabric confirms the possibility of using lignin copolymer as a potential durable press finishing agent.

The stability of the finish on the the crosslinked fabric to repeated washing and extraction with methylene chloride, reflects excellent adhesion of lignin to cellulose.

Retention of abrasion resistance and tensile strength opens new avenues of application.

Tailoring the characteristics of lignin by varying the length of the propylene oxide arm provides ample opportunity to improve the recovery angle.

Using methylene chloride as a medium of application of the finish makes this study incomplete in terms of evaluating the finish in comparison with other finishes presently used. Therefore, the following studies can be proposed to evaluate the lignin- isocyanate copolymer.

Sulfonation of the lignin-isocyanate copolymer to make it soluble in water and evaluating its behaviour when applied from a water medium.

Evaluation of the effect of the length of the propylene oxide arms on lignin towards the increase in the crease recovery property of crosslinked cotton.

Investigation of the possibility of making composites with materials to which cellulose has poor affinity (like polyethylene).

Investigation of using less toxic materials as alternatives to TDI and phenol to use lignin in a similar fashion to crosslink cotton.

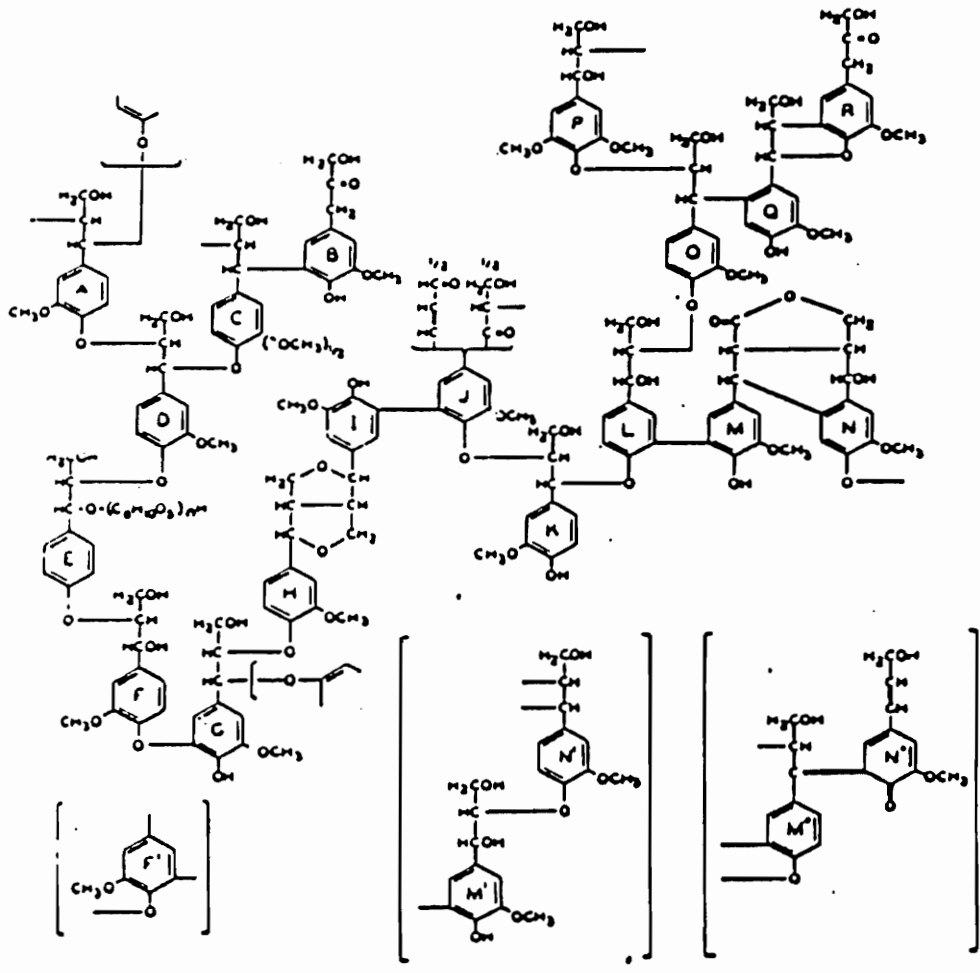


Figure 1
 Freudenburg's model of lignin
 (Demaret, 1986)

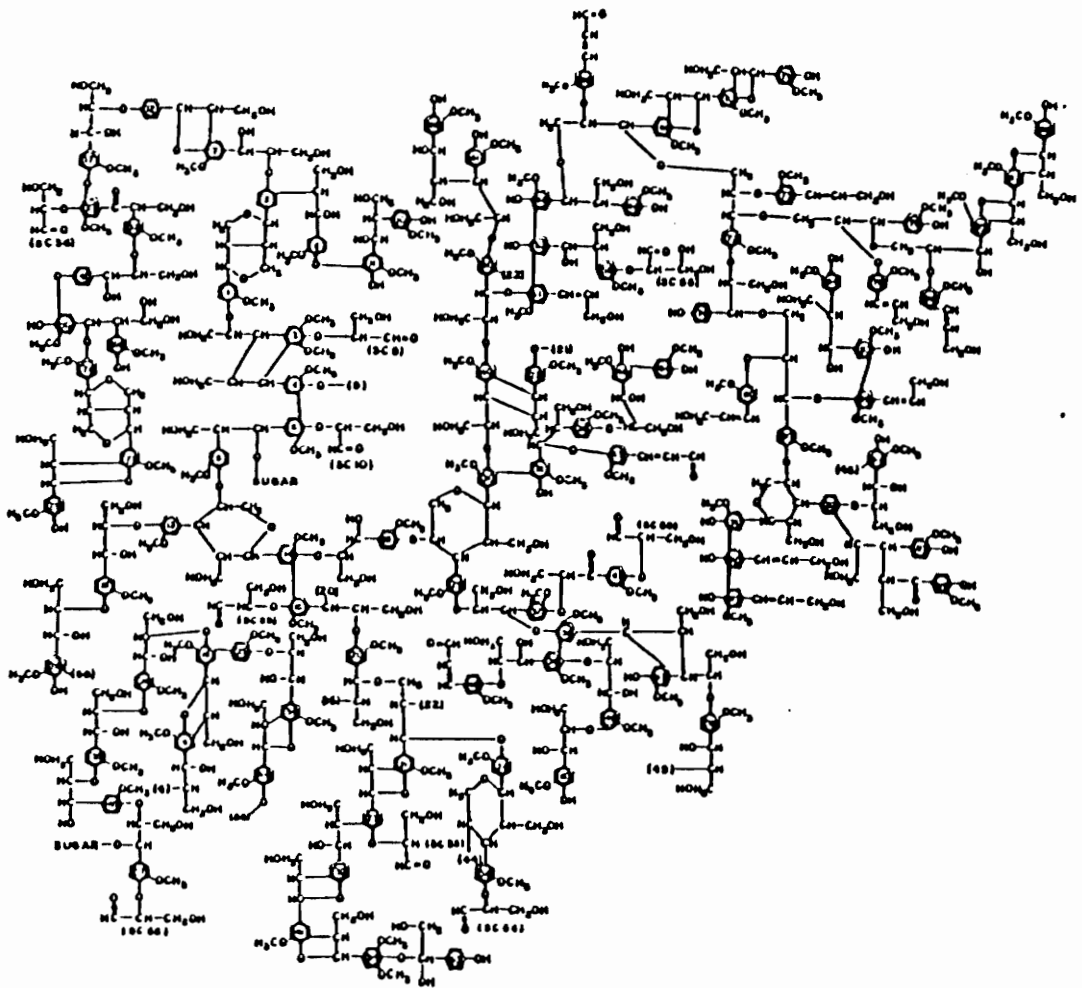


Figure 2

Glasser's model of lignin
(Demaret, 1986)

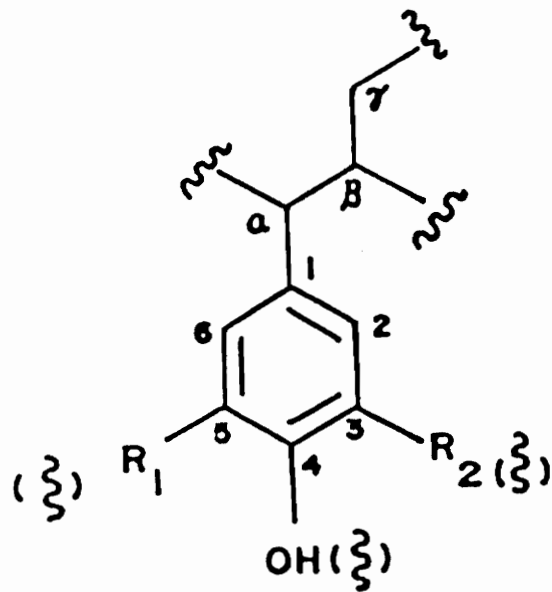
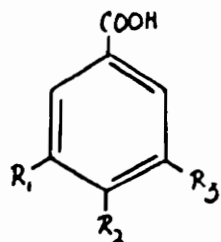
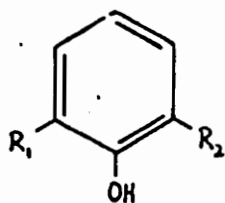


Figure 3

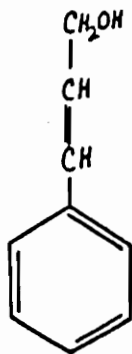
Phenylpropane building unit
of lignin



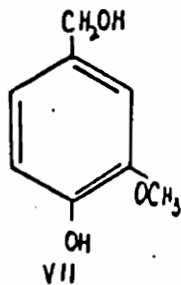
I; II; III; IV



V; VI



VIII



VII

I	$R_1 = R_2 = R_3 = H$	Benzoic acid
II	$R_1 = R_3 = OCH_3; R_2 = OH$	Syringic acid
III	$R_1 = OCH_3; R_2 = OH; R_3 = H$	Vanillic acid
IV	$R_1 = R_3 = H; R_2 = OH$	p-Hydroxybenzoic acid
V	$R_1 = R_2 = OCH_3$	2,6 Dimethoxyphenol
VI	$R_1 = OCH_3; R_2 = H$	Guaiacol
VII		Vanillyl alcohol
VIII		Cinnamyl alcohol

Figure 4

Model compounds of lignin
(Wu & Glasser, 1984)

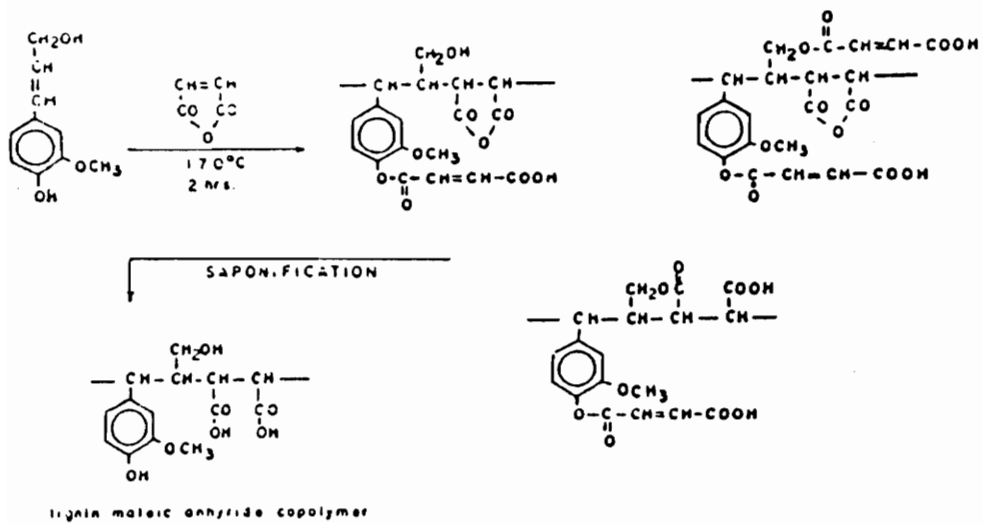


Figure 5 :
Reaction of lignin with maleic anhydride

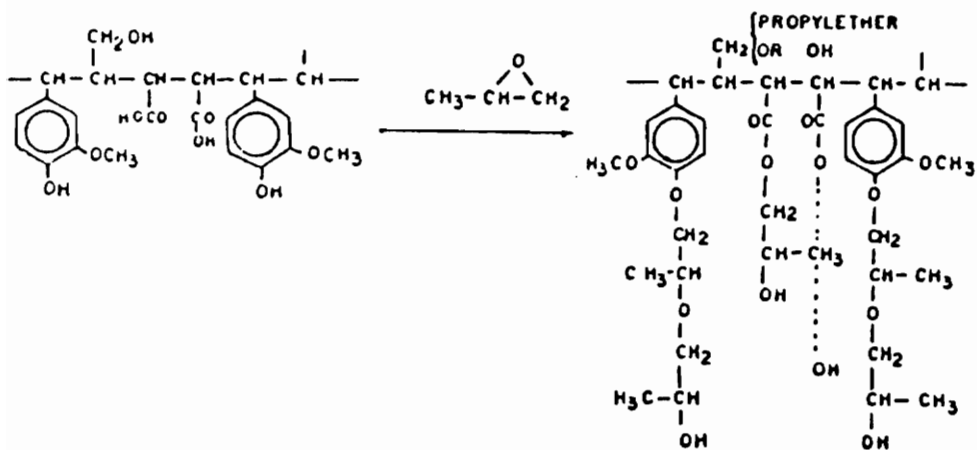


Figure 6
Reaction of carboxylated lignin with Propylene
oxide (Hsu & Glasser, 1975)

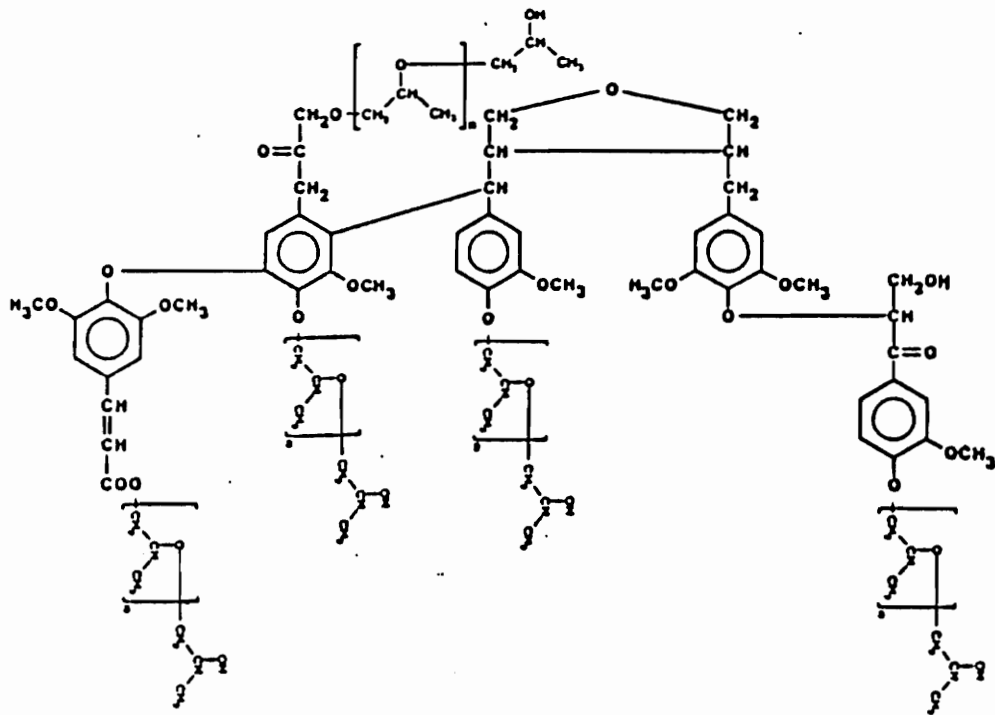


Figure 7

Schematic representation of HPL
(Demaret, 1986)

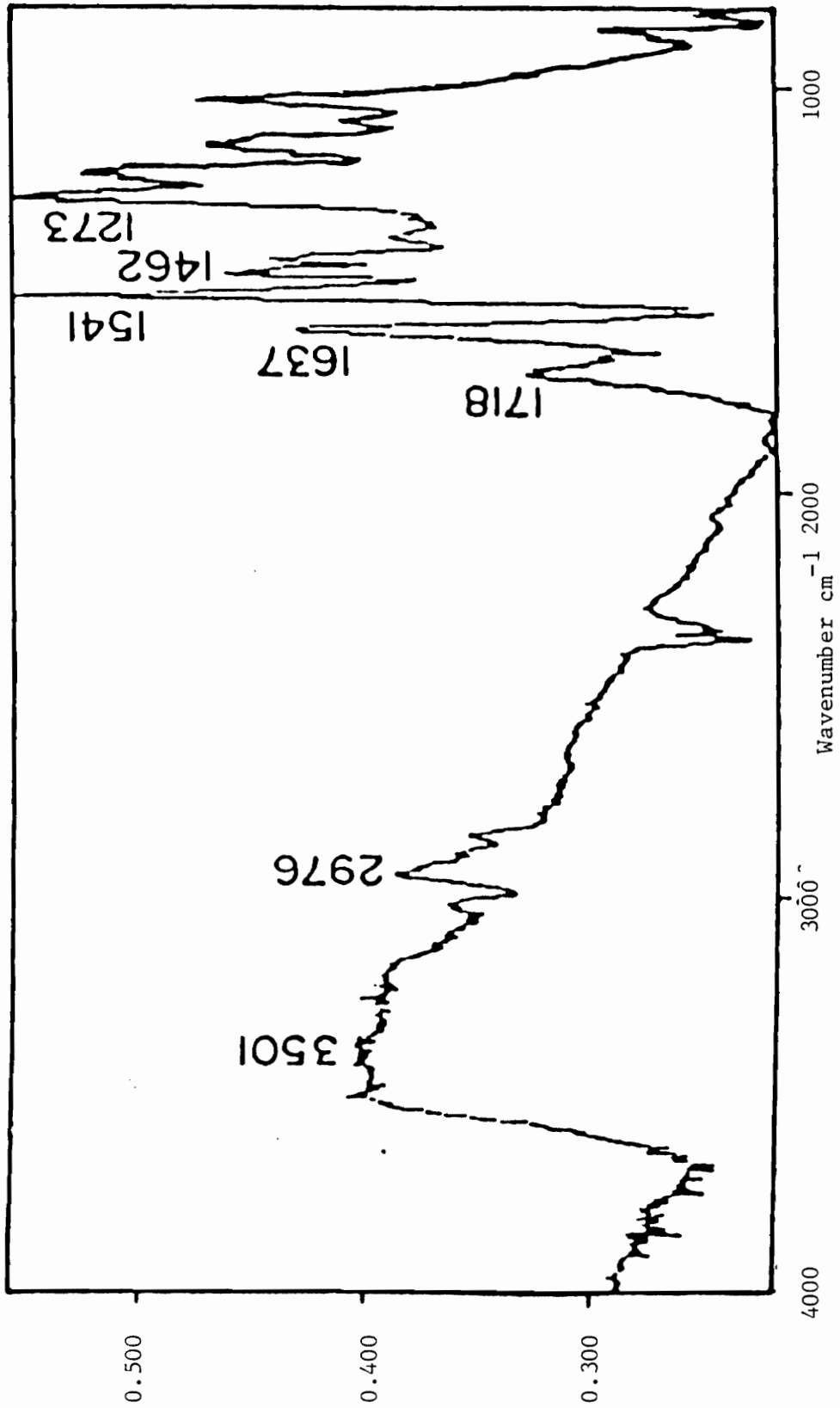


Figure 8
Infrared spectrum of kraft lignin (Cardamone, 1990)

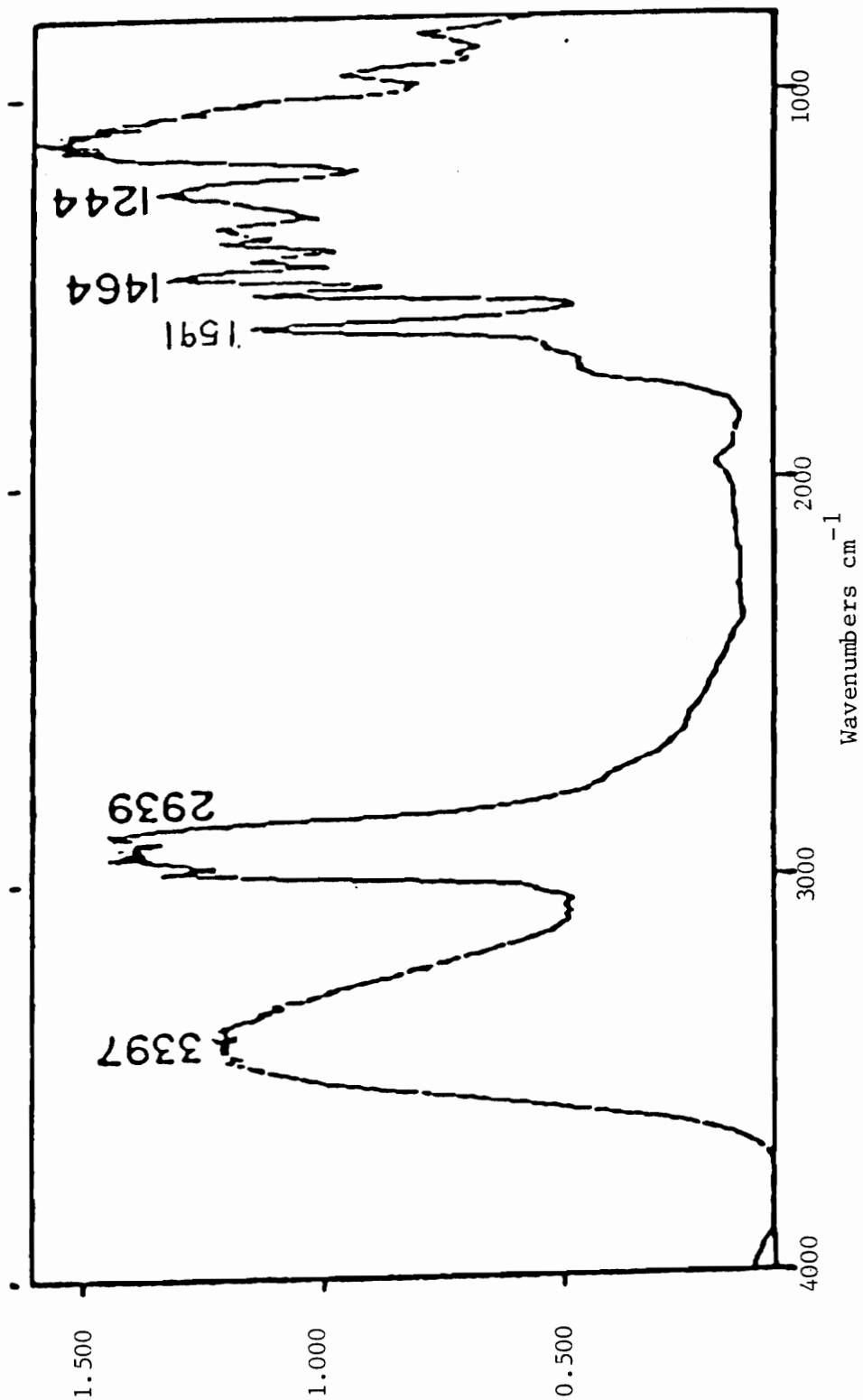


Figure 9
Infrared spectrum of HP-EL (Cardamone, 1990)

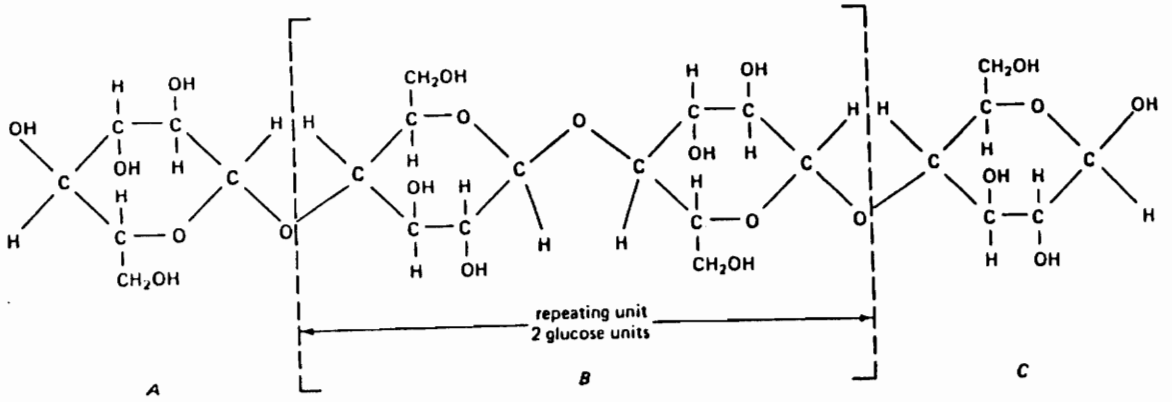


Figure 10
Conventional structure of cellulose

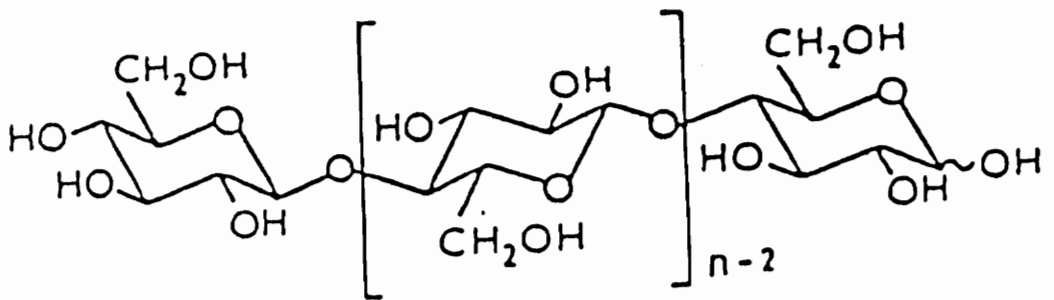


Figure 11
Chair conformation of cellulose

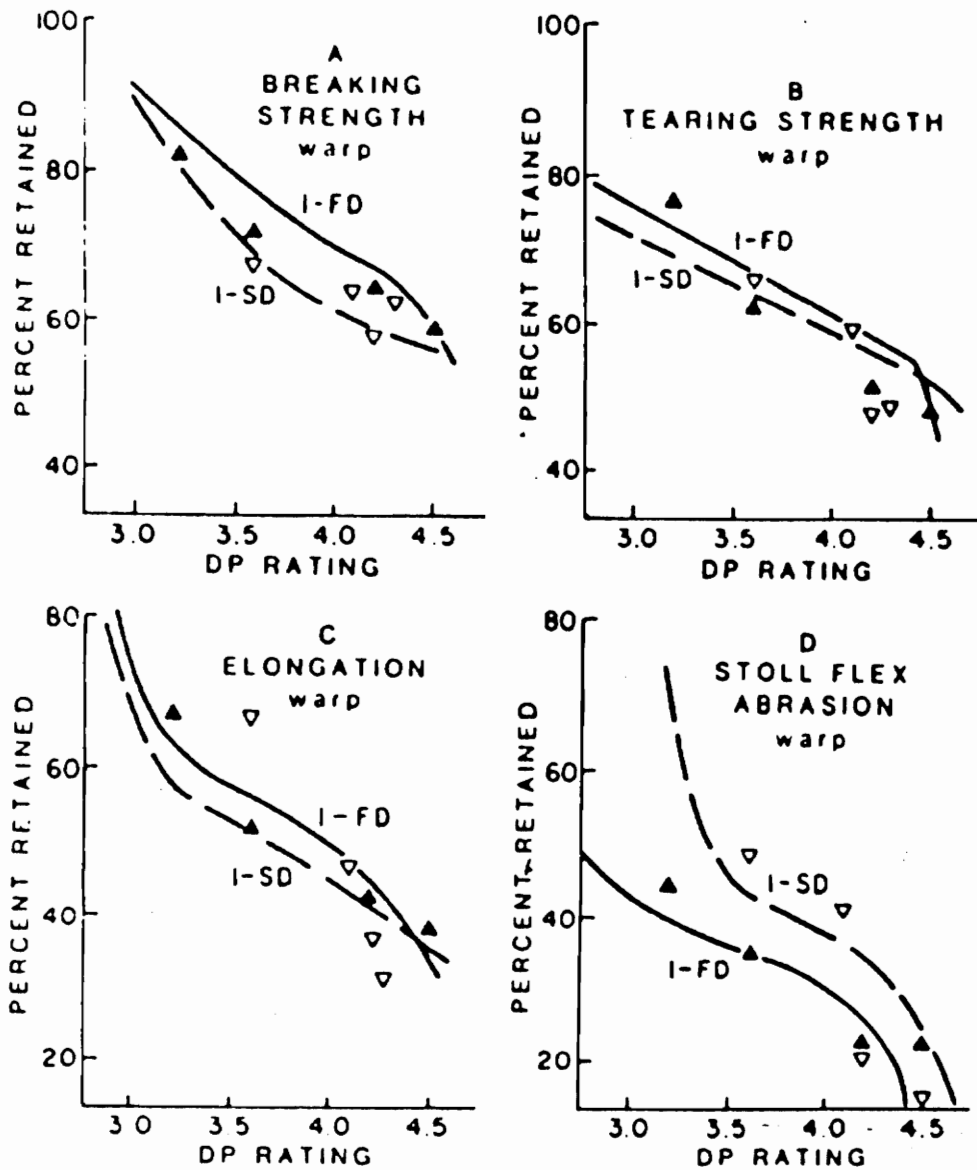


Figure 12

Effects of DMDHEU finishing on the physical properties of cotton (Rowland et al., 1983)

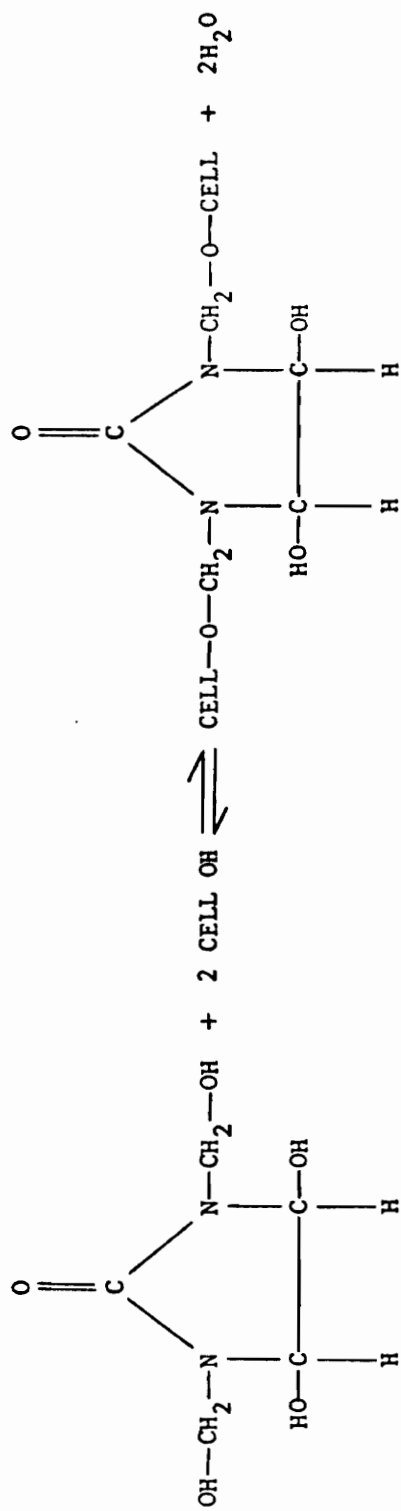
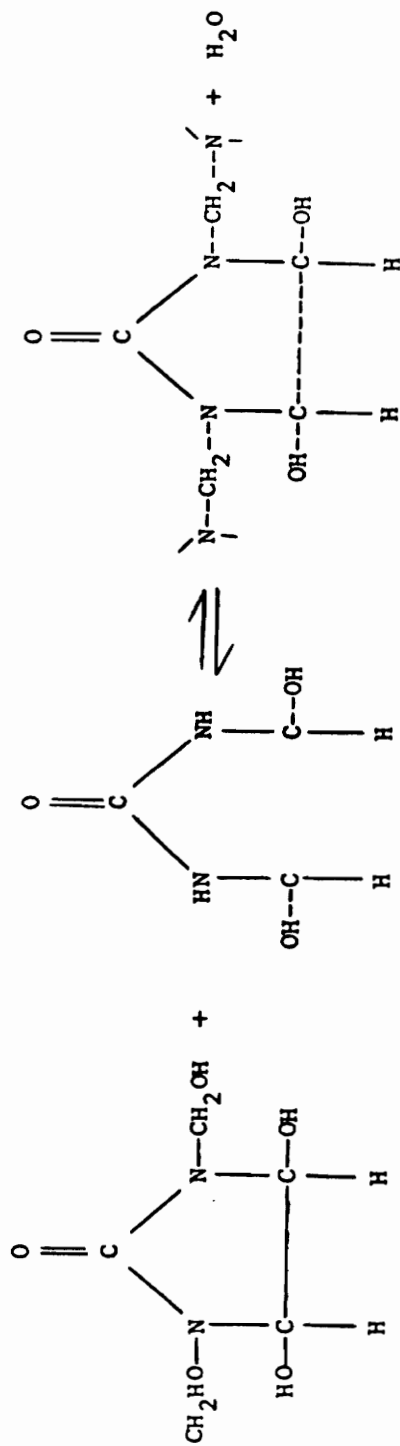


Figure 13

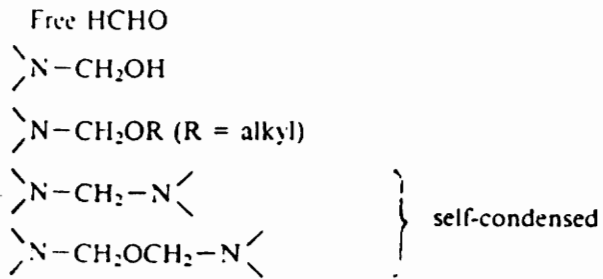
Crosslinking of cellulose with DMDHEU



DMDHEU
Self condensation

Figure 14
(Hilden, 1986)

Resin formulation



Resin-treated fabric

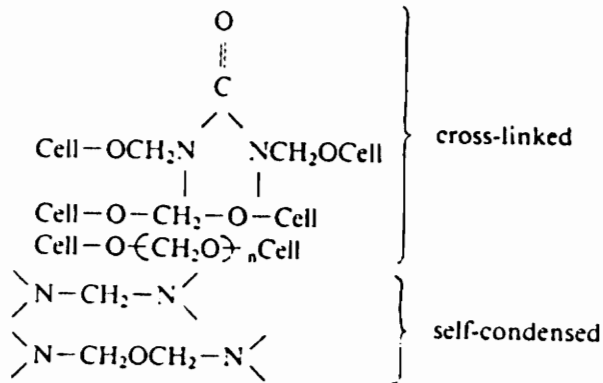
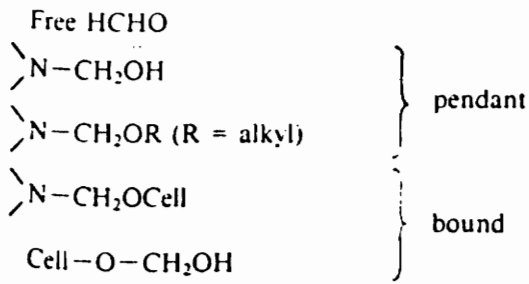


Figure 15

Forms of Formaldehyde present in a formaldehyde finished fabric
 (Kim & Smith, 1985)

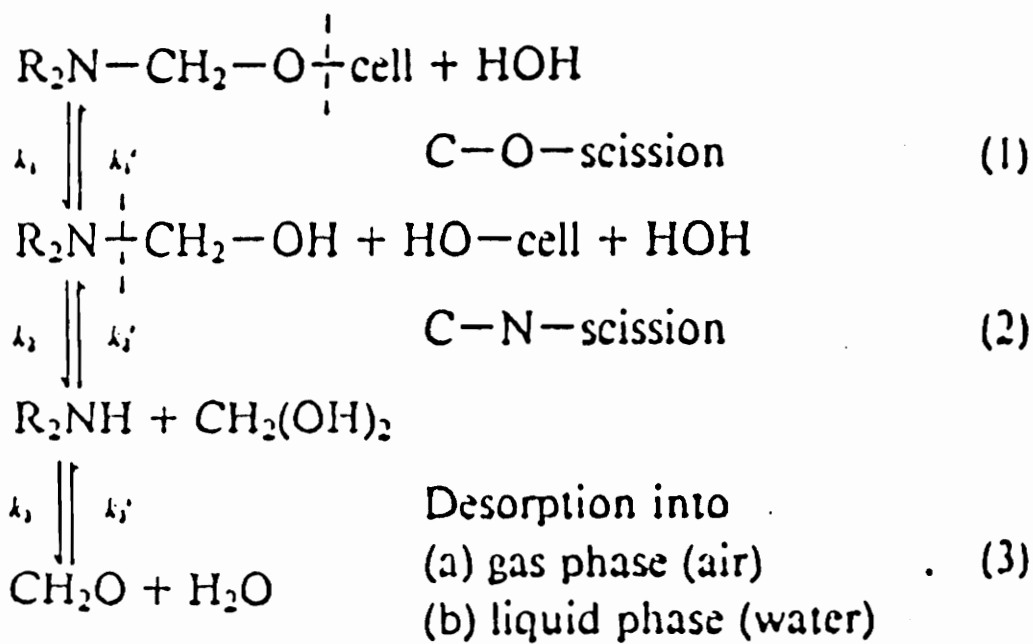


Figure 16

Release of Formaldehyde (Kamath et al., 1985)

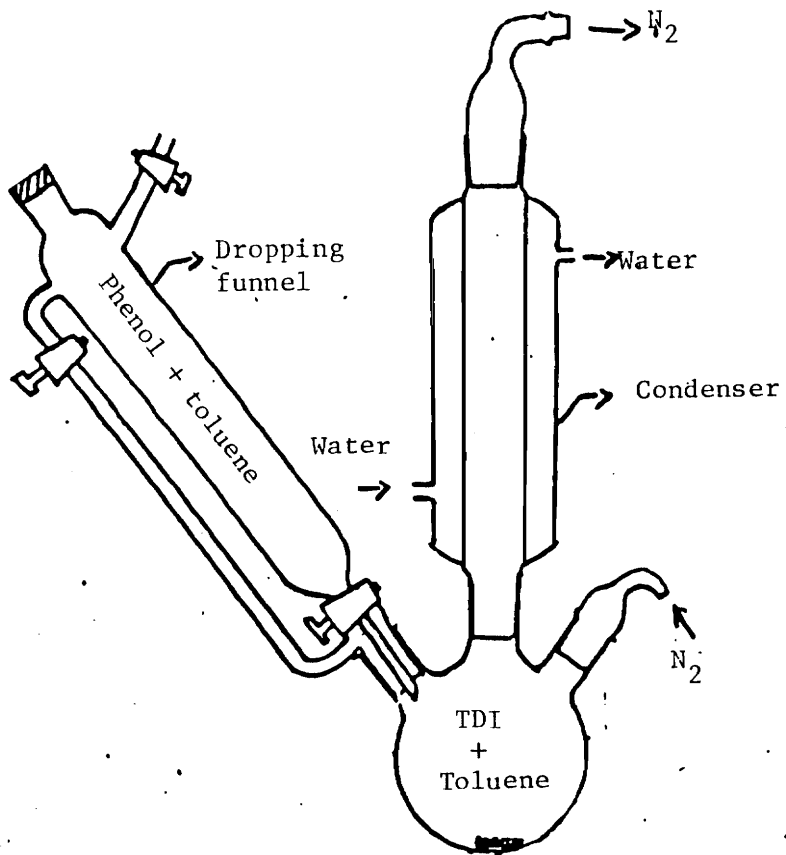


Figure 17

Reaction apparatus

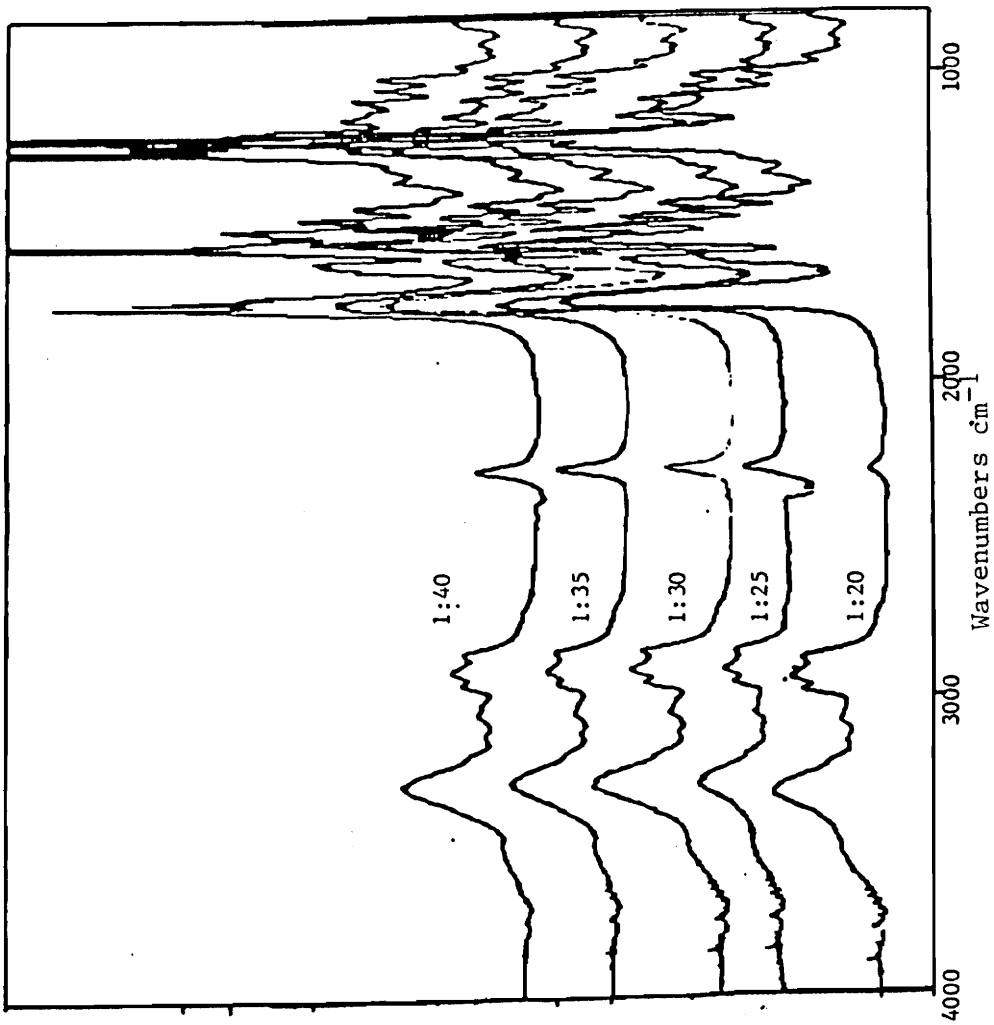


Figure 18
Infrared spectra of HP-EL PU at different molar ratios of
HP-EL to (I)

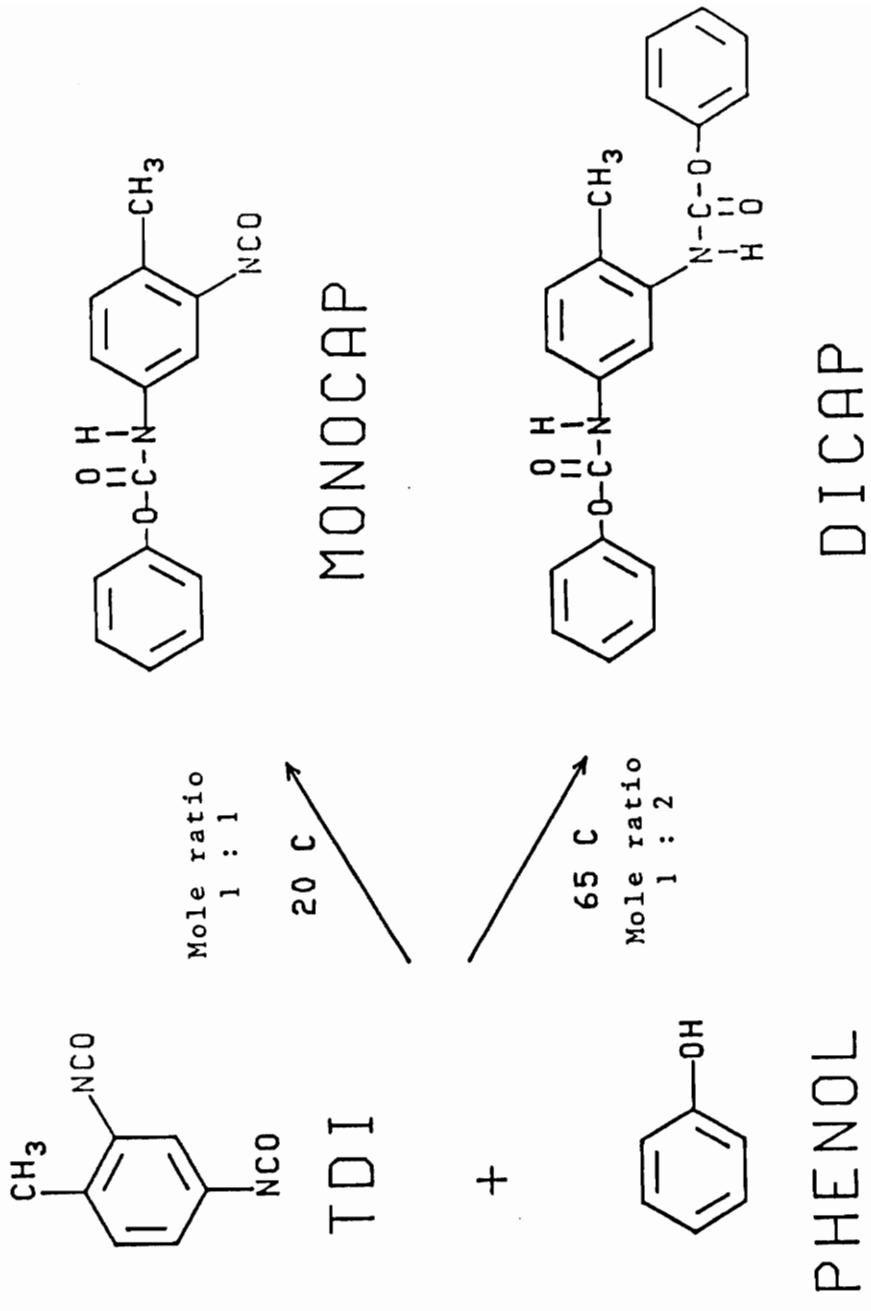
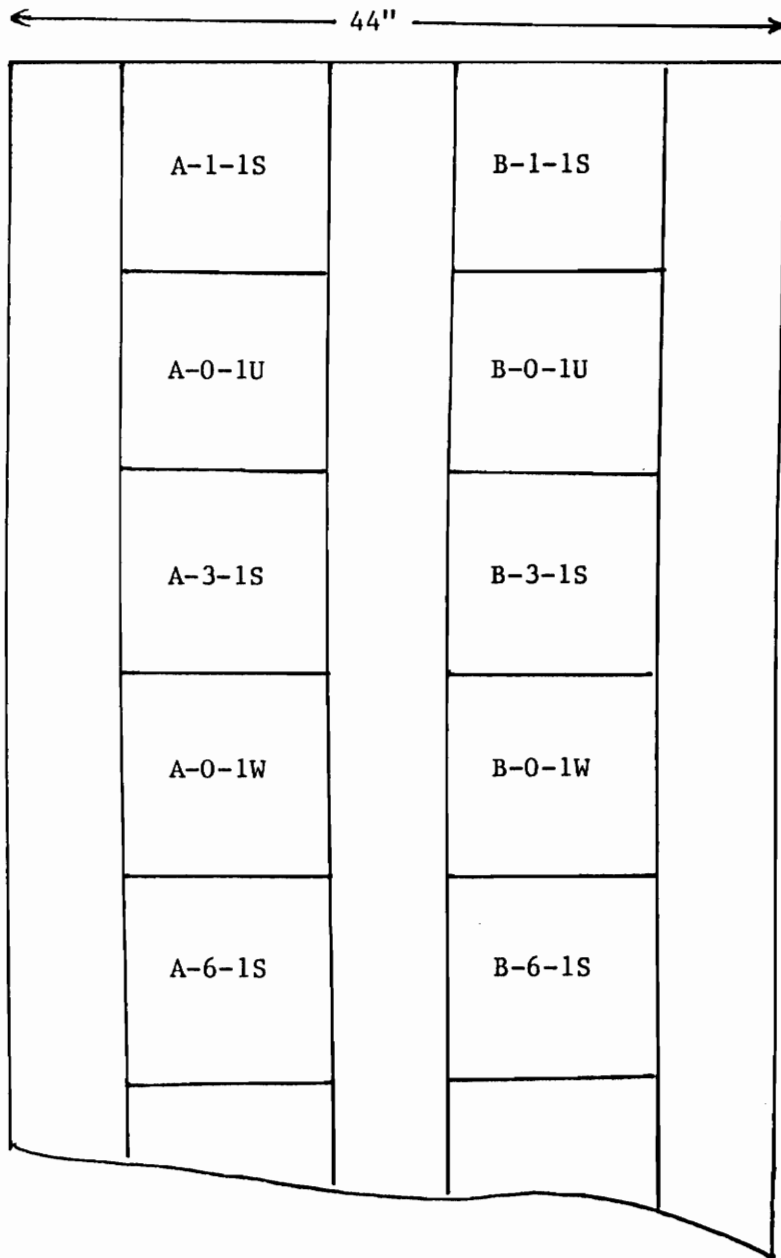


Figure 19
Reaction of phenol with TDI



A - 0 - 1U

→ U or W or S indicating unfinished-unwashed, unfinished-washed and finished-washed samples respectively

→ 1 to 5 indicating the set the sample is taken

→ 0, 1, 3 or 6 indicating the percentage add-on (intended)

→ A or B indicating the column the sample is taken

Figure 20

Schematic representation of the sampling method
(Total of 5 sets; only one is represented above)

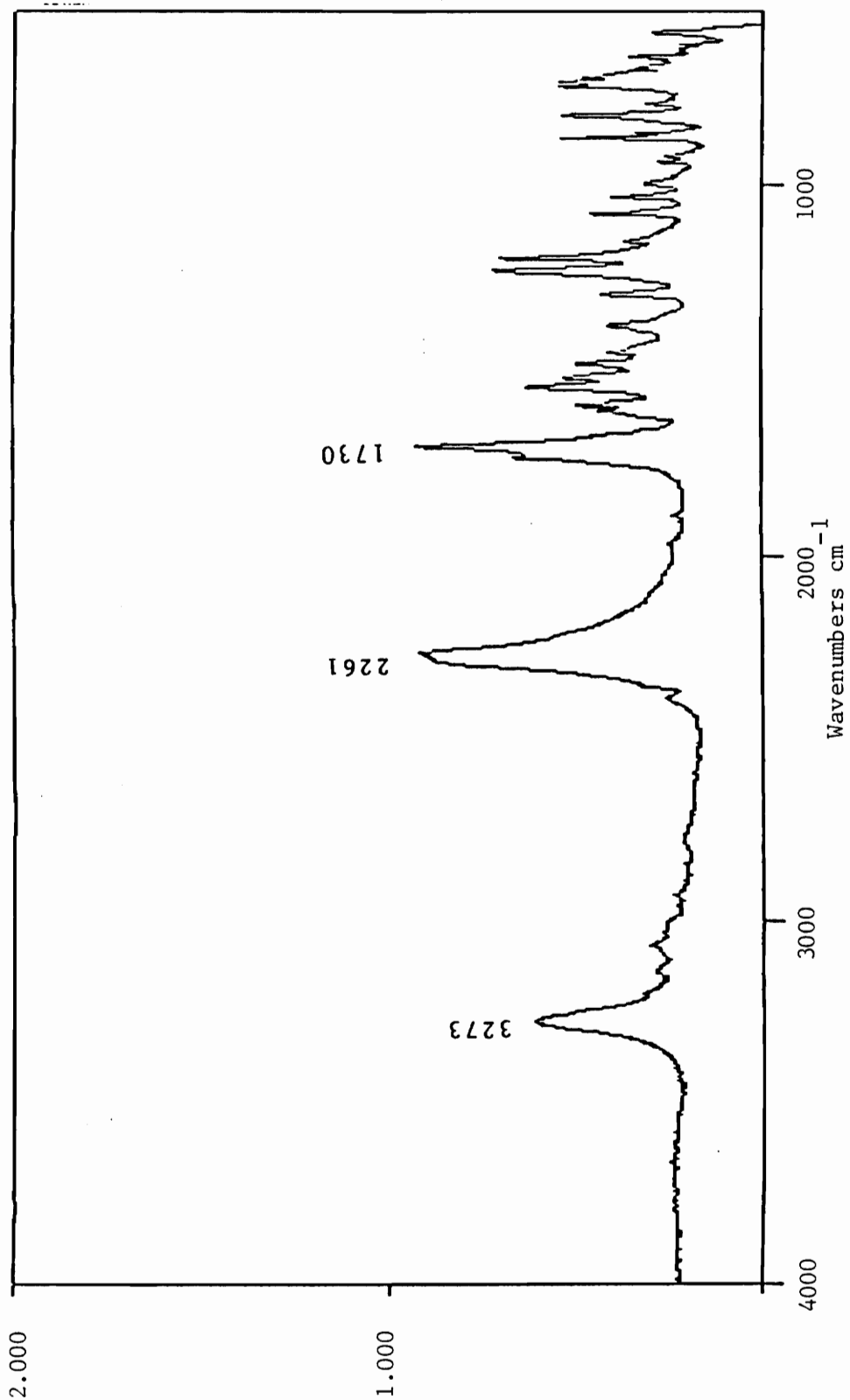


Figure 21

Infrared spectrum of blocked isocyanate (I)

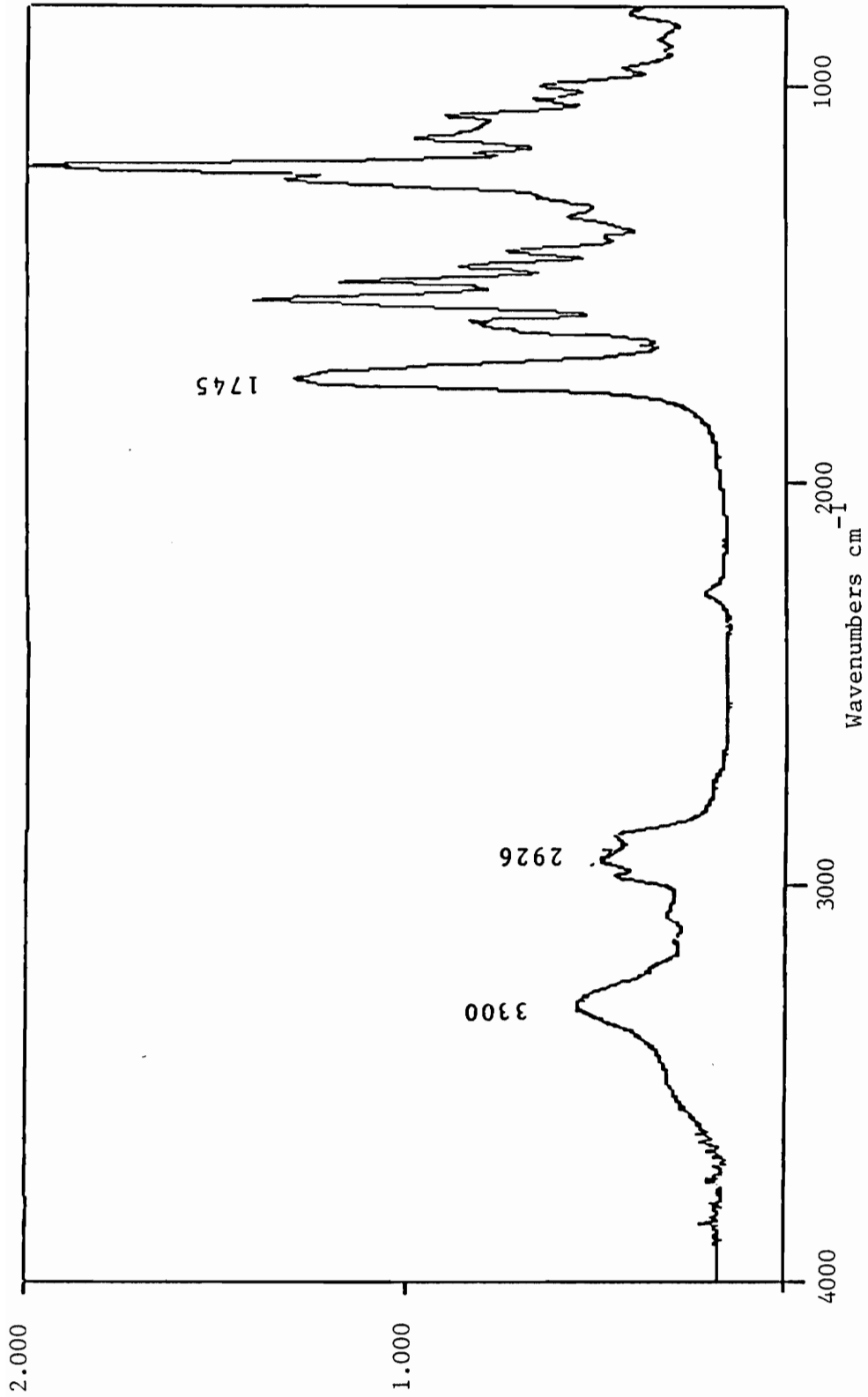


Figure 22
Infrared spectrum of lignin-isocyanate (HP-EL PU)

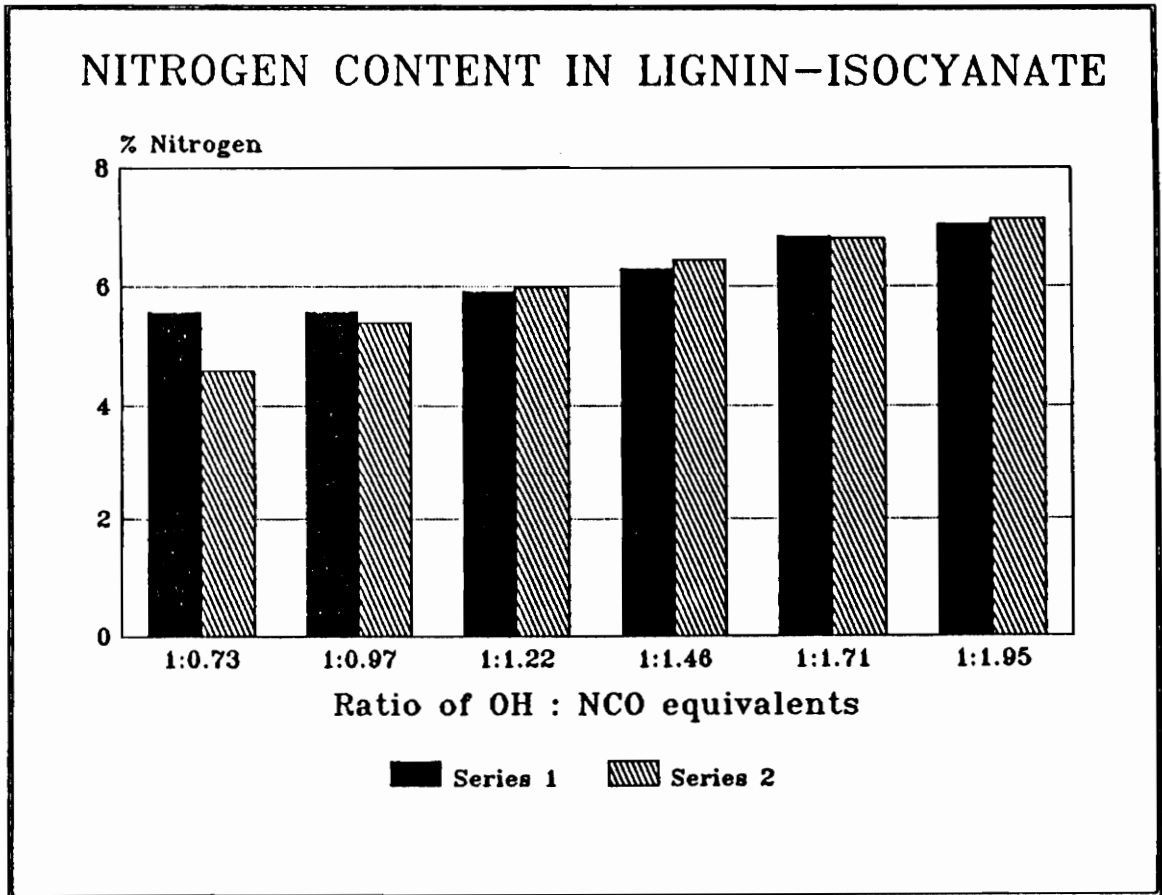


Figure 23

Graphical representation of nitrogen content in HP-EL PU copolymer

Series 1 : Actual nitrogen content

Series 2 : Theoretical nitrogen content

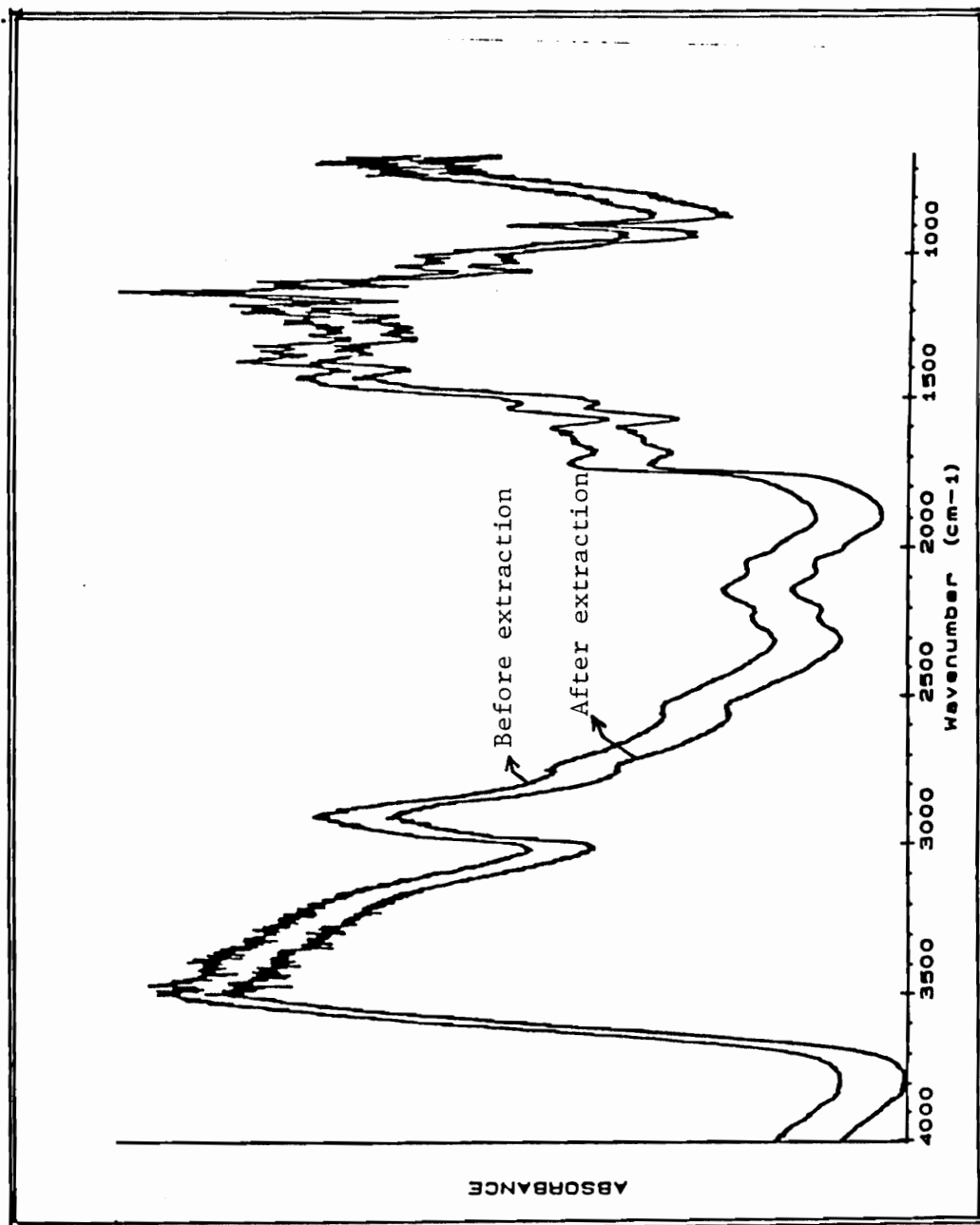


Figure 24
Infrared spectrum of finished fabric

COMPARISON OF L,a,b VALUES (Hunter colorimeter)

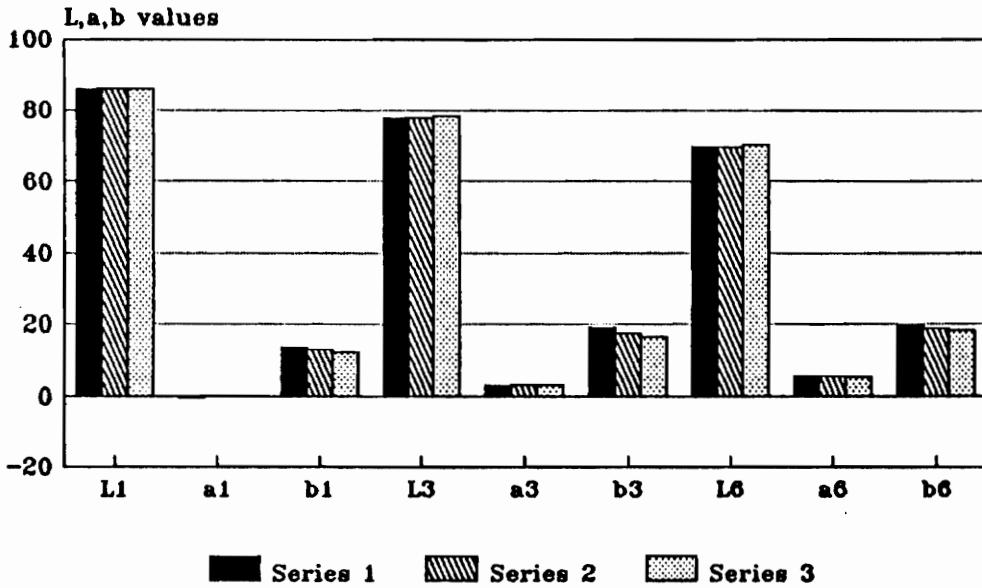


Figure 25

Graphical representation of change in color values

All readings were taken on finished fabric

L1, a1, b1 - values for 2.2% add-on

L3, a3, b3 - values for 3.1% add-on

L6, a6, b6 - values for 5.7% add-on

Series 1 - Unwashed (add-on's 2.2%, 3.1%, 5.7%)

Series 2 - Washed 3 times (" ")

Series 3 - Washed 5 times (" ")

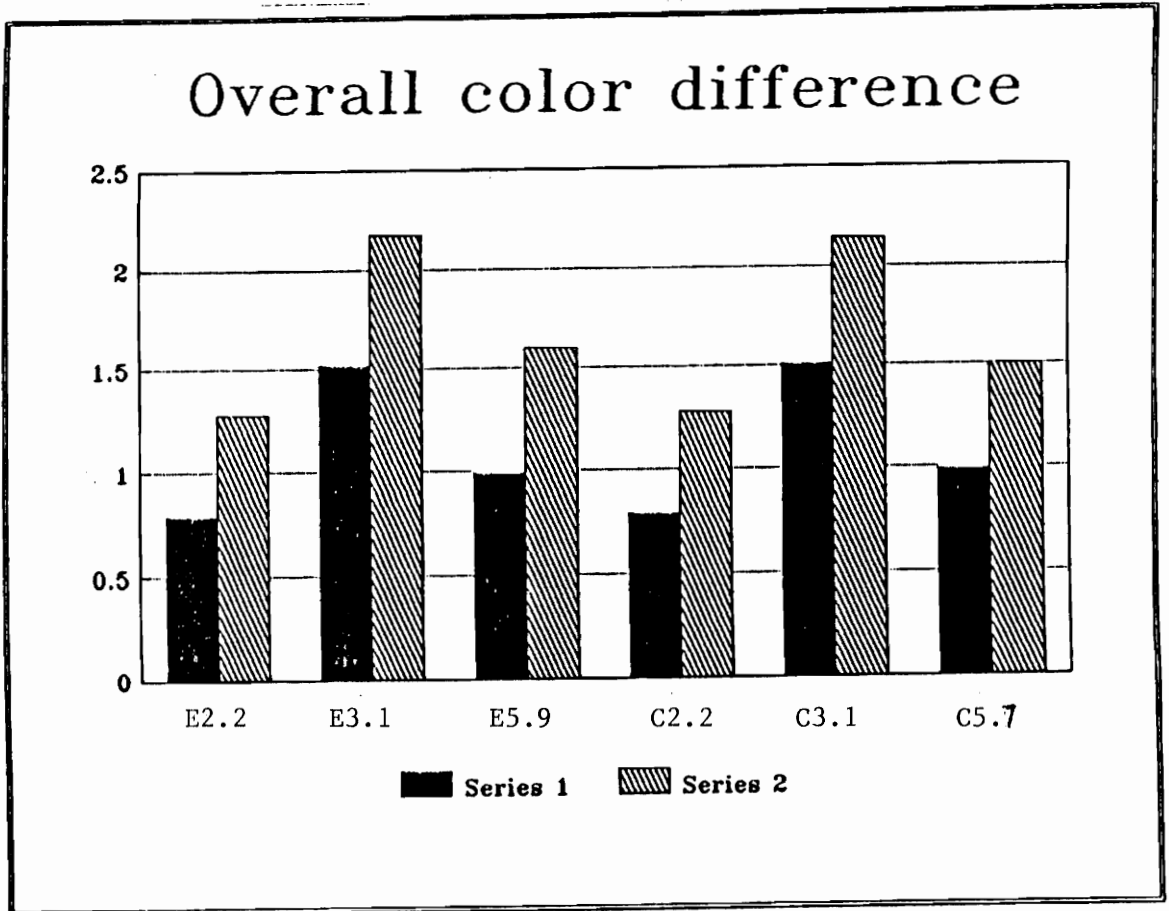


Figure 26

Graphical representation of overall change in color

Series 1 - Compares unwashed fabric with fabric washed 3 times

Series 2 - Compares unwashed fabric with fabric washed 5 times

E2.2 - E values of fabric with 2.2% add-on

E3.1 - E value of fabric with 3.1% add-on

E5.9 - E value of fabric with 5.7% add-on

C2.2 - C value of fabric with 2.2% add-on

C3.1 - C value of fabric with 3.1% add-on

C5.9 - C value of fabric with 5.7% add-on

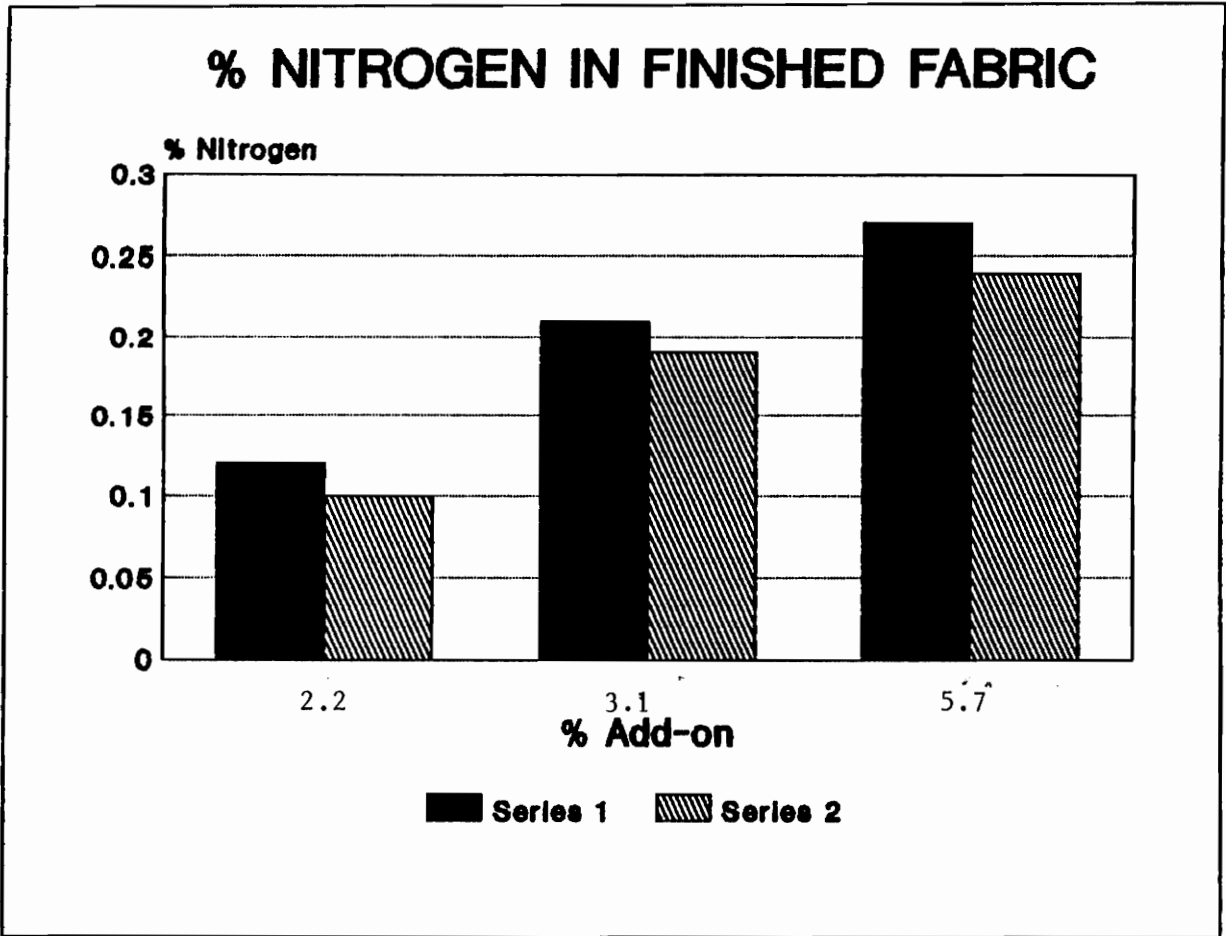


Figure 27

Nitrogen content in the finished fabric

Series 1 - Nitrogen content before washing

Series 2 - Nitrogen content after 5 wash/dry cycles

DURABLE PRESS RATING

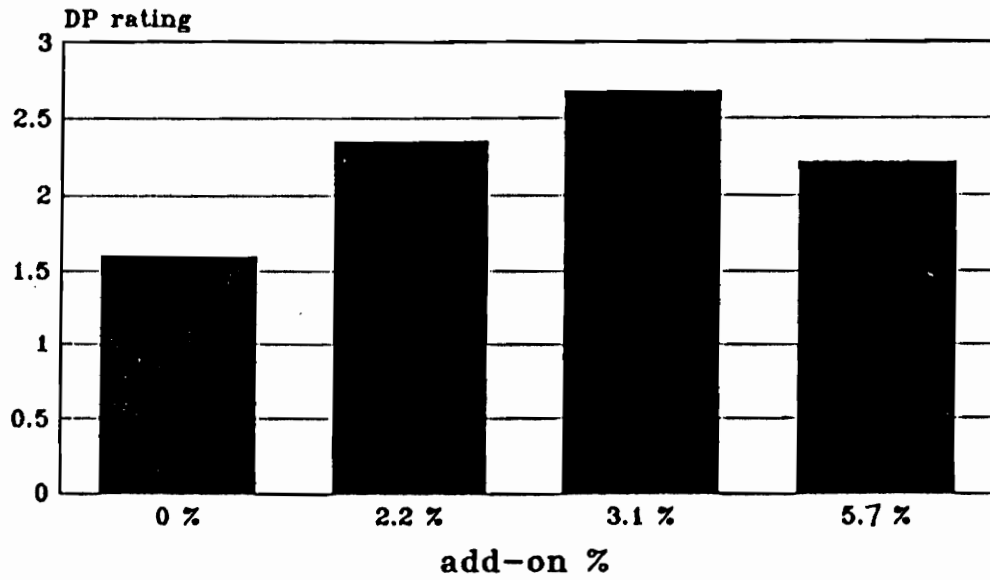


Figure 28

Graphical representation of change in DP rating

DP rating was done after the samples were washed & dried 5 times

TENSILE STRENGTH (in kilograms)

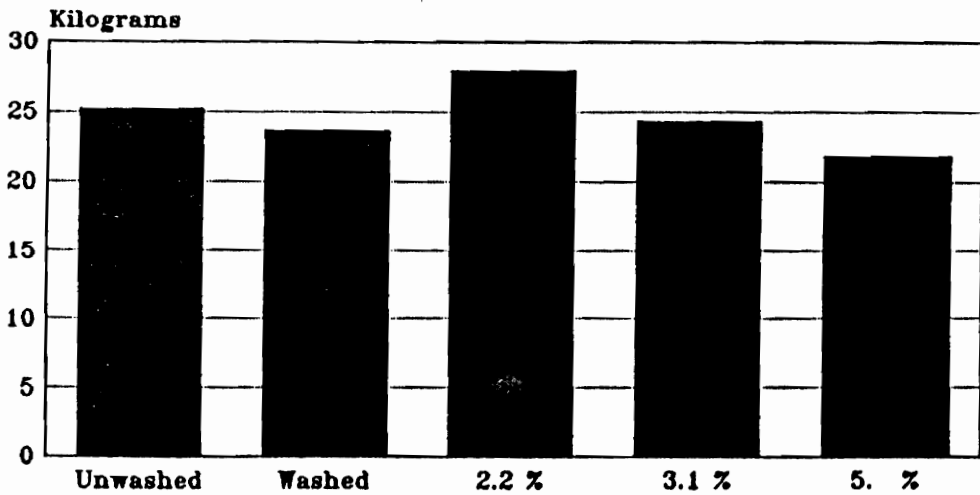


Figure 29

Graphical representation of change in tensile strength

Tensile testing was done after the samples were washed & dried
5 times

ABRASION RESISTANCE (FLEX) (Number of cycles)

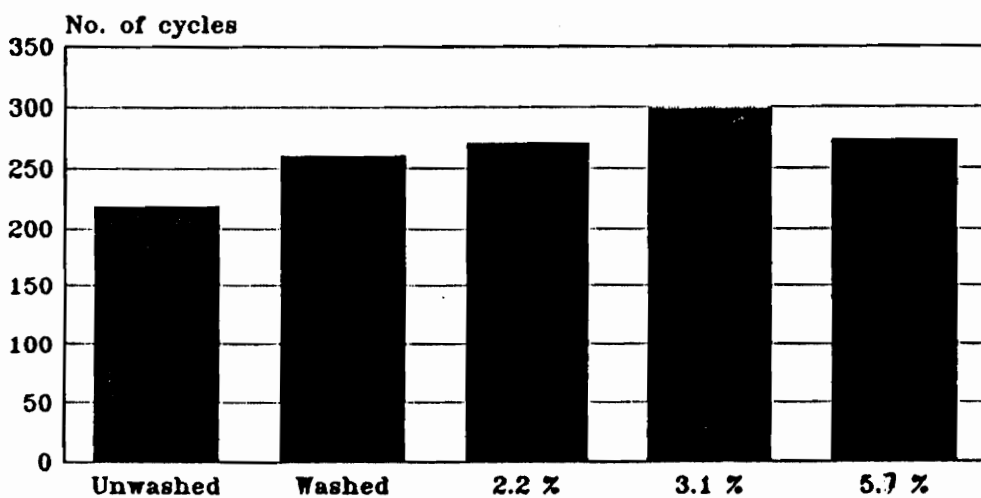


Figure 30

Graphical representation of change in abrasion
resistance

Samples were tested after being washed & dried 5 times

Table 1

Bath composition with 64% wet pick-up

Intended add-on based on 84% wet pick-up	1%		3%		6%	
(Fabric weight)						
125.15 g for 1%						
125.71 g for 3%						
127.01 g for 6%						
	%	Amount	%	Amount	%	Amount
Methylene chloride	98.43	123.2mL	95.31	119.8mL	90.58	115.0mL
HP-EL PU	1.57	1.96g	4.69	5.92g	9.42	11.96g
Wt. of HP-EL PU for 500mL of padding solution		7.95g		24.70g		52.00g

Table 2

Bath composition with 84% wet pick-up

Intended add-on based on 84% wet pick-up	1%		3%		6%	
(Fabric weight)						
125.15 g for 1%						
125.71 g for 3%						
127.01 g for 6%						
	%	Amount	%	Amount	%	Amount
Methylene chloride	98.81	123.7mL	96.43	121.2mL	92.86	117.9mL
HP-EL PU	1.19	1.49g	3.57	4.49g	7.14	9.07g
Wt. of HP-EL PU for 500mL of padding solution		6.02g		18.50g		38.50g

Table 3

Fabric weight gain after HP-EL finishing and actual add-on before
5 wash and dry cycles

Sample	Initial fabric wt.(g)	Fabric wt. gain before wash	Actual add-on%	Intended add-on%
A-1-1S to B-1-5S	12.52	12.79	2.2%	1%
A-3-1S to B-3-5S	12.57	12.96	3.1%	3%
A-6-1S to B-6-5S	12.70	13.43	5.7%	6%

Table 4

Nitrogen content in lignin-isocyanate copolymer

Ratio of moles of lignin to blocked isocyanate (I)	Ratio of equival. of -OH to -NCO	Actual Nitrogen %	Theoretical Nitrogen %
1 : 15	1 : 0.73	5.58 %	4.60 %
1 : 20	1 : 0.97	5.58 %	5.40 %
1 : 25	1 : 1.22	5.91 %	5.98 %
1 : 30	1 : 1.46	6.30 %	6.44 %
1 : 35	1 : 1.71	6.84 %	6.81 %
1 : 40	1 : 1.95	7.04 %	7.12 %

Molecular weight of HP-EL lignin = 5000
 Molecular wt. of blocked isocyanate = 268
 OH number = 230
 -OH % in HP-EL lignin = 6.97 %
 -NCO % in blocked isocyanate = 15.67 %

$$\begin{aligned} \# \text{ of OH groups per mole of HP-EL} &= \frac{6.97 \times 5000 \text{ g/m (lignin)}}{100 \times 17 \text{ g/m (OH)}} = 20.5 \end{aligned}$$

$$\begin{aligned} \# \text{ of -NCO groups per mole of (I)} &= \frac{15.67 \times 268 \text{ g/m (I)}}{100 \times 42 \text{ g/m (NCO)}} = 1.0 \end{aligned}$$

% Actual nitrogen content was determined by Galbraith Lab.

% Theoretical nitrogen content was calculated as follows:

$$\% \text{ N} = \frac{(28 \times n)}{5000 + (268 \times n)} \times 100$$

where n is the number of blocked isocyanate moles per mole of lignin. eg. when n=20,
 % N = 5.40 %

Table 5

Fabric weight change after HP-EL finishing and subsequent 5 wash and dry cycles

Sample	Finished fabric wt.(g) before wash**	Finished fabric wt.(g) after wash	% weight change	Adjusted add-on%
A-1-1S to B-1-5S	10.79	10.83	+0.04	2.2+0.04=2.24%
A-3-1S to B-3-5S	11.23	11.32	+0.80	3.1+0.80=3.90%
A-6-1S to B-6-5S	11.53	11.74	+1.80	5.7+1.80=7.50%

** This fabric weight is less than the weight given in Table 3 because a piece of each fabric was cut for % nitrogen by Kjeldahl analysis

Table 6

Nitrogen content in finished fabric

Intended % add-on	% Nitrogen before wash	% Add-on before wash	% Nitrogen after wash	% Add-on after wash
1%	0.118 ± 0.002	2.1	0.096 ± 0.001	1.70
3%	0.206 ± 0.003	3.7	0.193 ± 0.003	3.45
6%	0.272 ± 0.004	4.9	0.241 ± 0.003	4.30

Percentage of nitrogen in HP-EL PU = 5.58%

Percentage add-on from nitrogen content:

$$\% \text{ add-on} = \frac{\% \text{ nitrogen in fabric}}{\% \text{ nitrogen in HP-EL PU}} \times 100$$

Sample calculation (for 1% intended add-on):

$$\% \text{ Add-on} = \frac{0.118}{5.58} \times 100 = 2.1\%$$

%Finish Loss:

$$\frac{0.118 - 0.096}{0.118} \times 100 = 18.6\% \text{ at } 2.1\% \text{ add-on}$$

$$\frac{0.206 - 0.193}{0.206} \times 100 = 6.3\% \text{ at } 3.7\% \text{ add-on}$$

$$\frac{0.272 - 0.241}{0.272} \times 100 = 11.4\% \text{ at } 4.9\% \text{ add-on}$$

Table 7

Percentage add-on

Intended add-on	Add-on from weight gain before wash	Add-on% from weight gain after wash	Add-on% from nitrogen % before wash	Add-on% from nitrogen% after wash
1%	2.2%	2.24%	2.10%	1.70%
3%	3.1%	3.90%	3.70%	3.45%
6%	5.7%	7.50%	4.85%	4.30%

Table 8

L, a, b & YI values of fabric with 2.2% add-on

Sample	Mean	Significance
LU1	85.96	
L31	85.95	
L31	85.95	
L51	86.07	
LU1	85.96	
L51	86.07	
aU1	-0.06	
a31	0.37	***
a31	0.37	
a51	0.34	
aU1	-0.06	
a51	0.34	***
bU1	13.70	
b31	13.03	***
b31	13.03	
b51	12.48	***
bU1	13.70	
b51	12.48	***
YU1	32.26	
Y51	25.92	***

Table 9

L, a, b & YI values of fabric with 3.1% add-on

Sample	Mean	Significance
LU3	77.69	
L33	77.87	
L33	77.87	
L53	77.14	
LU3	77.69	
L53	77.14	
aU3	2.95	
a33	3.25	
a33	3.25	
a53	3.20	
aU3	2.95	
a53	3.20	
bU3	19.00	
b33	17.52	***
b33	17.52	
b53	16.87	***
bU3	19.00	
b53	16.87	***
YU3	46.79	
Y33	43.53	***
Y33	43.53	
Y53	41.78	
YU3	46.79	
Y53	41.78	***

Table 10

L, a, b & YI values of fabric with 5.7% add-on

Sample	Mean	Significance
LU6	69.79	
L36	69.53	
L36	69.53	
L56	70.14	
LU6	69.79	
L56	70.14	
aU6	5.42	
a36	5.45	
a36	5.45	
a56	5.29	
aU6	5.43	
a56	5.29	
bU6	19.73	
b36	18.74	***
b36	18.74	
b56	18.23	***
bU6	19.73	
b56	18.23	***
YU6	56.87	
Y36	54.54	
Y36	54.54	
Y56	52.47	
YU6	56.87	
Y56	52.47	***

Table 11
Overall color difference

Add-on%	Comparison	ΔE	ΔC
2.2%	No wash to 3 washes	0.79	0.79
	No wash to 5 washes	1.28	1.28
3.1%	No wash to 3 washes	1.52	1.51
	No wash to 5 washes	2.17	2.14
5.9%	No wash to 3 washes	0.99	0.99
	No wash to 5 washes	1.60	1.50

$$\Delta E = \text{Overall color change} = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}$$

$$\Delta C = \text{Chromaticity difference} = \sqrt{\Delta a^2 + \Delta b^2}$$

Table 12

Mean values of physical tests

	Add-on%		
	Unwashed	Washed	2.2% 3.1% 5.9%
Durable press rating		1.60	2.35
Tensile strength (kg)	25.22	23.65	27.90
Elongation (%)	10.96	22.18	16.06
Abrasion resistance (cycles)	218.50	260.20	270.00
Warp shrinkage (%)		3.54	1.45
Weft shrinkage (%)		13.55	6.90
Weight change (%) (after 3 washes)		0.12	0.03
Weight change (%) (after 5 washes)		0.11	0.04
Recovery angle (W+F)	187.30	190.60	172.40
			297.80
			1.25
			9.00
			0.05
			0.07
			173.10
			272.50
			1.95
			8.20
			0.17
			0.20
			132.50

Table 13

Statistical analysis
 *** indicates significance at 95% level

Comparison	Durable press rating	Wt. change (3 washes)	Wt. change (5 washes)	Warp shrinkage	Weft shrinkage	Tensile strength	Elongation	Abrasion resistance	Warp recovery angle	Weft recovery angle
Unwashed to 2.2%	***+	***	***	***	***		***+		***	
Unwashed to 3.1%	***+	***	***	***	***		***+	***+	***	
Unwashed to 5.9%							***+		***	***
Unwashed to washed										
0% to 2.2%	***+	***	***	***	***		***		***-	
0% to 3.1%	***+	***	***	***	***		***		***-	
0% to 5.9%		***	***	***	***		***	***		
2.2% to 3.1%		***			***					
3.1% to 5.9%			***				***		***-	***
2.2% to 5.9%			***						***-	***

+ improved
 - not improved

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RAW DATA

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Abbreviations used

ORIG WT.....Original weight of the sample in grams
FIN WT.....Final weight of the sample in grams
BEF WASH.....Weight of the sample before wash in grams
AFT WAS(3).....Weight of the sample after 3 washes
AFT WAS(5).....Weight of the sample after 5 washes
WARP.....Length of the warp in inches after 5 washes
WEFT.....Length of the weft in inches after 5 washes
L, a, b.....Color values from Hunter Colorimeter
YI.....Yellowness Index from Hunter Colorimeter
DP.....Durable Press rating
Ten.str.....Tensile strength
Elon.....Elongation%
Abr.Res.....Abrasion resistance
Warp. rec. angle..Warp recovery angle
Weft. rec. angle..Weft recovery angle
Warp shr.....Warp shrinkage%
Weft shr.....Weft shrinkage%

RAW DATA

PHYSICAL PROPERTIES OF SAMPLES TESTED

	ORIG WT	FIN WT	ADD ON%	BEF WASH	AFT WAS(3)	AFT WAS(5)	WARP	WEFT
A-0-1W	---	---	---	12.3023	12.4300	12.4147	9.62	8.62
A-0-2W	---	---	---	12.1492	12.2627	12.2342	9.62	8.62
A-0-3W	---	---	---	12.1753	12.2559	12.2536	9.62	8.62
A-0-4W	---	---	---	12.3500	12.4673	12.4685	9.62	8.62
A-0-5W	---	---	---	12.9839	12.0787	12.0612	9.62	8.62
B-0-1W	---	---	---	12.6550	12.7459	12.7677	9.75	8.62
B-0-2W	---	---	---	12.3797	12.6237	12.6077	9.62	8.75
B-0-3W	---	---	---	12.5089	12.6015	12.6208	9.62	8.62
B-0-4W	---	---	---	12.3840	12.5100	12.4864	9.62	8.62
B-0-5W	---	---	---	12.6447	12.7653	12.7700	9.75	8.62
A-1-1S	12.3230	12.7706	3.63	10.6000	10.6140	10.6013	9.70	9.20
A-1-2S	12.6133	12.8558	1.90	10.9822	11.0238	11.0140	9.85	9.00
A-1-3S	12.1700	12.3917	1.80	10.4300	10.4583	10.4712	9.85	9.10
A-1-4S	12.1135	12.3724	2.10	10.4030	10.4269	10.4272	9.85	9.15
A-1-5S	12.9073	13.1482	1.90	10.9632	11.9865	11.0512	9.90	9.50
B-1-1S	12.5329	12.7693	1.90	10.8915	10.9439	10.9371	9.90	9.35
B-1-2S	12.3569	12.6000	1.90	10.4327	10.4624	10.4750	9.90	9.30
B-1-3S	12.0716	12.3362	2.10	10.4390	10.4600	10.4569	9.85	9.40
B-1-4S	12.9754	13.2671	2.20	11.1644	11.1816	11.2089	9.90	9.10
B-1-5S	13.0900	13.3619	2.10	11.5880	11.6300	11.6355	9.85	9.50
A-3-1S	12.4400	12.8013	2.90	10.9943	11.0983	11.0851	9.90	9.30
A-3-2S	12.5817	12.9879	3.20	11.1621	11.2379	11.2685	9.85	9.10
A-3-3S	12.6263	13.0289	3.20	11.3729	11.4680	11.4512	9.85	9.20
A-3-4S	12.7800	13.1900	3.20	11.3762	11.3951	11.4844	9.90	9.00
A-3-5S	12.9463	13.3631	3.20	11.5499	11.6012	11.6149	9.90	9.10
B-3-1S	12.4200	12.7970	3.10	10.8352	11.9526	10.9309	9.80	9.10
B-3-2S	12.5320	12.9232	3.10	11.2548	11.2583	11.3600	9.90	9.00
B-3-3S	12.3272	12.7270	3.20	11.0931	11.0769	11.1972	9.85	9.20
B-3-4S	12.5084	12.9056	3.20	11.3368	11.4136	11.4049	9.90	9.10
B-3-5S	12.5489	12.9208	3.00	11.3116	11.4025	11.3938	9.90	9.00
A-6-1S	11.8587	12.5763	6.04	10.5285	10.7012	10.7400	9.80	9.40
A-6-2S	12.1000	12.8535	6.20	10.5149	10.6953	10.7474	9.70	9.10
A-6-3S	13.2977	14.0826	5.90	11.8721	12.0224	12.0124	9.90	9.30
A-6-4S	12.4624	13.1610	5.60	11.2400	11.3965	11.4569	9.85	9.10
A-6-5S	12.9491	13.6885	5.70	12.0753	12.2416	12.2984	9.90	9.20
B-6-1S	12.6169	13.3107	5.50	11.5784	11.7006	11.7700	9.70	9.10
B-6-2S	12.7717	13.5819	6.30	12.1220	12.4012	12.3384	9.80	9.30
B-6-3S	13.3678	14.0309	6.20	12.1727	12.3419	12.3744	9.90	9.00
B-6-4S	12.8338	13.5754	5.80	12.0400	12.1921	12.2483	9.80	9.10
B-6-5S	12.7555	13.4684	5.60	11.1500	11.3642	11.3753	9.70	9.20

L, a, b & YI values - for finished samples which were unwashed,
washed 3 and 5 times

BEFORE WASH

A-1-1S				B-1-1S			
L	a	b	YI	L	a	b	YI
86.53	-0.06	13.85	-----	86.22	-0.02	14.37	-----
86.70	-0.10	13.94	-----	86.30	-0.06	14.11	-----
86.90	-0.17	13.29	-----	86.75	-0.13	13.20	-----
86.51	0.01	13.13	-----	86.32	-0.08	14.04	-----
86.31	0.03	13.38	30.30	86.54	-0.08	14.01	31.15
86.70	-0.10	13.40	-----	86.35	0.06	13.53	-----
86.61	-0.06	13.70	-----	86.40	0.09	13.18	-----
86.26	0.04	14.00	-----	86.29	0.08	13.62	-----
86.40	-0.02	13.96	-----	86.57	-0.02	13.50	-----
86.62	-0.07	13.45	-----	86.50	0.00	13.67	-----

BEFORE WASH

A-1-2-S				B-1-2S			
87.13	-0.19	12.89	-----	86.36	-0.11	14.00	-----
87.63	-0.35	12.49	-----	86.45	-0.14	14.05	-----
86.80	-0.15	13.41	-----	86.71	-0.21	13.67	-----
86.56	-0.09	13.78	-----	86.42	-0.16	14.03	-----
86.95	-0.21	13.18	33.62	86.47	-0.08	14.03	30.73
87.51	-0.38	12.89	-----	86.42	0.02	13.60	-----
87.18	-0.32	13.11	-----	86.50	-0.02	13.56	-----
86.16	0.00	14.22	-----	87.00	-0.19	13.07	-----
87.07	-0.31	13.28	-----	86.56	-0.08	13.65	-----
87.02	-0.27	13.35	-----	86.07	0.07	14.01	-----

BEFORE WASH

A-1-3S				B-1-3S			
86.44	0.07	13.20	-----	87.21	-0.11	12.21	-----
86.39	0.02	12.75	-----	86.64	-0.03	13.64	-----
86.48	0.04	13.70	-----	86.76	0.02	12.62	-----
86.56	0.02	13.51	-----	86.65	0.00	13.33	-----
87.04	-0.22	13.01	32.20	86.40	0.03	13.70	32.49
86.01	0.05	14.19	-----	86.82	-0.21	14.01	-----
86.39	-0.09	13.62	-----	86.68	-0.05	13.41	-----
86.82	-0.16	13.61	-----	86.71	-0.15	14.28	-----
87.39	-0.33	12.99	-----	86.49	-0.10	14.39	-----
86.77	-0.14	13.66	-----	86.31	-0.04	14.41	-----

BEFORE WASH

A-1-4S				B-1-4S			
L	a	b	YI	L	a	b	YI
86.38	-0.10	14.10	-----	86.48	0.03	13.51	-----
86.17	-0.06	14.31	-----	86.60	-0.04	13.30	-----
86.17	-0.04	14.30	-----	86.45	0.03	13.56	-----
86.92	-0.21	13.49	-----	86.29	0.10	13.58	-----
86.96	-0.23	13.37	31.83	86.29	0.05	13.49	-----
86.66	0.02	12.82	-----	86.54	-0.14	14.25	-----
86.26	0.03	13.53	-----	86.70	-0.18	14.01	-----
86.38	-0.04	13.65	-----	86.05	0.00	14.60	-----
87.02	-0.14	13.15	-----	85.83	0.05	14.74	-----
86.74	-0.13	13.66	-----	86.23	-0.04	14.74	-----

BEFORE WASH

A-1-5S				B-1-5S			
86.76	-0.20	14.03	-----	86.08	0.06	14.09	-----
85.95	0.01	14.69	-----	86.23	0.02	13.97	-----
86.13	-0.06	14.50	-----	86.99	-0.16	13.37	-----
86.14	-0.09	14.14	-----	87.09	-0.15	13.32	-----
86.01	0.00	14.53	34.41	86.98	-0.18	13.36	-----
86.07	0.09	14.28	-----	86.81	-0.18	13.93	-----
86.64	-0.04	13.75	-----	86.36	-0.06	14.18	-----
86.60	-0.09	13.67	-----	86.32	-0.03	14.19	-----
86.73	-0.06	13.74	-----	86.73	-0.09	13.71	-----
86.12	0.11	13.75	-----	87.00	-0.18	13.49	-----

AFTER 3 WASHES

A-1-1S				B-1-1S			
86.19	0.28	12.47	-----	85.21	0.45	14.25	-----
86.24	0.26	12.60	-----	85.75	0.43	12.90	-----
85.49	0.47	13.86	-----	86.76	0.34	13.36	-----
86.31	0.26	12.95	-----	85.57	0.46	13.66	-----
85.70	0.52	12.92	-----	85.75	0.44	13.49	-----
85.30	0.46	13.89	-----	86.03	0.51	12.35	-----
86.44	0.21	12.71	-----	86.20	0.52	11.85	-----
86.42	0.21	12.41	-----	86.08	0.46	12.67	-----
86.08	0.43	12.30	-----	86.48	0.37	12.23	-----
86.32	0.51	11.03	-----	85.88	0.55	12.25	-----

AFTER 3 WASHES

A-1-2S				B-1-2S			
L	a	b	YI	L	a	b	YI
86.64	0.21	11.91	-----	85.35	0.38	14.11	-----
87.16	-0.01	11.55	-----	85.20	0.43	14.39	-----
85.92	0.50	12.93	-----	85.38	0.32	13.69	-----
86.67	0.16	11.92	-----	85.03	0.40	13.84	-----
85.65	0.13	12.25	-----	85.30	0.63	13.26	-----
86.94	-0.09	12.12	-----	85.25	0.58	13.54	-----
86.72	0.01	12.44	-----	85.86	0.39	12.89	-----
85.75	0.33	13.36	-----	85.86	0.44	13.19	-----
86.54	0.05	12.61	-----	84.79	0.68	14.05	-----
86.14	0.18	13.02	-----	85.13	0.62	13.92	-----

AFTER 3 WASHES

A-1-3S				B-1-3S			
86.62	0.38	11.18	-----	85.78	0.61	12.23	-----
85.68	0.52	12.48	-----	85.95	0.44	12.85	-----
85.57	0.59	12.90	-----	85.47	0.58	13.21	-----
86.25	0.42	12.30	-----	86.22	0.45	12.44	-----
86.87	0.19	11.54	-----	86.42	0.33	12.37	-----
85.14	0.49	13.94	-----	85.74	0.32	13.67	-----
86.39	0.20	12.60	-----	85.58	0.33	13.97	-----
86.25	0.18	12.81	-----	85.59	0.36	13.84	-----
86.21	0.17	12.96	-----	85.82	0.27	13.93	-----
86.06	0.29	13.04	-----	85.76	0.29	13.90	-----

AFTER 3 WASHES

A-1-4S				B-1-4S			
85.45	0.25	13.46	-----	85.80	0.40	12.44	-----
85.66	0.26	13.81	-----	85.53	0.55	12.89	-----
86.38	0.26	13.81	-----	85.46	0.57	12.92	-----
86.42	0.16	12.74	-----	85.54	0.63	12.70	-----
86.17	0.20	13.07	-----	85.38	0.59	13.45	-----
85.86	0.55	12.35	-----	84.96	0.51	13.85	-----
85.91	0.48	12.36	-----	85.16	0.35	14.49	-----
86.45	0.32	12.34	-----	85.59	0.28	13.93	-----
86.40	0.21	12.87	-----	85.41	0.37	14.21	-----
86.60	0.19	12.23	-----	85.08	0.49	14.48	-----

AFTER 3 WASHES

A-1-5S				B-1-5S			
L	a	b	YI	L	a	b	YI
86.78	0.66	14.34	-----	87.03	0.02	11.29	-----
84.75	0.57	14.80	-----	86.54	0.21	12.45	-----
85.45	0.43	14.14	-----	85.97	0.36	13.20	-----
86.13	0.17	13.30	-----	85.54	0.44	13.23	-----
85.98	0.29	13.64	-----	86.46	0.21	12.21	-----
85.61	0.50	13.00	-----	85.71	0.31	13.20	-----
85.46	0.62	13.09	-----	85.30	0.41	14.26	-----
85.75	0.58	12.86	-----	85.87	0.30	13.27	-----
86.10	0.41	12.81	-----	86.25	0.29	12.88	-----
85.94	0.45	12.94	-----	85.93	0.21	13.36	-----

AFTER 5 WASHES

A-1-1S				B-1-1S			
86.03	0.34	12.68	26.39	86.56	0.40	13.12	27.51
86.04	0.47	12.38	25.92	86.08	0.28	12.74	26.45
86.28	0.46	11.41	23.71	86.15	0.34	12.63	26.24
86.65	0.37	10.94	22.51	86.11	0.31	12.93	26.78
86.86	0.31	10.95	22.46	86.07	0.25	12.78	26.45
86.03	0.47	11.88	24.82	86.24	0.39	11.43	23.72
86.50	0.33	11.83	24.43	86.45	0.47	11.33	23.55
86.33	0.19	12.63	26.06	85.91	0.57	12.38	26.01
86.56	0.21	11.99	24.63	86.36	0.40	11.88	24.66
86.69	0.33	11.24	23.10	86.31	0.40	11.98	24.88

AFTER 5 WASHES

A-1-2S				B-1-2S			
86.77	0.11	11.43	23.37	85.95	0.26	13.02	27.01
87.10	0.02	11.37	23.02	85.80	0.38	13.37	28.00
86.06	0.42	12.58	26.25	85.72	0.35	13.48	28.18
84.92	0.72	13.61	29.09	85.57	0.42	13.61	28.59
86.80	0.11	11.73	23.93	85.80	0.37	13.31	27.82
87.05	-0.07	11.58	23.35	85.44	0.66	12.91	27.38
87.04	-0.01	11.75	23.78	86.01	0.47	12.41	25.96
85.98	0.27	13.28	27.61	85.90	0.53	12.69	26.62
86.59	0.11	12.44	25.48	85.82	0.46	12.85	26.93
86.08	0.29	12.80	26.56	85.14	0.68	13.22	28.15

AFTER 5 WASHES

A-1-3S				B-1-3S			
L	a	b	YI	L	a	b	YI
86.34	0.42	11.38	23.62	86.94	0.40	10.03	20.62
86.16	0.49	11.84	24.70	86.51	0.29	11.88	24.49
85.79	0.69	12.05	25.47	86.41	0.46	11.52	23.94
86.33	0.44	11.89	24.73	86.26	0.37	12.16	25.25
86.83	0.29	10.90	22.36	86.15	0.49	12.28	25.67
85.38	0.46	13.26	27.93	85.79	0.25	13.41	27.87
86.03	0.34	12.44	25.86	86.38	0.14	12.80	26.32
86.58	0.16	12.18	24.97	86.05	0.27	13.02	27.01
86.72	0.07	12.19	24.87	85.64	0.38	13.77	28.85
86.28	0.22	12.69	26.19	85.85	0.32	13.34	27.78

AFTER 5 WASHES

A-1-4S				B-1-4S			
85.79	0.39	12.78	26.74	85.34	0.62	12.09	25.59
85.98	0.36	12.60	26.25	85.61	0.63	12.51	26.44
86.59	0.15	12.08	24.79	85.56	0.62	12.78	27.02
86.15	0.31	12.41	25.78	85.90	0.58	12.08	25.38
86.21	0.44	11.94	24.88	86.30	0.32	11.73	24.28
86.47	0.05	12.25	25.06	85.05	0.40	13.75	29.02
85.99	0.27	12.99	26.97	85.56	0.35	13.61	28.49
85.88	0.25	13.21	27.48	85.70	0.37	13.25	27.68
86.05	0.24	12.99	26.91	85.47	0.26	13.43	28.05
86.02	0.18	13.17	27.27	86.01	0.17	13.17	27.24

AFTER 5 WASHES

A-1-5S				B-1-5S			
85.79	0.17	13.10	27.17	85.80	0.40	12.59	26.35
85.71	0.38	13.28	27.77	86.00	0.42	12.61	26.34
85.45	0.42	13.84	29.10	86.56	0.24	11.97	24.60
85.40	0.40	13.68	28.76	87.33	0.03	11.09	22.34
86.39	0.18	12.73	26.22	86.96	0.11	11.47	23.35
85.71	0.55	12.71	26.74	85.77	0.25	12.59	26.16
85.75	0.52	12.76	26.81	87.23	-0.10	11.80	23.73
86.45	0.27	12.23	25.23	86.08	0.31	12.72	26.42
85.93	0.46	12.74	26.65	85.46	0.47	13.54	28.52
86.06	0.47	12.12	25.33	85.91	0.32	13.09	27.23

BEFORE WASH

A-3-1S				B-3-1S			
L	a	b	YI	L	a	b	YI
79.61	2.34	17.83	42.47	80.87	1.69	17.93	41.38
79.98	2.25	17.73	41.96	80.38	1.93	18.26	42.43
80.69	1.98	17.58	41.00	79.70	2.24	18.38	43.50
80.53	1.95	18.23	42.46	79.81	2.21	18.21	43.07
80.33	2.00	18.05	42.20	80.03	2.14	18.23	42.91
80.23	1.99	18.96	44.26	80.17	2.04	18.44	43.20
79.83	2.08	18.81	44.23	79.06	2.46	18.90	45.24
81.04	1.65	18.17	41.73	79.51	2.27	18.84	44.65
78.83	2.44	19.69	47.11	79.98	1.94	18.35	43.00
79.23	2.32	19.52	46.38	80.36	1.90	17.93	41.82

BEFORE WASH

A-3-2S				B-3-2S			
76.63	3.39	19.30	48.54	76.49	3.42	19.20	48.47
76.12	3.58	19.40	49.29	76.56	3.33	19.24	48.43
76.00	3.66	19.23	49.10	76.17	3.56	19.41	49.30
76.58	3.45	19.37	48.85	77.10	3.23	18.92	47.24
76.52	3.45	19.37	48.85	76.97	3.27	18.99	47.50
76.58	3.45	19.45	49.00	76.70	3.33	19.38	48.62
76.87	3.34	19.19	48.09	76.46	3.42	19.40	48.92
76.14	3.59	19.36	49.19	77.13	3.15	19.29	47.97
76.52	3.43	19.32	48.70	77.02	3.25	19.19	47.91
76.67	3.41	19.32	48.60	76.79	3.26	19.40	48.55

BEFORE WASH

A-3-3S				B-3-3S			
76.41	3.43	19.41	49.00	77.42	3.00	19.36	47.80
76.74	3.29	19.21	48.16	77.95	2.89	18.79	46.07
76.44	3.34	19.47	49.04	77.57	2.98	19.05	46.96
76.36	3.45	19.41	49.03	77.91	2.81	18.84	46.13
76.64	3.31	19.47	48.83	77.36	3.07	19.01	47.12
76.82	3.29	19.21	48.12	78.15	2.72	18.75	45.70
76.72	3.36	19.22	48.29	78.23	2.70	18.63	45.33
76.48	3.48	19.11	48.26	77.75	2.94	18.82	46.34
77.02	3.25	19.20	47.93	77.47	2.98	19.12	47.20
77.05	3.22	19.23	47.94	77.41	3.01	19.09	47.20

BEFORE WASH

A-3-4S				B-3-4S			
L	a	b	YI	L	a	b	YI
78.52	2.61	19.14	46.24	77.85	2.88	19.16	46.95
78.57	2.61	18.97	45.83	76.69	3.32	19.30	48.42
78.19	2.78	18.98	46.24	76.58	3.37	19.29	48.53
77.83	2.88	18.93	46.45	76.77	3.27	19.27	48.25
78.55	2.61	18.75	45.34	77.79	2.93	18.95	46.57
76.57	3.36	19.26	48.46	76.38	3.42	19.17	48.47
77.42	3.01	19.08	47.19	76.95	3.27	19.10	47.79
78.39	2.73	18.68	45.40	77.71	2.96	18.99	46.73
79.14	2.40	18.23	43.64	77.69	2.96	19.05	46.89
78.80	2.59	18.46	44.55	76.81	3.26	19.30	48.29

BEFORE WASH

A-3-5S				B-3-5S			
77.75	2.91	19.17	47.05	78.11	2.80	18.78	45.88
77.26	3.15	19.14	47.54	77.80	2.90	18.86	46.33
76.79	3.30	19.33	48.42	76.96	3.21	19.09	47.65
76.37	3.43	19.48	49.17	77.20	3.13	19.06	47.38
76.77	3.34	19.32	48.45	77.62	2.98	18.77	46.32
75.52	3.70	19.47	50.02	77.51	3.06	18.99	46.97
77.19	3.11	19.12	47.52	77.56	2.97	18.99	46.85
76.77	3.31	19.24	48.22	77.91	2.87	19.00	46.54
76.19	3.52	19.16	48.61	76.85	3.14	19.34	48.26
75.88	3.61	19.32	49.30	76.35	3.45	19.46	49.14

AFTER 3 WASHES

A-3-1S				B-3-1S			
79.58	2.70	17.18	41.27	79.52	2.77	17.07	41.13
79.51	2.74	17.35	41.72	79.36	2.78	17.22	41.56
79.81	2.67	17.05	40.86	80.10	2.56	16.91	40.26
80.03	2.54	17.05	40.60	80.51	2.38	16.76	39.55
80.40	2.35	16.83	39.71	80.17	2.51	16.80	39.93
80.46	2.29	16.82	39.58	80.76	2.28	16.61	38.97
79.55	2.69	17.83	42.72	79.55	2.67	17.80	42.64
79.76	2.67	17.46	41.79	79.53	2.64	17.81	42.62
79.20	2.73	18.08	43.70	79.83	2.52	17.63	41.97
79.82	2.63	17.45	41.66	80.25	2.47	17.11	40.56

AFTER 3 WASHES

A-3-2S				B-3-2S			
L	a	b	YI	L	a	b	YI
77.01	3.62	17.61	44.59	77.80	3.30	17.60	43.79
77.06	3.55	17.85	45.05	76.64	3.71	17.97	45.73
77.02	3.60	18.00	45.46	77.13	3.60	17.77	44.88
76.52	3.73	17.87	45.63	77.41	3.51	17.34	43.63
77.01	3.57	17.70	44.75	76.47	3.75	17.83	45.57
76.88	3.51	17.92	45.27	76.98	3.60	17.75	44.90
76.61	3.65	18.27	46.41	76.73	3.65	18.22	46.20
77.04	3.53	17.70	44.71	77.04	3.57	18.31	46.11
76.46	3.67	17.78	45.37	76.78	3.58	18.20	46.06
75.86	3.84	18.17	46.81	76.83	3.65	17.83	45.24

AFTER 3 WASHES

A-3-3S				B-3-3S			
76.35	3.76	17.76	45.46	77.67	3.30	17.68	44.05
77.00	3.64	17.80	45.06	77.55	3.38	17.54	43.87
77.62	3.57	18.30	45.72	77.58	3.36	17.61	44.01
76.85	3.65	18.08	45.78	77.96	3.21	17.45	43.25
77.27	3.69	17.95	45.58	77.67	3.23	17.35	43.21
77.50	3.41	17.45	43.72	78.20	3.12	17.14	42.35
77.08	3.64	17.63	44.64	77.85	3.24	17.34	43.10
76.98	3.60	17.60	45.56	78.23	3.20	17.06	42.21
76.50	3.81	17.70	45.30	77.51	3.41	17.62	44.11
77.31	3.52	17.61	44.30	77.90	3.18	17.29	42.88

AFTER 3 WASHES

A-3-4S				B-3-4S			
77.42	3.37	17.55	43.96	77.32	3.42	17.81	44.64
78.44	3.02	17.27	42.38	76.53	3.74	17.92	45.73
78.72	2.97	17.48	42.66	76.78	3.60	17.93	46.43
78.18	3.08	18.23	44.75	76.52	3.73	17.85	45.54
78.94	2.85	17.51	42.47	77.93	3.24	17.72	43.93
79.24	2.81	16.41	39.81	76.47	3.68	17.85	45.52
78.79	2.94	16.81	41.08	77.25	3.48	17.75	44.64
78.72	3.12	16.99	41.94	77.78	3.24	17.34	43.15
78.99	2.89	16.78	40.88	77.58	3.32	17.32	43.31
78.63	2.97	16.63	40.80	76.65	3.60	17.89	45.43

AFTER 3 WASHES

A-3-5S				B-3-5S			
L	a	b	YI	L	a	b	YI
77.86	3.22	17.14	42.61	78.54	3.00	17.02	41.75
78.17	3.25	17.02	42.19	77.86	3.33	17.12	42.70
77.87	3.34	17.15	42.74	77.11	3.59	17.39	44.00
76.87	3.56	17.81	45.03	77.37	3.54	17.10	43.13
76.63	3.63	17.57	44.73	77.67	3.33	17.10	42.73
75.56	4.02	18.11	47.06	77.03	3.53	17.44	44.09
77.11	3.50	17.76	44.74	77.46	3.42	17.42	43.39
78.01	3.24	17.43	43.22	78.12	3.20	17.53	43.35
78.13	3.25	17.12	42.43	78.08	3.19	17.60	43.49
76.92	3.59	17.51	44.39	76.97	3.60	17.79	45.00

AFTER 5 WASHES

A-3-1S				B-3-1S			
79.77	2.57	16.37	39.23	81.20	2.12	15.56	36.21
79.70	2.63	16.47	39.56	81.29	2.12	15.51	36.03
80.74	2.18	16.49	38.65	80.79	2.34	15.85	37.21
80.37	2.26	16.76	39.48	80.78	2.43	15.53	36.64
81.11	1.99	16.25	37.74	80.80	2.38	15.44	36.38
79.44	2.41	18.20	43.32	80.28	2.26	16.04	37.81
79.17	2.61	18.43	44.14	80.89	2.27	15.96	37.38
79.46	2.50	18.31	43.64	81.15	2.15	15.84	36.86
80.41	2.12	17.69	41.38	80.25	2.47	16.89	39.94
80.39	2.20	17.17	40.30	79.55	2.71	16.94	40.63

AFTER 5 WASHES

A-3-2S				B-3-2S			
77.59	3.42	17.02	42.66	78.19	3.21	16.70	41.40
77.01	3.64	17.09	43.34	77.26	3.53	17.26	43.50
76.68	3.76	17.30	44.15	76.98	3.62	17.14	43.45
77.12	3.63	17.20	43.54	77.03	3.65	16.97	43.08
76.91	3.64	17.34	43.98	77.08	3.55	17.17	43.41
76.38	3.83	17.54	44.92	77.26	3.47	17.06	42.96
77.27	3.56	17.27	43.56	77.16	3.50	17.45	43.96
76.85	3.71	17.24	43.86	76.89	3.68	17.22	43.77
77.10	3.58	17.33	43.79	77.33	3.58	17.07	43.03
76.89	3.69	17.24	43.80	76.95	3.59	17.58	44.46

AFTER 5 WASHES

A-3-3S				B-3-3S			
L	a	b	YI	L	a	b	YI
76.89	3.63	17.10	43.41	77.97	3.13	16.73	41.46
77.37	3.57	16.97	42.79	78.50	3.09	16.67	41.00
76.85	3.70	17.31	44.03	78.75	3.09	16.39	40.26
77.41	3.54	17.03	42.88	77.89	3.37	16.76	41.85
76.70	3.72	17.41	44.34	78.17	3.26	16.63	41.27
76.07	3.74	17.03	43.84	78.20	3.21	16.91	41.82
77.47	3.53	16.75	42.19	78.20	3.21	16.84	41.66
77.21	3.56	16.67	42.19	77.72	3.28	16.71	41.71
77.44	3.50	16.99	42.74	77.78	3.35	16.73	41.82
77.33	3.54	16.90	42.61	77.63	3.41	17.13	42.85

AFTER 5 WASHES

A-3-4S				B-3-4S			
78.51	2.98	16.83	41.22	76.41	3.65	17.21	44.03
78.83	2.87	17.22	41.84	77.07	3.60	16.92	42.90
78.40	2.98	17.29	42.35	77.40	3.47	16.84	42.40
79.23	2.83	16.62	40.23	77.82	3.34	16.81	41.96
79.25	2.83	16.32	39.55	78.02	3.25	16.97	42.09
79.08	2.95	15.88	38.76	77.32	3.42	17.02	42.81
79.12	2.93	15.74	38.41	76.67	3.74	17.34	44.24
79.25	2.88	15.88	38.61	76.68	3.69	17.15	43.73
79.18	2.98	16.05	39.07	77.83	3.35	16.93	42.21
79.46	2.78	16.11	38.93	77.90	3.34	16.88	42.07

AFTER 5 WASHES

A-3-5S				B-3-5S			
78.56	3.07	16.75	41.14	77.26	3.50	16.58	41.89
77.36	3.48	17.10	43.02	77.42	3.50	16.80	42.30
77.37	3.47	17.22	43.25	77.63	3.42	16.60	41.65
76.47	3.86	17.40	44.59	78.90	3.03	16.25	39.79
76.64	3.78	17.34	44.29	77.97	3.28	16.61	41.36
77.11	3.50	17.03	43.01	77.58	3.36	17.17	42.92
77.68	3.42	16.86	42.23	77.08	3.55	17.44	44.01
77.86	3.32	16.84	41.96	76.71	3.69	17.10	43.61
78.02	3.37	16.68	41.55	77.42	3.53	17.03	42.86
76.70	3.74	17.04	43.49	77.94	3.25	16.87	41.90

BEFORE WASH

A-6-1S				B-6-1S			
L	a	b	YI	L	a	b	YI
71.77	4.75	19.65	54.14	69.84	5.39	19.74	56.58
72.95	4.41	19.34	52.18	71.82	4.86	19.37	53.56
73.07	4.33	19.11	51.45	70.39	5.22	19.76	55.91
72.38	4.57	19.38	52.87	70.77	5.13	19.75	55.56
71.29	4.83	19.40	54.00	68.79	5.69	20.13	58.72
73.90	4.09	19.50	51.55	70.46	5.20	19.67	55.70
72.72	4.46	19.46	52.68	71.05	5.04	19.65	54.99
70.75	5.08	19.90	55.90	70.82	5.09	19.47	54.78
70.31	5.09	19.80	55.99	69.96	5.31	19.60	56.02
71.97	4.65	19.54	53.64	70.96	5.03	19.70	55.18

BEFORE WASH

A-6-2S				B-6-2S			
68.22	5.78	19.85	58.64	68.20	5.82	19.94	58.92
67.55	5.95	19.53	58.85	68.47	5.75	19.89	58.47
69.64	5.46	20.12	57.77	69.56	5.43	19.49	56.23
69.01	5.54	19.46	56.74	71.19	5.03	19.51	54.56
70.25	5.25	19.58	55.71	70.85	5.10	19.52	54.92
67.06	6.10	19.65	59.48	69.96	5.39	19.94	56.97
67.29	6.03	19.83	59.67	70.59	5.20	19.57	55.33
67.42	5.96	19.89	59.61	69.33	5.50	19.75	57.11
70.01	5.33	19.82	56.59	69.35	5.52	19.71	57.02
68.83	5.70	20.28	59.10	68.37	5.73	19.64	57.91

BEFORE WASH

A-6-3S				B-6-3S			
71.05	5.11	19.82	55.81	71.42	4.97	20.28	56.20
68.78	5.70	20.20	58.91	70.98	5.11	20.15	56.39
69.15	5.62	20.40	59.01	69.17	5.57	20.13	58.28
69.81	5.42	20.07	57.45	69.01	5.61	19.74	57.49
69.75	5.47	20.28	58.05	69.06	5.66	20.00	58.16
68.37	5.84	20.22	59.49	71.70	4.88	19.35	53.59
69.18	5.62	20.37	58.95	71.28	4.96	19.57	54.56
70.68	5.25	20.17	56.83	70.31	5.24	19.61	55.70
70.23	5.35	20.31	57.61	70.38	5.23	19.37	55.06
69.69	5.51	20.37	58.39	69.86	5.37	19.50	55.92

BEFORE WASH							
A-6-4S				B-6-4S			
L	a	b	YI	L	a	b	YI
63.35	6.90	19.79	64.28	71.08	5.14	19.89	55.69
63.63	6.80	19.36	62.70	71.06	5.12	19.44	54.57
65.13	6.56	19.80	62.19	70.71	5.25	19.77	55.77
64.27	6.68	19.69	62.81	71.46	5.03	19.71	54.81
63.51	6.84	19.63	63.59	71.37	5.08	19.73	55.02
65.43	6.38	19.47	60.79	70.47	5.30	19.81	56.13
64.17	6.67	19.54	62.53	70.22	5.31	19.52	55.64
65.33	6.38	19.39	60.73	71.38	5.05	19.66	54.79
63.15	6.89	19.56	63.84	71.16	5.12	19.65	55.01
64.09	6.67	19.51	62.51	72.31	4.75	19.45	53.26

BEFORE WASH							
A-6-5S				B-6-5S			
70.03	5.38	19.80	56.57	70.89	5.05	19.25	54.19
70.31	5.21	19.42	55.28	71.05	4.98	19.43	54.39
69.65	5.46	19.77	56.92	69.24	5.53	19.57	56.80
68.83	5.72	19.81	57.94	69.40	5.50	19.54	57.06
69.05	5.61	19.61	57.12	69.80	5.39	19.65	56.04
70.65	5.16	19.37	54.78	70.09	5.31	20.13	57.27
68.77	5.58	19.08	55.97	71.03	5.07	20.21	56.41
69.78	5.36	19.64	56.35	71.75	4.88	19.96	55.08
69.01	5.59	19.41	56.68	70.00	5.34	19.93	56.86
69.96	5.36	18.94	54.48	69.20	5.55	19.89	57.63

AFTER 3 WASHES							
A-6-1S				B-6-1S			
72.08	4.74	17.81	49.36	70.52	5.24	19.19	54.49
70.40	5.32	18.31	52.29	69.53	5.47	19.12	55.35
71.18	5.19	18.28	51.65	68.14	5.80	19.22	57.06
72.29	4.81	18.16	50.20	69.25	5.62	19.09	55.64
73.34	4.56	18.13	49.08	71.06	5.22	19.03	53.64
69.95	5.44	19.10	54.88	68.19	5.90	19.29	57.37
70.15	5.41	18.68	53.69	69.83	5.44	19.11	55.03
72.20	4.87	18.29	50.63	70.81	5.22	19.16	54.16
72.71	4.78	18.18	49.89	70.55	5.26	19.12	54.29
71.68	4.99	18.22	50.94	70.41	5.31	19.49	55.37

AFTER 3 WASHES

A-6-2S				B-6-2S			
L	a	b	YI	L	a	b	YI
65.77	6.37	19.50	60.50	71.21	5.01	18.17	51.20
67.69	5.92	18.64	56.11	70.89	5.15	18.63	52.71
67.48	6.00	18.98	57.24	70.14	5.45	18.95	54.38
68.54	5.86	19.45	57.41	69.02	5.59	18.92	55.37
68.18	5.85	19.05	56.68	69.27	5.50	18.42	53.82
66.67	6.27	19.55	59.67	70.20	5.39	19.34	55.26
67.84	5.95	18.52	55.69	71.29	5.08	18.74	52.60
67.57	6.02	19.44	58.40	70.92	5.14	18.83	53.17
69.16	5.71	19.71	57.40	68.92	5.59	18.42	54.17
69.40	5.60	19.29	56.07	68.81	5.58	18.29	53.91

AFTER 3 WASHES

A-6-3S				B-6-3S			
70.03	5.45	19.63	56.18	70.68	5.15	19.61	55.27
68.65	5.78	19.69	57.84	70.81	5.27	19.39	54.78
68.79	5.78	19.84	58.09	70.13	5.42	19.43	55.59
70.20	5.40	19.74	56.26	70.22	5.38	19.89	54.13
70.12	5.36	19.22	55.05	70.63	5.31	19.25	54.64
68.36	5.83	18.95	56.23	70.70	5.14	18.41	52.31
69.69	5.62	19.34	55.94	70.84	5.26	18.10	51.52
71.00	5.26	19.19	54.13	70.52	5.33	18.34	52.43
69.84	5.46	19.13	55.09	70.56	5.30	18.46	52.71
69.90	5.52	19.16	55.21	70.25	5.43	18.31	52.72

AFTER 3 WASHES

A-6-4S				B-6-4S			
65.35	6.35	18.79	59.02	70.60	5.18	18.45	52.35
66.36	6.20	18.39	57.11	71.61	5.04	18.13	50.84
64.09	6.66	19.04	61.22	72.30	4.86	18.44	50.92
63.73	6.85	19.18	62.16	71.86	4.82	18.62	51.59
63.59	6.78	18.94	61.58	71.60	5.03	18.39	51.45
65.62	6.23	18.35	57.45	71.17	5.12	18.54	52.24
66.51	6.04	18.35	56.50	71.35	5.11	18.54	52.11
64.68	6.49	18.55	59.11	72.21	4.92	18.56	51.32
64.79	6.56	18.78	59.76	71.61	5.06	18.70	52.25
65.19	6.43	18.70	59.01	72.40	4.86	18.42	50.80

AFTER 3 WASHES

A-6-5S				B-6-5S			
L	a	b	YI	L	a	b	YI
68.10	5.83	18.80	56.10	67.89	5.77	18.21	54.66
69.69	5.47	18.67	54.07	70.22	5.32	17.99	51.81
70.01	5.49	18.55	53.59	72.03	4.88	17.90	49.81
69.49	5.60	19.29	55.93	70.20	5.27	17.94	51.61
69.44	5.62	19.39	56.27	70.08	5.36	18.09	52.20
70.24	5.31	18.28	52.48	71.47	5.04	18.56	51.96
70.05	5.31	18.04	52.07	69.82	5.46	18.9	54.54
69.40	5.55	18.19	53.20	69.09	5.69	18.87	55.28
69.76	5.44	18.95	54.70	69.54	5.50	18.90	54.80
69.07	5.56	18.55	54.37	71.41	5.13	18.92	53.00

AFTER 5 WASHES

A-6-1S				B-6-1S			
72.35	4.68	17.82	49.19	70.73	5.12	18.35	52.07
72.63	4.77	18.01	49.50	71.60	4.93	18.29	51.12
73.23	4.63	17.63	48.03	69.37	5.54	18.90	54.95
71.35	4.02	17.92	50.44	70.93	5.13	18.79	53.03
71.55	4.98	17.67	49.66	70.99	5.09	18.40	52.00
74.15	4.28	17.78	47.42	69.32	5.44	18.65	54.27
73.43	4.53	17.53	47.53	71.72	4.88	18.40	51.23
72.29	4.84	17.75	49.17	70.64	5.20	18.42	52.41
71.41	5.10	18.18	51.13	71.38	4.97	18.49	51.80
71.38	5.06	18.04	50.77	71.61	4.95	18.65	52.01

AFTER 5 WASHES

A-6-2S				B-6-2S			
70.92	5.09	18.29	51.78	70.09	5.31	18.28	52.59
69.41	5.50	18.16	53.06	70.47	5.22	18.71	53.26
70.52	5.35	18.13	51.97	71.58	4.96	18.40	51.38
68.59	5.73	18.37	54.45	69.93	5.38	18.35	52.97
67.93	5.82	18.30	54.93	69.21	5.49	18.44	52.87
68.26	5.76	18.27	54.48	71.69	4.93	18.72	49.60
69.30	5.61	18.65	54.47	71.20	5.05	17.68	49.97
71.24	5.10	18.63	52.40	68.95	5.61	18.59	54.58
69.17	5.54	18.11	53.12	69.21	5.53	18.58	54.26
68.69	5.71	18.11	53.70	69.33	5.46	18.20	53.15

AFTER 5 WASHES

A-6-3S				B-6-2S			
L	a	b	YI	L	a	b	YI
70.95	5.10	18.79	52.98	69.95	5.32	17.92	51.79
70.08	5.36	18.82	54.00	70.74	5.15	18.22	51.91
70.08	5.30	19.32	55.17	70.27	5.32	18.73	53.61
70.73	5.14	18.79	53.18	70.81	5.15	18.61	52.68
70.05	5.33	18.96	54.35	71.11	5.03	18.55	52.17
69.70	5.48	19.43	56.00	71.41	5.00	17.35	48.99
70.19	5.34	19.37	55.27	71.45	5.02	17.11	48.39
70.63	5.23	18.74	53.22	70.53	5.27	17.76	50.94
70.59	5.23	19.02	53.94	70.79	5.15	18.31	51.95
71.38	5.00	18.97	52.96	71.32	4.99	18.33	51.42

AFTER 5 WASHES

A-6-4S				B-6-4S			
63.48	6.61	18.40	60.00	72.23	4.84	18.01	49.88
62.66	6.79	18.76	61.97	73.60	4.47	17.80	48.03
63.58	6.49	18.04	58.80	71.85	4.95	17.89	49.91
64.80	6.43	18.67	59.27	72.47	4.80	18.02	49.65
64.23	6.55	18.62	59.81	72.60	4.72	17.92	49.25
66.18	6.08	17.95	55.72	71.88	4.94	17.70	49.46
67.99	7.04	17.72	62.53	72.63	4.76	17.81	49.00
65.09	6.36	18.23	57.74	72.36	4.76	18.11	49.95
65.77	6.30	18.44	57.63	73.38	4.54	17.56	47.65
65.22	6.32	18.23	57.57	72.29	4.79	17.91	49.49

AFTER 5 WASHES

A-6-5S				B-6-5S			
70.54	5.26	18.69	53.23	68.40	5.67	17.97	53.48
70.68	5.24	18.27	52.07	70.33	5.25	17.59	50.59
70.54	5.26	18.18	51.98	72.52	4.70	17.28	47.71
68.60	5.76	19.19	56.58	72.69	4.72	17.44	48.03
69.84	5.37	18.38	53.09	71.42	5.02	17.52	49.43
69.41	5.39	17.50	51.25	71.05	5.04	18.11	51.13
69.89	5.29	17.26	50.16	70.86	5.19	18.30	51.90
70.34	5.27	18.05	51.80	69.99	5.41	18.23	52.63
69.79	5.36	18.21	52.69	69.85	5.48	18.33	53.07
70.20	5.18	17.39	50.13	71.16	5.17	18.31	51.68

Data from Kjeldahl analysis

Sample	mL HCL	mL blank	l HCl	Fabric wt.(g)	% N
B-0-4U	0.6	0.6	0.0000	1.1600	0.000
B-0-5U	0.6	0.6	0.0000	1.0739	0.000
A-0-5U	0.6	0.6	0.0000	1.0767	0.000
B-0-4W	0.7	0.6	0.0001	0.9337	0.015
B-0-5W	0.6	0.6	0.0000	1.0535	0.000
A-0-5W	0.6	0.6	0.0000	1.0537	0.000

Before 5 wash and dry cycles
(2.2% add-on)

Sample	mL HCL	mL blank	l HCl	Fabric wt.(g)	% N
A-1-2S	1.5	0.66	0.00084	1.1653	0.101
A-1-3S	1.5	0.66	0.00084	1.1517	0.102
A-1-4S	1.5	0.66	0.00084	1.2158	0.097
B-1-2S	1.7	0.66	0.00104	1.1443	0.171
B-1-3S	1.3	0.66	0.00064	0.9400	0.095
B-1-4S	1.6	0.66	0.00094	1.1334	0.116
B-1-5S	1.9	0.66	0.00124	1.1779	0.147

After 5 wash and dry cycles
(2.2% add-on)

A-1-2S	1.3	0.66	0.00064	1.0585	0.085
A-1-3S	1.6	0.66	0.00094	1.1748	0.112
A-1-4S	1.3	0.66	0.00064	0.9039	0.099
A-1-5S	1.5	0.66	0.00084	1.0764	0.109
B-1-2S	1.6	0.66	0.00094	1.1748	0.112
B-1-3S	1.4	0.66	0.00074	1.1182	0.093
B-1-4S	1.2	0.66	0.00054	0.9636	0.078
B-1-5S	1.4	0.66	0.00074	1.3059	0.079

Before 5 wash and dry cycles
(3.1% add-on)

Sample	mL HCL	mL blank	l HCl	Fabric wt.(g)	% N
A-3-2S	2.1	0.55	0.00155	1.1530	0.188
A-3-3S	2.4	0.55	0.00185	1.1814	0.219
A-3-4S	2.1	0.55	0.00155	1.1062	0.196
A-3-5S	2.5	0.60	0.00190	1.1375	0.234
B-3-2S	2.3	0.60	0.00175	1.0867	0.225
B-3-3S	2.4	0.60	0.00185	1.1814	0.219
B-3-4S	2.1	0.60	0.00155	1.1062	0.196
B-3-5S	1.9	0.60	0.00130	1.0917	0.167

After 5 wash and dry cycles
(3.1% add-on)

A-3-2S	2.1	0.55	0.00155	1.1300	0.192
A-3-3S	2.0	0.55	0.00145	1.0316	0.196
A-3-4S	2.2	0.55	0.00165	1.1838	0.195
A-3-5S	2.2	0.60	0.00160	1.3656	0.164
B-3-2S	2.2	0.60	0.00165	1.1376	0.203
B-3-3S	2.3	0.60	0.00175	1.3062	0.188
B-3-4S	2.2	0.60	0.00165	1.1700	0.197
B-3-5S	2.2	0.60	0.00165	1.1283	0.205

Before 5 wash and dry cycles
(5.7% add-on)

Sample	mL HCL	mL blank	l HCl	Fabric wt.(g)	% N
A-6-2S	2.8	0.55	0.00225	1.0934	0.288
A-6-3S	2.6	0.55	0.00265	1.0325	0.278
A-6-5S	2.3	0.60	0.00170	0.9450	0.252
B-6-2S	2.6	0.60	0.00205	1.0914	0.263
B-6-4S	3.0	0.60	0.00240	1.2844	0.262
B-6-5S	3.2	0.60	0.00265	1.3020	0.285

After 5 wash and dry cycles
(5.7% add-on)

A-6-2S	REJECT				
A-6-3S	2.4	0.55	0.00185	1.0500	0.247
A-6-5S	2.3	0.55	0.00175	1.1020	0.222
B-6-2S	2.6	0.55	0.00205	1.0400	0.276
B-6-5S	2.7	0.60	0.00210	1.1225	0.262
B-6-4S	2.3	0.60	0.00170	1.1652	0.204

Percentage add-on from nitrogen content in the fabric
before 5 wash and dry cycles
(2.2% add-on)

Sample	% N in fabric	$\frac{(\%N \text{ in fabric})}{5.58} \times 100 = \% \text{ ADD-ON}$
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(Average)

A-1-2S	0.101	
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A-1-3S	0.102	
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A-1-4S	0.097	
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A-1-5S	0.111	
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B-1-2S	0.171	2.1%
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B-1-3S	0.095	
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B-1-4S	0.116	
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B-1-5S	0.147	
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After 5 wash and dry cycles

A-1-2S	0.085	
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A-1-3S	0.112	
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A-1-4S	0.099	
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A-1-5S	0.109	
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B-1-2S	0.112	1.7%
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B-1-3S	0.093	
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B-1-4S	0.078	
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B-1-5S	0.079	
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Percentage add-on from nitrogen content in the fabric
before 5 wash and dry cycles
(3.1% add-on)

Sample	% N in fabric	$\frac{(\%N \text{ in fabric})}{5.58} \times 100 = \% \text{ ADD-ON}$
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(Average)

A-3-2S	0.188	
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A-3-3S	0.219	
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A-3-4S	0.196	
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A-3-5S	0.234	
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B-3-2S	0.225	3.7%
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B-3-3S	0.219	
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B-3-4S	0.196	
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B-3-5S	0.167	
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After 5 wash and dry cycles

A-3-2S	0.192	
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A-3-3S	0.196	
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A-3-4S	0.195	
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A-3-5S	0.164	
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B-3-2S	0.203	3.45%
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B-3-3S	0.188	
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B-3-4S	0.197	
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B-3-5S	0.205	
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Percentage add-on from nitrogen content in the fabric
before 5 wash and dry cycles
(5.7% add-on)

Sample	% N in fabric	$\frac{(\%N \text{ in fabric})}{5.58} \times 100 = \% \text{ ADD-ON}$
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(Average)

A-6-2S	0.288	
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A-6-3S	0.278	
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A-6-5S	0.252	4.85%
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B-6-2S	0.263	
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B-6-4S	0.262	
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B-6-5S	0.285	
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After 5 wash and dry cycles

A-6-2S	REJECT	
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A-6-3S	0.247	
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A-6-5S	0.222	4.3%
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B-6-2S	0.276	
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B-6-5S	0.262	
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B-6-4S	0.204	
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DP	TEN. STR. (Kgs)	ELON. %	ABR. RES. (cycl)	WARP REC. ANGLE	WEFT REC. ANGLE	WT. LOSS (3)	WT. LOSS (5)	WARP SHR. %	WEFT SHR. %
000.00	025.50	010.90	240.00	090.00	093.00	000.00	000.00	000.00	000.00
000.00	024.00	010.50	290.00	093.00	101.00	000.00	000.00	000.00	000.00
000.00	026.00	011.00	225.00	112.00	097.00	000.00	000.00	000.00	000.00
000.00	025.00	011.00	186.00	112.00	097.00	000.00	000.00	000.00	000.00
000.00	026.50	010.50	215.00	109.00	095.00	000.00	000.00	000.00	000.00
000.00	024.00	011.00	209.00	087.00	068.00	000.00	000.00	000.00	000.00
000.00	025.50	011.00	221.00	093.00	101.00	000.00	000.00	000.00	000.00
000.00	025.00	011.00	210.00	072.00	091.00	000.00	000.00	000.00	000.00
000.00	025.00	011.00	203.00	080.00	095.00	000.00	000.00	000.00	000.00
000.00	025.75	011.70	186.00	087.00	105.00	000.00	000.00	000.00	000.00

TESTING OF UNFINISHED UNWASHED FABRIC

DP	TEN. STR. (Kgs)	ELON. %	ABR. RES. (cycl) ANGLE	WARP REC. ANGLE	WEFT REC. ANGLE	WT. LOSS (3)	WT. LOSS (5)	WARP SHR. %	WEFT SHR. %
001.33	024.50	022.20	298.00 094.00	100.00	100.00	000.13	000.11	003.80	013.75
001.17	023.00	022.20	216.00 091.00	083.00	083.00	000.09	000.11	002.50	013.75
001.50	024.50	021.40	279.00 091.00	101.00	101.00	000.11	000.09	003.80	013.75
001.00	022.50	021.30	290.00 091.00	106.00	106.00	000.24	000.23	003.80	012.50
002.00	025.50	023.11	280.00 092.00	096.00	096.00	000.08	000.08	003.80	013.00
001.50	022.50	022.70	256.00 096.00	105.00	105.00	000.09	000.11	003.80	013.75
001.50	023.50	021.80	227.00 088.00	104.00	104.00	000.12	000.12	003.80	013.75
001.83	025.00	023.11	262.00 090.00	101.00	101.00	000.13	000.09	003.80	013.75
002.00	023.00	021.80	292.00 100.00	100.00	100.00	000.09	000.08	003.80	013.75
002.16	022.50	022.20	202.00 084.00	100.00	100.00	000.12	000.13	002.50	013.75

TESTING OF UNFINISHED WASHED FABRIC

DP	TEN. STR. (Kgs)	ELON. %	ABR. RES. (cycl)	WARP REC. ANGLE	WEFT REC. ANGLE	WT. LOSS (3)	WT. LOSS (5)	WARP SHR. %	WEFT SHR. %
003.16	029.50	015.70	283.00	071.00	089.00	000.01	000.00	003.00	008.00
001.50	030.50	016.57	320.00	080.00	094.00	000.04	000.06	001.50	010.00
002.50	028.00	018.60	284.00	085.00	095.00	000.03	000.04	001.50	008.50
002.50	028.50	014.40	257.00	078.00	103.00	000.02	000.02	001.50	008.50
002.67	030.00	018.30	273.00	074.00	100.00	000.02	000.09	001.00	005.00
002.00	027.00	014.80	242.00	081.00	105.00	000.05	000.05	001.00	008.50
002.17	028.00	015.26	257.00	072.00	090.00	000.03	000.04	001.00	007.00
002.00	028.50	018.28	257.00	072.00	090.00	000.02	000.02	001.50	008.00
002.50	028.50	018.10	278.00	080.00	085.00	000.02	000.04	001.00	009.00
002.50	028.50	016.60	270.00	083.00	097.00	000.04	000.05	001.50	000.50

TESTING OF FINISHED FABRIC (2.2 % ADD-ON; 5 WASHES)

DP	TEN. STR. (Kgs)	ELON. %	ABR. RES. (cycl)	WARP REC. ANGLE	WEFT REC. ANGLE	WT. LOSS (3)	WT. LOSS (5)	WARP SHR. %	WEFT SHR. %
003.13	022.50	011.30	194.00	095.00	095.00	000.10	000.09	001.00	007.00
002.67	025.00	019.10	370.00	072.00	101.00	000.08	000.02	001.50	009.00
002.67	025.00	019.10	370.00	072.00	101.00	000.09	000.08	001.50	009.00
002.57	027.50	018.30	248.00	086.00	100.00	000.02	000.02	001.00	010.00
002.00	024.00	018.30	360.00	085.00	094.00	-00.09	000.07	001.00	009.00
002.67	024.00	019.10	340.00	078.00	087.00	000.12	000.10	002.00	009.00
002.17	024.00	019.60	337.00	080.00	079.00	000.00	000.10	001.00	010.00
002.67	022.00	017.90	194.00	082.00	100.00	000.02	000.10	001.50	008.00
002.50	025.50	019.70	284.00	092.00	085.00	000.08	000.07	001.00	009.00
003.63	023.50	017.90	281.00	073.00	094.00	000.09	000.08	001.00	010.00

TESTING OF FINISHED FABRIC (3.1 % ADD-ON; 5 WASHES)

DP	TEN. STR. (Kgs)	ELON. %	ABR. RES. (cycl)	WARP REC. ANGLE	WEFT REC. ANGLE	WT. LOSS (3)	WT. LOSS (5)	WARP SHR. %	WEFT SHR. %
001.97	000.00	000.00	240.00	058.00	090.00	000.17	000.21	002.00	006.00
003.33	020.00	015.26	214.00	050.00	076.00	000.17	000.22	003.00	009.00
002.13	026.00	017.40	302.00	055.00	060.00	000.15	000.14	001.00	007.00
001.33	023.00	015.26	275.00	070.00	070.00	000.15	000.21	001.50	009.00
003.17	030.50	015.26	380.00	055.00	085.00	000.16	000.22	001.00	008.00
001.83	022.00	014.80	240.00	059.00	081.00	000.12	000.19	003.00	009.00
002.33	025.00	016.14	404.00	048.00	066.00	000.27	000.25	002.00	007.00
003.17	024.00	017.80	250.00	061.00	078.00	000.16	000.20	001.00	010.00
001.67	022.50	017.00	195.00	057.00	072.00	000.15	000.20	002.00	009.00
001.23	025.00	017.00	225.00	065.00	069.00	000.21	000.22	003.00	008.00

TESTING OF FINISHED FABRIC (5.9 % ADD-ON; 5 WASHES)

Before Wash				After 3 washes				After 5 washes			
L	a	b	YI	L	a	b	YI	L	a	b	YI
86.55	-0.05	13.62	30.30	86.05	0.36	12.72	0.00	86.39	0.35	11.79	24.40
86.42	-0.02	13.73	31.15	85.87	0.45	12.90	0.00	86.11	0.38	12.32	25.62
87.00	-0.23	13.26	33.62	86.51	0.15	12.41	0.00	86.40	0.21	12.31	25.24
86.50	-0.09	13.78	30.73	85.41	0.48	13.68	0.00	85.69	0.47	13.08	27.48
86.63	-0.08	13.43	32.20	86.10	0.34	12.58	0.00	86.24	0.36	12.09	25.07
86.56	-0.09	13.65	32.49	86.13	0.28	12.81	0.00	86.11	0.27	12.60	26.21
86.34	-0.01	13.94	31.83	85.89	0.48	13.53	0.00	85.65	0.41	12.87	26.92
86.34	-0.03	14.12	35.71	85.60	0.46	13.49	0.00	85.57	0.37	12.94	28.98
86.66	-0.09	13.77	34.41	86.06	0.28	12.93	0.00	86.31	0.24	12.35	25.50

L, a, b & YI values of finished fabric (2.2 % add-on) before wash, after 3 washes & after 5 washes

Before Wash				After 3 washes				After 5 washes			
L	a	b	YI	L	a	b	YI	L	a	b	YI
80.02	02.01	18.46	43.38	79.81	2.60	17.31	41.34	80.05	2.35	17.21	40.74
79.99	02.08	18.35	43.13	79.95	2.56	17.17	40.92	80.69	2.33	15.98	37.51
76.48	03.48	19.33	48.80	76.75	3.63	17.89	45.41	76.97	3.65	17.28	43.76
76.74	03.33	19.25	48.29	76.97	3.59	17.88	45.21	77.21	3.54	17.17	43.30
76.67	03.34	19.30	48.46	76.93	3.63	17.77	45.01	77.07	3.60	17.01	43.10
77.69	02.92	18.99	46.59	77.81	3.26	17.41	43.30	78.11	3.23	16.75	41.57
78.20	02.76	18.85	45.93	78.56	3.00	17.17	42.07	79.03	2.90	16.39	39.90
77.12	03.16	19.16	47.69	77.07	3.50	17.74	44.70	77.30	3.48	17.01	42.84
76.65	03.34	19.28	48.43	77.31	3.46	17.46	43.91	77.34	3.51	17.04	42.85
77.36	03.06	19.07	47.15	77.62	3.37	17.35	43.39	77.58	3.41	16.85	42.23

L, a, b & YI values of finished fabric (3.1 % add-on) before wash, after 3 washes & after 5 washes

Before Wash				After 3 washes				After 5 washes			
L	a	b	YI	L	a	b	YI	L	a	b	YI
72.11	04.63	19.52	53.44	71.09	4.58	17.83	51.26	72.37	4.79	17.83	49.28
70.49	05.20	19.70	55.70	69.83	5.45	19.19	55.24	70.82	5.13	18.53	52.49
68.53	05.71	19.80	58.22	67.83	5.96	19.21	57.52	69.39	5.52	18.30	53.44
69.59	05.44	19.70	57.14	70.06	5.35	18.67	53.66	70.16	5.29	18.29	52.46
69.67	05.49	20.23	58.05	69.65	5.54	19.39	56.00	70.43	5.25	19.02	54.11
70.32	05.26	19.78	56.14	70.54	5.30	18.82	53.61	70.82	5.14	18.09	51.39
64.21	06.69	19.58	62.59	64.94	6.46	18.72	59.29	64.04	6.50	18.31	59.10
71.12	05.12	19.68	55.07	71.69	5.00	18.48	51.58	72.52	4.76	17.87	49.23
69.60	05.45	19.49	56.21	69.52	5.51	18.67	54.28	69.98	5.34	18.11	52.30
70.34	05.23	19.79	56.17	70.17	5.34	18.43	52.97	70.83	5.17	17.91	50.97

L, a, b & YI values of finished fabric (5.9 % add-on) before wash, after 3 washes & after 5 washes

VITA

Srinivasan Venkataraman was born on 21 April 1959 in Madras, India. After graduating from high school, he attended Bangalore University and received a bachelor degree in Textile Technology. Before he started graduate study at Virginia Polytechnic and State University he was employed in various capacities in the textile industry.