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COTTON FABRICS.

The University of North Carolina at
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DETERMINATION OF SURFACTANT BUILD-UP
AND ITS EFFECTS ON RESIN
TREATED COTTON FABRICS

by

Uchenna Ogbogu

A Dissertation Submitted to
the Faculty of the Graduate School at
The University of North Carolina at Greensboro
in Partial Fulfillment
of the Requirements for the Degree
Doctor of Philosophy

Greensboro
1976

Approved by

[Signature]
Dissertation Adviser
This dissertation has been approved by the following committee of the Faculty of the Graduate School at the University of North Carolina at Greensboro.

Dissertation Adviser

Committee Members

October 29, 1976
Date of Acceptance by Committee

This study investigated the build-up of anionic and nonionic surfactants on untreated and resin-treated cotton fabrics and the effects of this build-up on the tensile strength, abrasion resistance and wrinkle recovery of the fabrics.

A laboratory experiment was employed for data collection, using a four-factor experimental design with two levels of each factor. The factors were fabric finish, surfactant type, laundering temperature and laundering cycle. A sample size of three was assigned to each level of the factors and three unlaundered samples of each fabric served as the control. The samples were washed for 10 minutes and rinsed for 6 minutes in an automatic home washer. Drying was carried out in an automatic home dryer for 30 minutes at a high temperature setting. Swatches of the laundered samples were extracted for three hours in distilled water and tested for residual surfactant, using the methylene blue indicator method for the anionic and the phosphomolybdic acid method for the nonionic surfactant. AATCC and ASTM standard test methods were used to test the tensile strength, wrinkle recovery and abrasion resistance of each sample. To determine the significance of the
observed changes, the data were subjected to a multivariate
analysis of covariance with the surfactant content, warp
and filling wrinkle recovery and the abrasion resistance of
the control samples serving as the covariates and the warp
and filling tensile strength, warp and filling wrinkle
recovery, abrasion resistance and surfactant content of the
laundered samples as the dependent variables. Multiple com­
parisons were made, using the Newman Keul's method, to
identify the different means where the univariate analysis
of covariance indicated a significant effect. A .05 level
of significance was chosen for the test of hypotheses.

Twelve of the 15 multivariate effects were signi­
ficant at the .05 level or less. The canonical correlations
showed that surfactant content and abrasion resistance were
responsible for most of the significance. The univariate
analysis for surfactant content yielded 12 significant
effects. Multiple comparisons of the means showed that the
untreated fabric retained a significantly larger quantity
of surfactant than the resin-finished fabric and that the
build-up of the anionic surfactant was significantly higher
than that of the nonionic surfactant. It was found that
increases in temperature and laundering cycle resulted in
increased build-up, but while temperature was important in
the build-up of the anionic surfactant, laundering cycle was
more important for the nonionic surfactant. No significant relationship was found between surfactant content and the other fabric properties. It was concluded that cotton, untreated and finished, did not retain large quantities of surfactant.
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CHAPTER I

INTRODUCTION

Textile fabrics become readily soiled in use. The process of soil removal by an aqueous solution of surface active agent is of considerable interest to both the manufacturer who is concerned with the removal of waxes and other natural impurities from textile fibers and the consumer for whom soil removal is a means of restoring the fabric to a serviceable state. Early investigations of the effectiveness of soaps in detergency included extensive research into the mode of action of surfactants. Meader and Fries described this in their well-known equation:

\[
\text{Fabric.Dirt} + \text{Soap} = \text{Fabric.Soap} + \text{Dirt.Soap}
\]

What happens to the "fabric.soap" portion of the equation


\[\text{2} \quad \text{Arthur L. Meader and Bernard R. Fries, "Adsorption in the Detergent Process," Industrial and Engineering Chemistry, 44 (July, 1952), 1632-1638.}\]
when the "dirt.soap" is flushed away in the wash water is still subject to some controversy and is the subject of this investigation.

Surfactants are characterized by the fact that they are unbalanced. A surfactant molecule consists of an oil-soluble portion (hydrophobe) and a water-soluble portion (hydrophil). This dual characteristic causes instability in water solutions which is overcome in two ways: (1) the formation of micelles and (2) orientation at the interface. The latter characteristic, known as adsorption, is responsible for detergency. The process of adsorption is further aided by fabric characteristics such as electrophoretic charge and the presence of chemically reactive substances on the fiber. Most of the surfactant is removed in the rinsing process but some is left behind and the possibility of a build-up exists. Such foreign matter, if present in large enough quantities, could affect several fabric performance properties.

This study was designed to explore the build-up of anionic and nonionic surfactants on resin-treated and

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untreated cotton, and to determine the effects of such a build-up on the tensile strength, abrasion resistance, and wrinkle recovery of the fabrics. The experimental conditions were those which take place in machine washing, using surfactants at concentrations normally used and with the regular cycle of laundering.

The adsorption of surfactant by cotton has been studied. This study, however, deals specifically with the build-up of surfactant after rinsing. During the washing cycle, there is an equilibrium between the fiber and the wash bath. This equilibrium changes with the introduction of fresh water in the rinse. This study differs from other work on adsorption in that previous research methods dealt with the distribution of the surfactant and did not consider that rinsing, as in the wash cycle, sets up a new equilibrium and reduces the adsorbed surfactant.

Although studies have pointed out the fact that desorption is not complete and that surfactant build-up could occur, few studies have undertaken to determine the extent of these phenomena. More specifically, no investigation has been undertaken into the conditions under which it occurs or the effect of such a build-up on fabric properties. Researchers have recognized that adsorbed surfactant
affects fabric properties. The practice of not using cationic surfactants in the processing of cotton, since this class of surfactants is strongly chemisorbed on cotton, demonstrates explicit recognition of this problem. Another serious handicap with the few available studies on the build-up of surfactants is the lack of statistical analysis of the data. This casts some doubts on the conclusions reached. In the absence of statistical tests, it becomes difficult to determine any real significance of the observed build-up.

Incomplete as surfactant build-up studies on cotton are, more research exists on untreated cotton than there is on resin-treated cotton. A thorough search of the literature failed to yield any studies on the desorption of surfactants by resin-treated cotton in spite of the commercial importance of this group of fabrics, and the fact that they behave differently from cotton in laundering.

Some researchers have pointed the characteristics of textile fibers which could affect adsorption. These include:

1. the presence of chemically reactive groups.

---

2. electrophoretic charge,
3. the presence of chemically reactive substances resulting from chemical action or deposition by intent, and
4. free energy forces and Van Der Waal forces and hydrogen bonding.

The durable press process on cotton involves an alteration of the chemical nature of the fiber by an introduction of chemically reactive crosslinking compounds as well as the introduction of softeners to counteract the stiffening of the fibers that result from the crosslinking. The high temperature under which curing is carried out and the presence of acidic catalysts invariably result in some degradation of the fiber. Degraded cotton differs from native cotton in its reactivity to chemical compounds. This would tend to suggest that cotton treated for durable press should adsorb and desorb surfactant differently from native cotton.

An attempt is made in this study to provide information that is lacking on the build-up of surfactant on cotton under varied conditions found in laundering as well

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as comparative information on resin-treated cotton. Untreated cotton fabrics form the bulk of the production of Nigerian cotton mills and so is of prime importance to this researcher. Although very limited quantities of cotton treated with resin to give durable press fabrics are marketed, it is of importance to establish the behavior of resin-treated fiber as a component of the polyester-cotton blend.

OBJECTIVES

The objectives of the study were: (1) to determine surfactant build-up on untreated and resin-treated cotton, (2) to determine the effects of such a build-up on the tensile strength, wrinkle recovery and abrasion resistance of the fabrics.

HYPOTHESES

The hypotheses developed for the study were:

1. There is no significant surfactant build-up attributable to the durable press finish.

2. There is no significant surfactant build-up attributable to surfactant type.

3. There is no significant surfactant build-up attributable to laundering temperature.
4. There is no significant surfactant build-up attributable to any combination of fabric finish, laundering temperature, and surfactant type.

5. There is no significant difference in fabric properties attributable to surfactant build-up.

ASSUMPTIONS

It was assumed that changes in fabric properties are detectable after three and eight laundering cycles.

LIMITATIONS

1. Some wear is expected as a result of the mechanical action of the washing machine. This should be borne in mind when interpreting the data.

2. The surfactant could be trapped between yarns because of the inefficiency of rinsing. Therefore the results of the study are applicable only to fabrics of similar construction.

3. The results of the study are limited to a pH of 5 which was the pH obtained using tap water which was delivered at an acidic pH of 5.

4. The study does not include the effect of builders which not only raise the pH but give a different system.
DEFINITION OF TERMS

Critical Micelle Concentration. A concentration at which surfactant molecules form micelles. It is in the micellar form that detergent action is postulated by some investigators as taking place.

Durable Press Fabric. A fabric that has been treated with resin to retain its smooth appearance, shape, and creases. This term is used interchangeably with permanent press fabric and resin treated fabric in this paper.

Detergency. The term refers to the process of cleaning a solid material by means of an aqueous solution of a surface active compound.

Soil. All foreign matter which is not deposited by intent on textile fabrics. Such soils generally consist of oil and finely divided solids or one of these.

Adsorption. The attraction of gases, liquids, or solids to the surface areas of textile fibers, yarns, fabrics, or any other material resulting in deposition.

Degradation. The loss of desirable physical properties by a textile material as a result of some process or some physical/chemical phenomenon.
CHAPTER II

REVIEW OF LITERATURE

The adsorption of surfactants (both soap and synthetic) on natural fibers has been studied extensively. Few of the reported studies, however, were concerned with the desorption of surfactant from cotton: rather, more emphasis has been given to this phenomenon in wool. Though the mechanism of adsorption of surfactants on cotton and wool differ, the studies on wool provided some valuable insights into possible problems in cotton and were included in this review of literature.

Studies on adsorption were reviewed only as a background to desorption. As a result, only those studies that most directly related to the problem were included.

The first part of this chapter provides a general background of detergency studies. This is followed by a review of the structure of surfactants and their role in the process of detergency. The studies related to the adsorption of surfactant are also reviewed. The chapter concludes with a review of the studies on the desorption of surfactants.
from cotton fabrics. Particular attention is given to the phenomenon of build-up which represents the affinity of the surfactant for the fabric with recognition of the importance of desorption of surfactant in the rinsing cycle.

GENERAL BACKGROUND OF DETERGENCY STUDIES

It has long been recognized that detergent action is to a large extent, surface action. As early as 1907 Spring suggested that:

... not only dispersion and emulsification, but also detergent action depend on the alteration of the hydrophobic surface of the solid being dispersed, the liquid being emulsified, or the grease or black being washed off the fibre, into a more hydrophylic or water-attracting surface.6

This occurred through the adsorption of the surfactant on the hydrophobic surface. He described the useful properties of the paraffin chains that made them ideally suited for this purpose.

The process of detergency is however, more complex than the conversion of a hydrophobic surface to a hydrophylic surface. Pickering pointed out the useful properties

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of suds in the suspension of soils in the wash liquor to prevent redeposition. Adam, using more complex equipment, came to the conclusion that the essential part of detergent action was the displacement of soil from the solid surface by an aqueous detergent solution. To this end, the contact angle formed by the detergent solution and the oil was more crucial than the lowering of interfacial tension or surface tension alone.

Though most of these early studies related what took place rather than why they occurred, they formed the background for more complex investigations. The studies also laid the foundation for the methods used in studying detergency. They established that the process of detergency consists of wetting, emulsification of liquids, lubrication, and deflocculation of soils. These processes were recognized as a result of the adsorption of surfactant molecules on hydrophobic surfaces. The ability of surfactants to do this is a result of their structure.

Since these early works, more complete explanations have been provided for the process of detergency and work

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8 Adam, op. cit, p. 125.
is still in progress to explore all the areas of this complex subject.

STRUCTURE OF SURFACTANTS

Chemical analysis of the structure of surfactants reveals that a surfactant molecule contains a hydrophobic portion and a hydrophylic portion. This combination of a water-soluble system with an oil-soluble system is responsible for the special properties of these compounds.  

Surfactants are classified into anionic, cationic, amphoteric, and nonionic surfactants, depending on the charge on the surface ion in an aqueous solution. The structure of surfactants varies with the group. Some of these groups are of less commercial importance than others so while mention will be made of these, the discussion will be centered on anionic and nonionic surfactants.

**Cationic Surfactants**

These compounds dissociate in water to give positively charged ions. They form less than five percent of the surfactants produced. Cationic surfactants may have

---

special uses in manufacturing processes, but are not used in household detergents. Their positively charged ions are adsorbed on the fiber and bind the negatively charged dirt particles closely to the fiber.\(^9\)

**Amphoteric Surfactants**

The molecules of amphoteric surfactants dissociate into positively or negatively charged particles, depending on the pH of the solution. They, therefore, behave essentially like anionic or cationic surfactants.

**Anionic Surfactants**

Anionic surfactants form the most numerous category of all surfactants. They contain groups that impart a negative charge to the surface active ions. These compounds differ from one another in the composition of their solubilizing groups. The basic pattern for anionic surfactants can be seen in the alkali salt of fatty acids (alkali soap) as shown in Figure 1.

Anionic surfactants were the first surfactants in use. Their detergent power is dependent on the length of

---

the hydrocarbon chain. Good soaps contain fatty acids with twelve to eighteen carbon atoms. The hydrophylic carboxyl group is found at the end of the hydrophobic hydrocarbon chain. Soap, however, suffered the handicap of being precipitated by water containing magnesium and calcium. It has been gradually replaced by synthetic surfactants.¹⁰

\[ C_{17}H_{35}COO^- Na^+ \]

**Figure 1**

Sodium Stearate

Synthetic surfactants substitute the carboxylic acid with mineral acids. They are found as sulfuric acid esters, sulfonic acid derivatives, and as esters of phosphoric acid. Sulfonic acid derivatives are the most important class of anionic surfactants. A member of this group, alkyl benzene sulfonate, is the most widely used surfactant for household detergents.¹¹

¹⁰*Ciba-Geigy Review*, op. cit., p. 3.

Sulfonic acid derivatives have ten to fourteen carbon atoms on a hydrophobic alkyl residue. They have the structure shown in Figure 2.

![Structure of Sodium Dodecylbenzene Sulfonate](image)

Figure 2

Sodium Dodecylbenzene Sulfonate

Anionic surfactants were found to be effective detergents for cotton when suitably built. The formulation for detergents includes not only surfactants but also builders of diverse nature such as sodium phosphates and sodium silicates. These raise the alkalinity of the system and act as sequestering agents for calcium and magnesium and they are postulated to have auxiliary action in neutralizing attractive forces between the soil and the fiber. This fact, coupled with the availability and relatively low cost of the starting materials, (n-paraffin and benzene), has made anionic surfactants commercially important. They form sixty percent of the world's surfactant consumption.  

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Nonionic Surfactants

Nonionic surfactants are the second largest group of surfactants used. They are gaining increasing importance for both domestic and industrial uses. The hydrophilic component of this group is made up of a hydroxyl group and a chain of ethoxy groups. The most important starting materials are alkyl phenol and ethylene oxide. It has the chemical structure shown in Figure 3.

\[
R \quad \quad \quad \quad OCH_2-CH_2O-(CH_2-CH_2O)_8-CH_2-CH_2OH
\]

Figure 3
A Nonionic Agent

The water solubility of this group is dependent on the ability of the oxygen in either to form a loose bond with water through hydrogen bonding links. Figure 4 explains the hydrophilic action.

In summary, surfactants are capable of wetting out, emulsifying oils, and deflocculating aggregate solids because

\[13\] Davidsohn and Milwidsky, loc. cit.
they contain a water-compatible portion and an oil-compatible portion in the same molecule. The oil-soluble portion, usually located at the end of the hydrocarbon chain for maximum detergency in an anionic surfactant, is an acid group. Nonionic surfactants lack this solubilizing group. They depend on the water-bonding ability of their oxygen atoms for their solubility. The function of the surfactants is to convert the large soil aggregates into smaller particles which are then surrounded by surfactant molecules in micellar form. This loosens the soil from the fabric and suspends it in the wash water and prevents re-aggregation and redeposition. In the case of oils and waxes, the surfactant breaks up the continuous film into small droplets which are in an emulsified form being surrounded by micelles as in the case of the solid particles.

\[
\begin{align*}
R & - O-C_2H_4 - O & \cdots & \cdots & \cdots & O & - C_2H_4 & - OH \\
\text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} \\
H & H & H & H & H & \text{.} & \text{.} & \text{.} \\
\text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} \\
O & O & O & O & O & \text{.} & \text{.} & \text{.} \\
\text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} & \text{.} \\
H & H & H & H & H & \text{.} & \text{.} & \text{.} \\
\end{align*}
\]

Figure 4

Solubilization of Nonionic Agent

SURFACTANTS IN THE PROCESS OF DETERGENCY

Detergency involves the wetting out of the substrate, adsorption at solid interfaces, emulsification, removal of soil and its dispersion in the wash solution. This process involves a complicated reaction between the fiber, soil and surfactant in aqueous solution.

Soil on clothes is composed of sand, grit, and food particles, covered by oily substances secreted by the skin. The oily coating presents a problem in laundering since laundering takes place in an aqueous medium and oil and water are incompatible. Surfactants alter this incompatibility at the contact surface. This is made possible by the tendency for the hydrophobic portion to seek an escape from water. This causes surfactant molecules to aggregate at interfaces, with the oil-compatible end outward toward the soil and the water-compatible portion in the water. This bridges the gap.


Once the fabric is wetted out, it becomes necessary to break the bond between the soil and the fabric. This is the function of the anions. The surfactant molecules which are adsorbed on the fabric and the soil give both materials negative charges. This causes them to repel each other. The soil is thus gradually removed from the fabric. Enough surfactant is left in the wash liquor to maintain the equilibrium. During the rinse cycle, a new equilibrium is established.\footnote{R. C. Aiken, "The Adsorption of Sodium Alkyl Sulphates by Wool and Other Fibres," \textit{Journal of the Society of Dyers and Colourists}, 60 (March, 1944), 60-64.} This implies that some surfactant is always left on the fabric.

Nonionic surfactant molecules do not dissociate into charged particles. They do, however, acquire the charge necessary for soil removal by their ability to form micelles. Such micelles possess a weak but effective negative charge. No useful work is attained until this tendency to form micelles is satisfied.\footnote{Harry T. Zika, "Using Nonionic Surfactants," \textit{Textile Chemists and Colorists}, 1 (July, 1969), 26-31.} The concentration at which this occurs (critical micelle concentration), is considerably lower for nonionic than for anionic surfactants. A number
of factors may result in a lowering of this concentration for this group of surfactants.\textsuperscript{19}

\textbf{ADSORPTION OF SURFACTANTS BY TEXTILE FIBERS}

\textbf{Effect of Substrate on Adsorption}

It has been fairly well established that the adsorption of surfactants on fabrics occurs in two main forms. The first is the formation of a chemical bond. Such a reaction is irreversible. This occurs in wool and other amine-containing fibers. The second is of a physical nature. Such surfactant is removed more easily from the fabric. This characterizes the adsorption of surfactant on native cellulose.

Cellulose is a polymer containing anhydrous glucose molecules in a linear chain of high molecular weight. Unlike wool or nylon, it does not contain polar groups capable of forming ionic bonds. However, cellulose has an oriented structure in which the polymer molecules exert free energy forces from Van Der Waals forces and hydrogen bonding. These free energy forces are also available for holding other chemical compounds. The importance of these forces

\textsuperscript{19}Aiken, op. cit., p. 60.
is more readily seen in dyeing. Thus direct dyes are substantive to cotton. Their affinity and resistance to loss in washing is due to the attractive forces of the cellulose molecules for the free energy forces of the benzene rings and the hydroxy and amino groups of the dye molecule. This affinity is sufficient to cause exhaustion of the dye from the dye bath. However, on subsequent washing, new equilibrium is obtained so that the dye is lost to the wash water.  

Surfactants contain benzene rings and, at least in the case of the nonionics, the opportunity for hydrogen bonding exists. The attractive forces may not be as strong as that of dyes but could still be strong enough to account for adsorption and some degree of build-up.

Aiken conducted a study on the adsorption of sodium alkyl sulphate on wool and other fibers. Adsorption was measured by determining the amount of surfactant in a 100 ml surfactant solution before 2 gms of fabric sample was added and 24 hours after sample immersion. It was found that wool adsorbed more surfactant than cotton or nylon.

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To determine the mode of adsorption, another series of experiments using wool, silk, nylon and cotton was conducted. The first three fibers were polyamides. A decrease in adsorption with decreasing numbers of amino groups was reported. To verify the role of amino groups, the adsorption on wool in the presence of hydrochloric acid was measured. This broke up the salt linkages with the formation of carboxy groups and an ionized ammonium type radical associated with the chlorine ion. The ammonium ion can be readily displaced by the alkyl sulphate ion. Aiken reported an increase in adsorption.  

Aiken's study showed rather conclusively that adsorption on polyamides was of a chemical nature. It was, however, not so detailed on the mechanism of adsorption on cotton. It was merely pointed out that the adsorption mechanism was of a different nature. The weakness of this study was the number of samples used. One has the impression that the conclusions were reached on the basis of a single sample. Studies by other workers, however, seem to support his findings.

\(^{21}\)Aiken, loc. cit.
Lambert, using more sophisticated techniques, produced data which confirmed the idea that characteristics of the substrate affect adsorption. Though the investigation was primarily concerned with developing the radio tracer technique as a means of studying detergency, the results produced are of interest in the present study.

The study used radio-active calcium as an indirect means of measuring surfactant adsorption by used and unused cotton fabrics. It was found that used cotton adsorbed more surfactant than new cotton. He attributed this to the chemical changes in the fiber which resulted in an increase in the number of the carboxyl groups as well as in the amorphous areas of the fiber. A lengthy discussion of the results was not presented since this was not his concern. This technique has since proved a useful means of studying detergency.

Meader and Fries, using the radio tracer technique conducted a comprehensive study of the adsorption of alkyl benzene sulphonate by wool and cotton fabrics. The alkyl benzene sulphonate was prepared with radioactive sulphur.

Fabric swatches were soaked in surfactant solutions of predetermined radioactivity, for 24 hours (the length of time was greater for wool). The radioactivity of the blotted samples was measured.

They observed, among other things, that the rate of adsorption was greatest in the first ten minutes. The adsorption isotherm for cotton showed an almost vertical climb in the first ten minutes and then levelled off. The isotherm for wool was different. It showed the initial rapid climb as was obtained for cotton, then the curve became more gradual, lasting over a much longer period. The initial rapid rate represented the physical adsorption in both fibers. The more gradual adsorption rate in wool represented a chemical reaction. Their results confirmed Aiken's findings.

The foregoing studies showed that the substrate was an important consideration in adsorption and that adsorption on cotton was of a physical nature. These authors did not go into the details of the mechanism involved.

The nature of surfactant adsorption on cotton was studied by Schwarz et al., using radio-active dodecyl

\(^{23}\)Meader and Fries, op. cit., p. 1648.
benzene sulphonate. A 0.5 percent surfactant solution was heated to 51 °C, and a count of the specific activity was taken. Cotton swatches that had undergone the following treatments were used:

1. commercially bleached and finished cotton,
2. commercially bleached and finished cotton, treated to remove metallic ions,
3. commercially bleached and finished cotton, treated to remove metallic ions and further treated with sodium chloride.

The washing lasted for ten minutes in a terg-o-to-meter. Rinsing, when it was employed, was carried out in the same equipment for the same length of time. The samples were wrung dry and a count of their specific activity was taken. From their observations, the authors postulated that polyvalent cations were responsible for the adsorption of anionic surfactants on cotton.

In a second article, the authors established the role of calcium in adsorption on cotton. A 0.5 percent surfactant solution was prepared from artificially hardened

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water. The fabric swatches were washed in the solution and wrung dry. Another set of swatches was washed in water which had been hardened with radiocalcium. The surfactant bound calcium was determined by subtracting the quantity of calcium adsorbed in the absence of surfactant from that adsorbed in the presence of surfactant. The study also investigated the build-up of surfactant on cotton. It was concluded that surfactant adsorption was in the form of calcium dodecylbenzene sulphonate salt.25

This study is of special merit because the washings were carried out under conditions that were close to household laundering conditions. Secondly, the precision of the radio tracer technique and the number of replications involved leave no doubt as to the validity of conclusions.

Other studies have also shown that impurities in cotton could affect the adsorption of surfactant. Ginn and co-workers, using dewaxed and natural cotton showed that surfactant was adsorbed on the wax. This study employed an indirect method in determining the amount of surfactant

adsorbed. Conditioned samples were introduced into a surfactant solution and stirred for ten minutes. The liquid was decanted and the quantity of surfactant left in the solution was determined. The two-phase method was used for the anionic surfactant and the methylene blue extraction method was used for the cations. The adsorbed surfactant was calculated using the formula:

\[
mg \text{ sorbed/gm cotton} = \frac{\% \text{ conc. x flask sol. vol x 1000}}{gm \text{ cotton x cotton % solids}}
\]

It was shown that wax-containing samples adsorbed more surfactant than the dewaxed samples. Adsorption on the latter was negligible. It was concluded that the hydrophobic surface of the wax was responsible for adsorption. At critical micelle concentration the wax solubilized, leaving a wax-free surface which resulted in a decline in adsorption.\(^{27}\)

This is a plausible explanation and still does not dispute the fact that hardness minerals in water affect adsorption. Instead, it supports the view that adsorption

\(\text{27 Ibid.}\)

of surfactant on fabrics is a complex process which is
affected by many things. Among these are soil, natural
impurities and polyvalent cations.

The absence of statistical analysis was clearly
evident in these studies. However, Ginn and others attempted
to correlate adsorption with fiber swelling. The data were
so variable that no conclusions could be reached. This
raises the question of whether these kinds of data lend
themselves to statistical analysis. This shortcoming may
weaken individual cases, but there is such a consistency in
the reported findings that one may safely conclude that
characteristics of the substrate are an important factor in
adsorption.

These studies and others also pointed out other
factors that could affect adsorption. These include the
concentration of the surfactant, the laundering temperature,
the class of the surfactant, and the pH of the wash liquor.

Effect of Surfactant Concentration
on Adsorption

Researchers seem to agree that adsorption increases
with surfactant concentration to a point that corresponds

28
Ginn, Kinney, and Harris, op. cit., p. 138.
to critical micelle concentration. Beyond this point, there is no agreement on the effect of surfactant concentration.

Aiken, in the study discussed in the previous section, showed that adsorption increased with surfactant concentration up to a point that corresponds with critical micelle concentration, then declined. After a temporary minimum, another climb was started and maintained. The slope of the second increase was, however, more gradual than that of the initial increase. This pattern was attributed to a fluctuation of the single ions. Single ions were involved in adsorption. At critical micelle concentration their number was reduced. As more of these ions were removed from the solution after critical micelle concentration, the equilibrium between the micelles and the single ions was upset and more of these were released to restore the equilibrium. This increase in the number of the single ions led to another increase in adsorption.

Rose and others, using carbon black as a substrate, showed that adsorption increased with increasing surfactant concentration, up to critical micelle concentration. They, however, failed to observe the cubic trend reported by Aiken. They reported a levelling of adsorption at critical micelle
concentration. These differences could probably be attributed to the differences in the substrates used. Carbon black, being chemically inert, would probably react differently from wool or cotton. The use of different substrates has been blamed for many of the inconsistencies found in detergency studies.

Weatherburn and Bailey also reported studies on cotton in which the decline at critical micelle concentration was absent. They found that for the anionic and non-ionic surfactants, adsorption increased up to critical micelle concentration and then levelled off. Adsorption of the cationic surfactants, however, continued to increase even after the other had levelled off. They attributed the levelling off of the anionic and nonionic surfactants to the formation of micelles. Micelles have no hydrophobic-hydrophilic properties so are not involved in adsorption.


Cationic micelles possess a positive charge and so have electrostatic attraction.  

The explanation of the behavior of cationic compounds is not entirely in agreement with the views of other researchers. Zika stated that nonionic micelles possess a weak but effective negative charge. In such a case the nonionic surfactant would be expected to behave in a similar manner to the cationic surfactant used in the study.

Meader and Fries observed peaks in their adsorption isotherms. They, however, stated that the point at which the peaks occurred was greater than critical micelle concentration in all cases.

Flett and Walter reported sharp increases in adsorption with small increases in concentration up to .2 percent. At this point, small increases in concentration caused a sharp drop in adsorption till a minimum was reached at a


33Meader and Fries, op. cit., p. 1368.
concentration of .4 percent. Another increase started after this and exceeded the first maximum.\textsuperscript{34}

It was not possible to compare this study with the other studies reported since no indication was given of the critical micelle concentration of the surfactants used. No explanation for the observed maxima was provided and, on the whole, the details of the study were not clear.

Ginn et al. observed drops in adsorption at around critical micelle concentration. It was attributed to a decrease in the single ion concentration. The drops were evident in both the built and unbuilt surfactants. While the built surfactant showed a rise after a temporary minimum, as observed by other investigators, the unbuilt surfactant levelled off at the minimum.\textsuperscript{35}

In summary, there is agreement on the fact that adsorption is affected by concentration. There is, however, no consensus on the details of this effect. The available evidence has established that initial increases in

\textsuperscript{34}L. H. Flett and J. Walter, "Quantitative Data on the Adsorption of Detergents by Cotton Sheeting Under Textile Processing Conditions," \textit{American Dyestuff Reporter}, 41 (March, 1952), 139-143.

\textsuperscript{35}Ginn, Kinney, and Harris, op. cit., p. 138.
concentration result in increased adsorption. At concentrations around critical micelle concentration, a change occurs in the adsorption isotherm. There is no agreement on the direction of this change. The studies suggesting a drop and then a rise after a temporary minimum seem to have employed more rigid controls and used more valid techniques.

Effect of Temperature on Adsorption

The temperature of the wash bath has been shown to be an important factor in adsorption. As with concentration, there is no agreement on the exact effect of this factor.

Aiken showed that increases in temperature resulted in slight increases in adsorption. It also resulted in a decrease in the concentration at which the change in the adsorption isotherm occurred. 36

This information appears contradictory to his explanation of the break in the isotherm. If the break was a result of micelle formation and temperature lowered the concentration at which the break occurred, then, in essence, the critical micelle concentration was being lowered.

36 Aiken, op. cit., p. 64.
Rose and others reported findings which contradict Aiken's findings. They found that adsorption increased with temperature within the range of 30 C to 70 C. Above 70 C, adsorption decreased with increase in temperature. The concentration at which the break in the curve occurred increased slightly with increasing temperature, within the temperature range given above. 37

Flett and others produced data in support of this. They showed increased adsorption between the temperature of 80 F and 180 F. Beyond this point, adsorption dropped substantially. 38

Meader and others found that increases in temperature affected the adsorption of surfactant only when certain builders were present. Temperature had no effect on surfactant alone. 39

There appears to be quite a bit of contradictory evidence regarding the effect of temperature. One can safely conclude that temperature affects adsorption. It is impossible to state the manner of this effect from the available literature.

37 Rose, Weatherburn, and Bailey, loc. cit.
38 Flett and Walter, op. cit., pp. 139-143.
Effect of Surfactant Class on Adsorption

Few studies were found which reported the adsorption of different classes of surfactant on cotton. Those found presented contradictory views.

Weatherburn and Bailey investigated the adsorption of anionic, cationic and nonionic surfactants on a group of fibers. It was found that nonionic surfactants were absorbed the least while the cationic surfactants had the highest adsorption. Cotton absorbed more nonionic surfactant than it adsorbed the other categories. This result is contrary to what is known of the behavior of cationic surfactants on cotton. It cannot, however, be blamed on experimental conditions since these were rigidly controlled.

Ginn et al. employed the same classes of surfactant in their study. Their data showed that wax-containing cotton adsorbed more anionic surfactant than the other two classes of surfactant. The dewaxed cotton on the other hand, absorbed more of the cationic surfactant.

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40 Weatherburn and Bailey, op. cit., p. 797.
41 Ginn, Kinney, and Harris, op. cit., p. 138.
Summary

Though opinion is still divided on various aspects of adsorption of surfactant on textile fibers, there is agreement on at least four points:

1. Adsorption of surfactants on textile fibers is of both chemical and physical nature.

2. Adsorption of surfactants on native cotton is due in part to polyvalent cations which bind the surfactant molecule to the fiber.

3. The quantity of surfactant adsorbed by cotton increases with increases in concentration up to critical micelle concentration when a change occurs in the adsorption isotherm.

4. Temperature has a yet undetermined effect on adsorption.

The points on which more information is required can be summarized as:

1. the effect of temperature on the adsorption of surfactants by cotton,

2. the direction of change in the adsorption isotherms for the effect of concentration,

3. the class of surfactants most adsorbed by cotton.
These studies were not concerned with the desorption of the adsorbed surfactant from the fabric. A few studies have suggested that some relationship may exist between these two aspects of detergency.

BUILD-UP OF SURFACANT ON COTTON FABRICS

Few studies have been concerned with the build-up of surfactant on cotton. Each of the few available studies was concerned with a different aspect of surfactant build-up so the conclusions reached by individual researchers have not been verified by the works of others.

Schwarz et al. investigated the build-up of surfactant on fabric as part of the study reviewed. The study was conducted using:

1. commercially bleached and finished cotton;
2. commercially bleached and finished cotton, treated to remove metallic ions;
3. commercially bleached and finished cotton, treated to remove metallic ions and further treated with sodium chloride.

A build-up of surfactant which reached a maximum on the third laundering for the commercially bleached and finished
cotton was reported. After further investigations, it was noted that adsorption took place as a calcium dodecyl-benzene sulphonate salt. It was also shown that at the same time as the anionic exchange, cation exchange was occurring between the bath and the cotton. Some of the calcium was being replaced by sodium. This sodium salt of cotton carboxylic acid, being monovalent, was incapable of binding the surfactant to cellulose. This accounted for the maximum.

Meader and Fries observed that all the surfactant adsorbed on the fabric was not washed off. After two hours of rinsing, cotton swatches that had been soaked in surfactant solution for twenty-four hours still retained ten percent of the adsorbed surfactant. After forty-eight hours of rinsing, two percent of the surfactant was still left on the fabric. It was also found that distilled water removed more surfactant than hard water. This shows that equilibrium exists but that rinsing reduces the quantity of surfactant adsorbed. It also shows that the surfactant is not held very strongly.

\[42\] Schwarz, Martin, Ruthowski, and Davis, op. cit., p. 37.

\[43\] Schwarz, Martin, and Davis, op. cit., p. 1.

\[44\] Meader and Fries, op. cit., p. 1636.
This result also lends strength to the argument that hardness minerals account for some of the bound surfactant. In addition, the authors reported that the initial concentration did not affect the rate of adsorption.

Maple investigated the build-up of surfactant on laundered cotton fabrics. He was primarily concerned with the effectiveness of the rinse cycle of the automatic household washer. In a preliminary experiment, cotton swatches were soaked in a one-percent solution of linear alkyl benzene sulfonate. It was found that twenty-five percent of the surfactant remained on the fabric after rinsing. In a follow-up experiment simulating household conditions, he found that after forty laundering cycles in a household washer, .05 mg of the surfactant was retained per gram of fabric. Maple also reported that less surfactant was retained as the temperature increased from 60 F to 100 F.

In summary, the few available studies on the build-up of surfactant on cotton agree that:

1. The surfactant used in laundering is not completely rinsed off and a build-up occurs over several launderings.

\[45\] Maple, op. cit., p. 505.
2. This build-up is caused by polyvalent cations which are chemisorbed on the cotton fabric. These cations bind the surfactant molecules to the fiber.

There are, however, areas where more information is required. The reported studies have not clearly specified the effect of temperature on the build-up of surfactant, and the effect of surfactant concentration still needs to be explored.
CHAPTER III

PROCEDURE

The purpose of this study was to investigate the build-up of anionic and nonionic surfactants on untreated and resin-treated cotton fabrics and to determine the effects of such a build-up on the tensile strength, wrinkle recovery and abrasion resistance of the fabrics. Analytical methods were used to determine residual surfactant after repeated launderings. The experiment was conducted in the laboratory under conditions obtained in the regular cycle of machine washing and surfactants were used in normal concentrations.

Eighty yards of cotton twill with a thread count of 120 x 44 were obtained from the fabric manufacturer. Forty yards of this fabric had been scoured, bleached and mercerized and the remaining forty yards had been further treated with glyoxal, a resin used for durable press performance, and cured. Tests were conducted to confirm the fiber content and to determine: (1) the extent of cross-linkage and curing, (2) fabric weight and (3) percent resin
on the fabric. The fabric was cut into smaller widths and laundered in an automatic home washer and dryer using unbuilt anionic and nonionic surfactants. After three and eight laundering cycles the fabrics were analyzed for surfactant content by accepted chemical procedures. The tensile strength, wrinkle recovery and abrasion resistance of the fabrics were also tested using standard ASTM test methods. Multivariate analysis of variance was used to determine the significance of the observed changes.

PRELIMINARY TESTS

Tests were made to confirm the fiber content of the fabrics obtained from the manufacturer, to determine the extent of crosslinking and curing, the weight and resin content of the fabric.

Fiber Identification

The fiber content of the fabrics was determined using the AATCC test method 20-1973. Microscopical examinations and solubility tests were performed as stated in the test method. 46

**Determination of the Extent of Crosslinkage and Curing**

The extent of crosslinkage and curing was determined using the Cone Microscopic method. In this procedure, the yarn is untwisted and the fibers feathered out by using a pick needle. The fibers are spread out on a dry slide and covered with a cover glass. The microscope is focused on the dry longitudinal mount at a 150 X magnification. A few drops of cupriethylene diamine (cuen) are applied to the edge of the cover glass and allowed to flow into the mount. Observed changes are recorded immediately and after fifteen minutes in cuen. The observations are interpreted as follows:

1. Immediate degradation indicates the absence of a crosslinking resin.
2. A. Rapid initial swelling and slight degradation after 15 minutes indicates a resin either with low crosslinking properties or one that has been dried but not cured.
   B. Cure a new sample and retest. If fibers show no initial reaction to the solvent this confirms a good finish capable of being post cured and crosslinked. If results are the same as in A, the resin has little crosslinking properties.
3. A. Slow initial swelling and fiber deconvolutions and no degradation after 15 minutes indicate a resin with some crosslinking and part curing.
   B. Cure a new sample and retest. If fibers show no initial reaction to the solvent it confirms an incompletely cured fabric. If results are the same as in A, the resin has only moderate crosslinking properties.
4. No initial swelling and only moderate irregular swelling after 15 minutes indicate a well cured and good crosslinking finish.\textsuperscript{47}

**Percent Resin on the Fabric**

The method recommended by Mitzner was used to determine the amount of resin on the fabric. A preweighed oven dry sample is heated for the specified time in .1N hydrochloric acid at a temperature of 65°C. This results in an acid hydrolysis of the resin. The resin-free sample is washed and reweighed. The resin content is calculated as a percent of the dry weight of the extracted sample using the formula:

\[
\% \text{ resin (material removed by hydrolysis)} = 100 \left( \frac{Wm - w}{Wm} \right)
\]

Where

- \( M \) = weight of the moisture sample
- \( m \) = weight of moisture sample after oven drying
- \( W \) = weight of original fabric before hydrolysis
- \( w \) = weight of original fabric after hydrolysis

\textsuperscript{47}"Qualitative Analysis for Crosslinkage and Curing of Resin Treated Cellulosic Fibers," Cone Mills R & D Laboratory Microscopic Methods, 1966.

Fabric Count

The ASTM test method D 1910-64, (reapproved 1970), was used to determine the fabric count. The test method involves the counting of the number of warp yarns (ends) and filling yarns (picks) per inch of fabric. An Alfred Suter yarn counter was used. Since the fabric was a closely woven twill, the samples were ravelled down for one inch before the fabric count was made for greater accuracy.

Fabric Weight

The determination of the fabric weight was made as specified in the ASTM test method D 1910-64, (reapproved 1970). The sections applicable to narrow fabrics, sections 40 and 41 were used. No allowance was made in the calculations for the selvedge. The results of these tests are shown in Table 1.

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Table 1

Results of Preliminary Tests

<table>
<thead>
<tr>
<th>Fabric Weight</th>
<th>7 oz/sq yd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent Resin</td>
<td>6%</td>
</tr>
<tr>
<td>Extent of Crosslinkage and Curing</td>
<td>Fully Crosslinked and Cured</td>
</tr>
</tbody>
</table>

LAUNDERING OF SAMPLES

Samples of unfinished and resin treated cotton fabrics were laundered at temperatures of 105 °F and 130 °F, using anionic and nonionic surfactants. Half of the samples within each temperature and surfactant groups were laundered three times while the other half were laundered eight times. Three unlaundered samples from each class of fabrics served as the control.

Preparation of Laundering Samples

The untreated and resin-treated fabrics were cut into 36 x 45 inch widths. A code was inserted in the selvedge of each sample with a laundry marker. The selvedge was not included in any of the chemical or physical tests. The samples from the resin-treated fabric were sorted into four different loads to be washed with different surfactants and
at different temperatures. Each load contained six samples, three of which were laundered three times and the other three laundered eight times. The loads were as follows:

1. anionic surfactant group washed at 105 F
2. anionic surfactant group washed at 135 F
3. nonionic surfactant group washed at 105 F
4. nonionic surfactant group washed at 135 F.

The untreated fabric samples were sorted into corresponding loads. Each load weighed 4½ lbs. A table of random numbers was used to assign the order of washing of the loads within each laundering cycle.

**Laundering Procedure**

Since the experiment was designed to simulate service conditions as closely as possible, the fabric was not prewashed to remove excess finish. The fabric would therefore contain, at the start, adsorbed surfactant residual from the scouring process. The resin treated fabric will contain some nonionic surfactant as a constituent of the resin system, catalyst, and polyethylene softeners. Regular tap water of medium hardness, 38 parts per million, was used. The temperatures of 105 F and 135 F chosen for the study represent temperatures used for hand wash and hot wash
in the home setting. Unbuilt anionic surfactants were used. The anionic surfactant was a dodecylbenzene sulphonate of the structure widely used in household detergents. It contains small quantities of sodium sulphate but is close to 100 percent active. It has the formula given in Figure 5.

![Figure 5](image)

**Sodium Dodecyl Benzene Sulphonate**

The nonionic surfactant was Triton X 100 (Rohm and Haas). This is an ethylene oxide condensation product with t-octylphenol. It is essentially 100 percent active and has the formula shown in Figure 6.

![Figure 6](image)

The household detergent formulation contains fluorescent whiteners, sequestering agents, phosphates and anti-redeposition compounds. Since the objective of this research was to examine the behavior of the surfactants, the presence of these additional materials would not
necessarily interfere with adsorption but could introduce interferences in analytical procedures.

\[
\begin{align*}
\text{CH}_3 & \text{C} & \text{CH}_2 & \text{C} & \text{(OCH}_2 \text{CH}_2 \text{)}_9 \text{OH} \\
\text{CH}_3 & & \text{CH}_3
\end{align*}
\]

Figure 6
Alkyl Phenoxy Polyethoxy Ethanol

The washing machine was set to deliver 13 gallons of water. The water was adjusted to the desired temperature. Fifty grams of surfactant were added to the wash water, to give a surfactant concentration of 0.08%. The pH of the wash bath as taken with a pH paper was 5. The washer was set for a washing time of ten minutes and a rinsing time of six minutes. The samples were added and the washer was allowed to proceed to the final spin. The fabrics were removed immediately and tumble dried for 30 minutes at a high temperature setting. After three laundering cycles half of the total number of samples from each of the eight loads

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was withdrawn for testing. The other half was laundered eight times before testing. Analytical methods for determinations of surfactant was carried out on each of the laundered sample as well as the unwashed samples which served as the control. The samples were also subjected to standard tests for abrasion resistance, wrinkle recovery and tensile strength.

ANALYSIS OF SURFACTANT CONTENT

The laundered samples were extracted with distilled water and analyzed for surfactant content. Each sample was analyzed for the particular class of surfactant in which it was laundered. The control samples were analyzed for both the anionic and nonionic surfactants.

Extraction

Anionic surfactant. A nine-inch square sample of approximately 11.12 gm was cut from each laundered sample and dried to constant weight. The samples were allowed to cool in a dessicator before being weighed on an analytical balance. The samples were prepared and extracted in a soxhlet extraction equipment as recommended by Maple, using 200 cc of distilled water. Each sample was extracted for
three hours. A sintered glass crucible rather than the Buchner funnel was used to filter the hot extract. The methylene blue indicator method was used for quantitative determinations.

**Titration of Extract**

Two solutions were prepared for the titration of the extract and the known concentration of surfactant used as a control.

1. Methylene Blue Indicator Solution. A one-tenth gram methylene blue was dissolved in 100 ml of distilled water. Thirty milliliters of this was transferred to a 1-liter volumetric flask. Five milliliters of distilled water, 6.8 ml of concentrated sulphuric acid and 50 gms of sodium biphosphate, were added. The solution was diluted to one liter and mixed thoroughly.

2. CTAB Solution. Cetyltrimethylammonium bromide, 1.8 gms was transferred to a 1-liter volumetric flask and made up to one liter with distilled water.

A 50 ml aliquot of the extract was pipetted into a 100 ml graduated cylinder. Twenty-five milliliters of

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52 Maple, op. cit., p. 505.
methylene blue indicator solution and 15 ml of chloroform were added. The solution was titrated with CTAB solution to the correct end point. The correct end point was taken at the point where there was a change from a reddish-blue chloroform layer, greenish-blue water to a reddish-blue water, greenish-blue chloroform.

A solution containing a known amount of surfactant was titrated in the same manner as the extract. This established the ratio of CTAB solution to surfactant. 53

The quantity of surfactant retained per gram of fabric was calculated using the formula:

\[
\text{mg surfactant/gm fabric} = \frac{%\text{conc.} \times \text{flask sol. vol.} \times 1000}{\text{gm cotton} \times \text{cotton % solids}}
\]

**Determination of Nonionic Surfactant**

The extraction of the samples was carried out as described for the anionic surfactant, using distilled water. The dissolved surfactant was precipitated by the phosphomolybdic acid method described by Oliver and Preston. Three solutions were prepared for the precipitation.

53


54 Ginn, Kinney and Harris, op. cit., p. 139.
1. Hydrochloric Acid Solution. One volume of concentrated hydrochloric acid was diluted to four volumes with distilled water.

2. Barium Chloride Solution. Ten grams of barium chloride was dissolved in 100 ml of distilled water.

3. Physphomolybdic Acid Solution. Ten grams of phosphomolybdic acid was dissolved in 100 ml of distilled water.

One hundred milliliters of the extract were pipetted into a 250 volumetric flask to which was added in the following order: 5 ml each of hydrochloric acid, barium chloride, and phosphomolybdic acid solutions. The content of the flask was made up to 150 ml with distilled water. The yellowish-green precipitate was flocculated by raising the mixture to the boil. The flask was covered and allowed to stand for 18 hours. The precipitate was filtered through a tarred no. 4 sintered glass crucible, previously heated at 100 °F for 15 minutes and cooled to room temperature in a dessicator. The precipitate was washed in a minimum of 100 mls of distilled water and dried to a constant weight.

A solution containing a known amount of surfactant was precipitated in the same manner as the extract. This
established the ratio of weight of complex to the weight of detergent.  

The amount of surfactant retained per gram of fabric was calculated as in the anionic surfactant.

PHYSICAL TESTS

Abrasion Resistance

The samples were tested for abrasion resistance using the ASTM test method D 1175, Rotary Platform Double Head method. The Taber Abraser was used along with carborundum wheels with a pressure of 500 gms.

This method measures the abrasion resistance of the fabric by subjecting the fabric sample to a rotary rubbing action under controlled conditions of pressure and abrasive action. The abraded portion was not large enough to permit the measuring of the braking strength so the number of cycles at the appearance of the first hole was reported. Although the reporting of the number of cycles at the appearance of the first hole is not a precise method, it is


the most highly recommended of the four methods suggested for research purposes.\textsuperscript{57}

The wheels were resurfaced after every five samples by abrading a carborundum-coated paper of medium coarseness for ten cycles. The resurfacing disk was discarded after every six resurfacings.

**Tensile Properties**

The tensile strength of the samples was tested as specified in the ASTM test method D 1682, (reapproved 1970), Ravel Strip method. The Scott Tester Model J was used. This pendulum-type strength tester operates on the constant rate of elongation (CRE).

The test method measures the breaking load and elongation of textile fabrics by continually increasing the load applied in the longitudinal direction until the sample ruptures.\textsuperscript{58} It is recommended that when possible, samples that differ in only one respect should be used for comparison since this method measures the minimum rather than the


\textsuperscript{58} 1974 *Book of ASTM Standards*, op. cit., p. 306.
average strength of the samples. Under these conditions the test method gives precise results.

Wrinkle Recovery

The wrinkle recovery of the samples was determined as stated in the AATCC test method 66-1972. The test involves the creasing of the fabric sample under controlled conditions of time and load. The creased sample is suspended from the instrument for a five-minute recovery period after which the recovery angle is measured.

The data obtained from the tests were subjected to a multivariate analysis of covariance to determine the significance of the changes in fabric properties. The surfactant content, warp and filling wrinkle recovery, and abrasion resistance of the control samples were used as covariates.

59 Skinkle, loc. cit.

STATISTICAL PROCEDURE

A factorial experimental design was employed for the analysis of the data. The levels of the four factors involved were:

Surfactant - Anionic and Nonionic
Finish - Untreated and Resin Treated
Temperature - 105 F and 135 F
Laundering Cycle - 3 and 8 Cycles.

A sample size of three samples per cell was used. Un laundered untreated, and resin-treated fabrics were used as control. The data thus collected was subjected to a multivariate analysis of covariance with surfactant content, warp and filling tensile strength, warp and filling wrinkle recovery, and abrasion resistance as the dependent variables. The warp and filling wrinkle recovery, abrasion resistance and surfactant content of the control samples served as the covariates. The Roy's Maximum Root Criteria obtained were tested for significance using the table of Greatest Characteristic Root Distribution provided by Harris. Where the univariate analysis of covariance indicated a significant

---

effect, multiple comparisons were made to determine which of the means differed using Newman Keul's method.
CHAPTER IV

PRESENTATION OF DATA

The objectives of the study were to determine the build-up of anionic and nonionic surfactants on untreated and resin-treated cotton fabrics and to ascertain the effects of this build-up on the tensile strength, wrinkle recovery, and abrasion resistance of the fabrics. To this end, data were collected in a laboratory experiment, using a factorial experimental design. The four factors and the levels involved were:

1. fabric finish - untreated and resin treated
2. temperature - 105 F and 135 F
3. surfactant - anionic and nonionic
4. laundering cycle - three and eight.

Unlaundered fabric samples served as the control. The dependent variables were surfactant content, warp and filling tensile strength, warp and filling wrinkle recovery, and abrasion resistance.

Multivariate analysis of covariance was used to determine the significance of the observed differences. A
5% level of significance was chosen for the test of hypotheses. Where the univariate analysis of covariance showed a significant effect a multiple comparison was made, using Newman Keul's method, to determine which of the means differed.

These data are presented in the following order:

1. multivariate analysis of covariance
2. univariate analysis of covariance
3. correlation between surfactant content and the other dependent variables.

MULTIVARIATE ANALYSIS OF COVARIANCE

Multivariate analysis of covariance was performed on the six dependent variables, surfactant content, warp and filling tensile strength, warp and filling wrinkle recovery, and abrasion resistance. The surfactant content, warp and filling wrinkle recovery, and abrasion resistance of the control samples were used as covariates. The result of the analysis are shown in Table 2.

All but two of the multivariate effects were significant at the .05 level. The canonical weights revealed that mainly surfactant content accounted for the significance of the analyses for surfactant and finish. Abrasion
# Table 2

Multivariate Analysis of Covariance Summary Table

<table>
<thead>
<tr>
<th>Source</th>
<th>Roy's Max. Root Criterion</th>
<th>Deg. of Freedom</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Covariates</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Surfactant Content</td>
<td>0.26</td>
<td>1</td>
<td>28</td>
</tr>
<tr>
<td>Warp Wrinkle Recovery</td>
<td>.23</td>
<td>1</td>
<td>28</td>
</tr>
<tr>
<td>Filling Wrinkle Recovery</td>
<td>.18</td>
<td>1</td>
<td>28</td>
</tr>
<tr>
<td>Abrasion Resistance</td>
<td>.38</td>
<td>1</td>
<td>28</td>
</tr>
<tr>
<td>Surfactant</td>
<td>1.07</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Finish</td>
<td>.98</td>
<td>6</td>
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<td>Temperature</td>
<td>3.55</td>
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<td>23</td>
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<td>Laundering Cycle</td>
<td>17.57</td>
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<td>23</td>
</tr>
<tr>
<td>Surfactant x Finish</td>
<td>.57</td>
<td>6</td>
<td>23</td>
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<tr>
<td>Surfactant x Temperature</td>
<td>1.14</td>
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<td>23</td>
</tr>
<tr>
<td>Surfactant x Laundering Cycle</td>
<td>6.82</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Finish x Temperature</td>
<td>.88</td>
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<th>Source</th>
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</thead>
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<tr>
<td></td>
<td></td>
<td>Num.</td>
<td>Den.</td>
</tr>
<tr>
<td>Finish x Laundering Cycle</td>
<td>3.35</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Temperature x Laundering Cycle</td>
<td>3.20</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Surfactant x Finish x Temperature</td>
<td>.94</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Surfactant x Finish x Laundering Cycle</td>
<td>1.4</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Surfactant x Temperature x Laundering Cycle</td>
<td>2.70</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Finish x Temperature x Laundering Cycle</td>
<td>.99</td>
<td>6</td>
<td>23</td>
</tr>
<tr>
<td>Surfactant x Finish x Temperature x Laundering Cycle</td>
<td>.61</td>
<td>6</td>
<td>23</td>
</tr>
</tbody>
</table>

*p = .02

**p = .01

Num. = Numerator
Den. = Denominator
resistance also received average weights. Of the thirteen significant multivariate effects, the dependent variables, surfactant content and abrasion resistance accounted for most of the significance, with surfactant content receiving the heavier weight. The exception was the interaction between finish and temperature where equal weights were assigned to surfactant content, filling tensile strength, and warp and filling wrinkle recovery. The canonical correlations are shown in Appendix A.

The multivariate analyses were used to examine the composite effects of the dependent variables. The results of the univariate analyses will now be examined for the interpretation of the individual dependent variables. Where a univariate significance was obtained, the adjusted means are given in the body of the text. Corresponding original means can be found in Appendix B.

**UNIVARIATE ANALYSIS OF COVARIANCE**

**Surfactant Content**

The univariate analysis of covariance for the variable surfactant content, yielded significant results for 13 of the 15 effects. Where more than two means were involved, as in the case of the interactions, multiple comparisons
were made to determine which of the means differed. The results of the analysis are shown in Table 3.

A significant surfactant effect was obtained. The means are shown in Table 4. An examination of the means revealed that the anionic surfactant had a greater affinity for the fabrics than the nonionic surfactants.

The effect for finish was also significant at p=.02 level of significance. The means in Table 5 indicate that the untreated fabric had a higher mean surfactant content than the resin-treated cotton fabrics.

The laundering temperature was found to be an important factor in the quantity of surfactant retained by the fabric. The quantity of surfactant left on the fabric increased as the temperature was raised from 105 F to 135 F. The means are given in Table 6. More surfactant was also deposited on the fabric as the number of times the fabric was laundered increased from three to eight times. Table 7 shows these means.

The interaction between surfactant and finish was significant and a multiple comparison was made to determine which of the means differed. It was found that the untreated and resin-treated fabric differed appreciably in the amount of anionic and nonionic surfactant they contained. The
Table 3
Analysis of Covariance Summary Table
For Surfactant Content

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>Deg. of Freedom</th>
<th>Mean Square</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Covariates</td>
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<td></td>
<td></td>
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<tr>
<td>Surfactant Content</td>
<td>36.59</td>
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<td>36.59</td>
<td>666.4</td>
</tr>
<tr>
<td>Warp Wrinkle Recovery</td>
<td>9.11</td>
<td>1</td>
<td>9.11</td>
<td>160.9</td>
</tr>
<tr>
<td>Filling Wrinkle Recovery</td>
<td>1.45</td>
<td>1</td>
<td>1.45</td>
<td>25.6</td>
</tr>
<tr>
<td>Abrasion Resistance</td>
<td>.15</td>
<td>1</td>
<td>.15</td>
<td>2.7</td>
</tr>
<tr>
<td>Surfactant</td>
<td>.98</td>
<td>1</td>
<td>.98</td>
<td>17.5**</td>
</tr>
<tr>
<td>Finish</td>
<td>.31</td>
<td>1</td>
<td>.31</td>
<td>5.5*</td>
</tr>
<tr>
<td>Temperature</td>
<td>4.74</td>
<td>1</td>
<td>4.74</td>
<td>83.8**</td>
</tr>
<tr>
<td>Laundering Cycle</td>
<td>17.14</td>
<td>1</td>
<td>17.14</td>
<td>302.8**</td>
</tr>
<tr>
<td>Surfactant x Finish</td>
<td>.78</td>
<td>1</td>
<td>.78</td>
<td>13.8**</td>
</tr>
<tr>
<td>Surfactant x Temperature</td>
<td>.59</td>
<td>1</td>
<td>.59</td>
<td>10.4*</td>
</tr>
<tr>
<td>Surfactant x Laundering Cycle</td>
<td>9.59</td>
<td>1</td>
<td>9.59</td>
<td>169.4**</td>
</tr>
<tr>
<td>Finish x Temperature</td>
<td>.15</td>
<td>1</td>
<td>.15</td>
<td>2.6</td>
</tr>
</tbody>
</table>
Table 3 (continued)

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>Deg. of Freedom</th>
<th>Mean Square</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Finish x Laundering Cycle</td>
<td>.13</td>
<td>1</td>
<td>.13</td>
<td>.3</td>
</tr>
<tr>
<td>Temperature x Laundering Cycle</td>
<td>0.02</td>
<td>1</td>
<td>0.02</td>
<td>.3</td>
</tr>
<tr>
<td>Surfactant x Finish x Temperature</td>
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<td>1</td>
<td>.33</td>
<td>5.8*</td>
</tr>
<tr>
<td>Surfactant x Finish x Laundering Cycle</td>
<td>2.35</td>
<td>1</td>
<td>2.35</td>
<td>41.5**</td>
</tr>
<tr>
<td>Finish x Temperature x Laundering Cycle</td>
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<td>.74</td>
<td>13.0**</td>
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<tr>
<td>Surfactant x Finish x Temperature x Laundering Cycle</td>
<td>.36</td>
<td>1</td>
<td>.36</td>
<td>6.4*</td>
</tr>
<tr>
<td>Error</td>
<td>1.58</td>
<td>28</td>
<td>.08</td>
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</tr>
<tr>
<td>Total</td>
<td>88.18</td>
<td>47</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*p = .02  
**p = .001
Table 4

Adjusted Mean Surfactant Content
For Surfactant Type
(mg/gm fabric)

<table>
<thead>
<tr>
<th>Surfactant</th>
<th>No. of Samples</th>
<th>Mean Surfactant Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anionic</td>
<td>24</td>
<td>3.28</td>
</tr>
<tr>
<td>Nonionic</td>
<td>24</td>
<td>.87</td>
</tr>
</tbody>
</table>

Table 5

Adjusted Mean Surfactant Content for Finish
(mg/gm fabric)

<table>
<thead>
<tr>
<th>Finish</th>
<th>No. of Samples</th>
<th>Mean Surfactant Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated Cotton</td>
<td>24</td>
<td>2.28</td>
</tr>
<tr>
<td>Resin Treated Cotton</td>
<td>24</td>
<td>1.87</td>
</tr>
</tbody>
</table>
Table 6

Adjusted Mean Surfactant Content
For Temperature
(mg/gm fabric)

<table>
<thead>
<tr>
<th>Temperature</th>
<th>No. of Samples</th>
<th>Mean Surfactant Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>105 F</td>
<td>24</td>
<td>1.76</td>
</tr>
<tr>
<td>135 F</td>
<td>24</td>
<td>2.39</td>
</tr>
</tbody>
</table>

Table 7

Adjusted Mean Surfactant Content
For Laundering Cycle
(mg/gm fabric)

<table>
<thead>
<tr>
<th>Laundering Cycle</th>
<th>No. of Samples</th>
<th>Mean Surfactant Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>24</td>
<td>1.48</td>
</tr>
<tr>
<td>8</td>
<td>24</td>
<td>2.67</td>
</tr>
</tbody>
</table>
resin-treated fabric retained more anionic surfactant than the untreated fabric while the latter retained more nonionic surfactant than the resin-treated fabric. The means are shown in Figure 7.

![Figure 7](image)

**Figure 7**
Adjusted Mean Surfactant Content for Surfactant by Finish Interaction (N=12)

Key:

UN = Untreated

RT = Resin-Treated

A highly significant interaction between surfactant and laundering temperature was obtained. The slope of the lines in Figure 8 shows that an increase in temperature seemed to cause a greater increase in the retention of the
anionic surfactant than in the retention of the nonionic surfactant.

Figure 8

Adjusted Mean Surfactant Content for Surfactant by Temperature Interaction
(N=12)

Key:

_________ Anionic Surfactant

___ ___ ___ Nonionic Surfactant
anionic surfactant than in the retention of the nonionic surfactant.

The surfactant content of the fabrics also varied with the different levels of surfactant and laundering cycle, as indicated by the significant interaction. The multiple comparisons were significant for all of the means. As in the case of the preceding interaction, the anionic surfactant increased at a faster rate than the nonionic surfactant as the number of times the fabric was laundered increased from 3 to 8. Figure 9 illustrates the nature of this interaction.

![Figure 9](Image)

**Figure 9**

*Adjusted Mean Surfactant Content for Surfactant by Laundering Cycle Interaction (N=12)*

**Key:**
- Anionic Surfactant
- Nonionic Surfactant
No significant effect on surfactant content was found for the interactions of finish with temperature, finish with laundering cycle, and temperature with laundering cycle.

A significant interaction was obtained between surfactant, finish, and temperature. A multiple comparison of the means showed that the two groups of fabric did not differ significantly in their anionic surfactant content, when laundered at 105 F. At 135 F however, the resin-treated fabric accumulated more anionic surfactant than the untreated fabric. The position was reversed for the nonionic surfactant. The untreated fabric retained more nonionic surfactant than the resin-treated fabric at both temperatures. Furthermore, while the amount of surfactant on the untreated fabric increased with temperature, the quantity left on the finished fabric remained stable. The means are shown in Figure 10.

The interaction between surfactant, finish, and laundering cycle was significant. The means, shown in Figure 11, revealed that both fabrics contained about the same amount of anionic surfactant at the third laundering cycle. At the eighth cycle, however, the resin-treated fabric had accumulated more of this class of surfactant. The reverse seemed to be the case for the nonionic surfactant. The
Figure 10

Adjusted Mean Surfactant Content for Surfactant by Finish by Temperature Interaction (N=6)

Key:

- Anionic Surfactant, Resin-Treated
- Anionic Surfactant, Untreated
- Nonionic Surfactant, Resin-Treated
- Nonionic Surfactant, Untreated
Figure 11

Adjusted Mean Surfactant Content for Surfactant by Finish by Laundering Cycle Interaction (N=6)

Key:

_____________ Anionic Surfactant, Resin-Treated

— __ __ __ Anionic Surfactant, Untreated

_____________ Nonionic Surfactant, Resin-Treated

-x-x-x-x-x-x-x- Nonionic Surfactant, Untreated
untreated fabric contained more nonionic surfactant at the third laundering cycle than the resin-treated fabric and this quantity increased appreciably at the eighth cycle while the amount on the resin-treated fabric remained fairly stable.

A multiple comparison between the means for the surfactant by temperature by laundering cycle interaction showed that increases in temperature resulted in an increase in the quantity of anionic surfactant on the fabric. This held true for both laundering cycles. The fabric samples laundered in the nonionic surfactant behaved differently. Increasing the temperature resulted in an increase in the surfactant content of the fabrics only at the third laundering cycle. At the eighth laundering cycle, while the surfactant content of the samples laundered at 105 F had increased, that of the fabrics laundered at 135 F remained essentially unchanged, and approximately the same value as that of the fabrics laundered at 105 F. An illustration of this interaction is given in Figure 12.

The interaction between temperature, finish, and laundering cycle was not significant nor was the four-way interaction between the four independent variables.
Figure 12

Adjusted Mean Surfactant Content for Surfactant by Temperature by Laundering Cycle Interaction (N=6)

Key:

---------- Anionic Surfactant, 105 F

_____ _____ Anionic Surfactant, 135 F

----------- Nonionic Surfactant, 105 F

-x-x-x-x-x-x-x- Nonionic Surfactant, 135 F
Warp Tensile Strength

A univariate analysis of covariance for the variable warp tensile strength yielded a significant F ratio for the main effect of surfactant and for the interactions between surfactant and laundering cycle, and surfactant, finish, and laundering cycle. The results are summarized in Table 9. Multiple comparisons were made to determine which of the means differed in cases where more than two means were involved.

On examination of the mean tensile strength for the variable surfactant, it was found that the fabrics laundered in anionic surfactant had a higher warp tensile strength than the fabrics laundered in nonionic surfactant. Table 8 shows the means.

Table 8

<table>
<thead>
<tr>
<th>Surfactant</th>
<th>No. of Samples</th>
<th>Warp Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anionic</td>
<td>24</td>
<td>1.51</td>
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<tr>
<td>Nonionic</td>
<td>24</td>
<td>1.39</td>
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</tr>
<tr>
<td><strong>Covariates</strong></td>
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<td></td>
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<tr>
<td>Surfactant Content</td>
<td>0.06</td>
<td>1</td>
</tr>
<tr>
<td>Warp Wrinkle Recovery</td>
<td>1.34</td>
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</tr>
<tr>
<td>Filling Wrinkle Recovery</td>
<td>.06</td>
<td>1</td>
</tr>
<tr>
<td>Abrasion Resistance</td>
<td>.01</td>
<td>1</td>
</tr>
<tr>
<td>Surfactant</td>
<td>.03</td>
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</tr>
<tr>
<td>Finish</td>
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<td>1</td>
</tr>
<tr>
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</tr>
<tr>
<td>Laundering Cycle</td>
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<td>Surfactant x Finish</td>
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</tr>
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</tr>
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### Table 9 (continued)

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<th>F</th>
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</thead>
<tbody>
<tr>
<td>Finish x Laundering Cycle</td>
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<td>.00</td>
<td>.0</td>
</tr>
<tr>
<td>Temperature x Laundering Cycle</td>
<td>.001</td>
<td>1</td>
<td>.001</td>
<td>.1</td>
</tr>
<tr>
<td>Surfactant x Finish x Temperature</td>
<td>.001</td>
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<td>.001</td>
<td>.2</td>
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<td>Surfactant x Finish x Laundering Cycle</td>
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<td>.02</td>
<td>7.5*</td>
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<td>.01</td>
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<tr>
<td>Finish x Temperature x Laundering Cycle</td>
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</tr>
</tbody>
</table>

*p = .05
A comparison of the means was made for the interaction between surfactant and finish. It was found that the difference in the warp tensile strength of the two fabrics was more pronounced in the fabrics laundered with anionic surfactant.

The interaction between surfactant, finish, and laundering cycle was significant. The mean warp tensile strength of the fabrics did not change appreciably with changes in laundering cycle. However, the difference in the strength of the samples washed with the two surfactants was more noticeable in the resin-treated fabric than in the untreated fabric.

**Filling Tensile Strength**

The result of the univariate analysis for the filling tensile strength, given in Table 10, showed that only the interaction between finish and laundering temperature was significant. Increasing the temperature caused an increase in the tensile strength of the untreated fabric while the resin-treated fabric remained essentially unchanged.

**Warp Wrinkle Recovery**

Only one of the fifteen effects was significant for the variable warp wrinkle recovery. The resin-treated
Table 10

Analysis of Covariance Summary Table for Filling Tensile Strength

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>Deg. of Freedom</th>
<th>Mean Square</th>
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<td>Finish</td>
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*p = .05
fabric had a greater mean recovery angle than the untreated fabric. The results of the analysis are summarized in Table 11.

**Filling Wrinkle Recovery**

No significant main effect was obtained for the filling wrinkle recovery. It was however, found that the interactions of finish and laundering cycle; surfactant, temperature and laundering cycle; and finish, temperature, and laundering cycle produced significant changes in filling wrinkle recovery. The univariate results are shown in Table 12. Multiple comparisons were made in cases where more than two means were involved.

A comparison of the means for laundering cycle by finish interaction revealed that while the untreated fabric lost its wrinkle recovery as the laundering cycle increased, the resin-treated fabric did not change significantly. The significant interaction of surfactant with temperature and laundering cycle showed, on examination of the means, that the recovery angle of the fabrics laundered in nonionic surfactant was stable to changes in temperature and laundering cycle. For the anionic surfactant however, a significant increase in recovery angle was observed at the third
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Table 11 (continued)

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*p = .05
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<td>3.6</td>
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Table 12 (continued)

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<tr>
<td>Total</td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

*p = .05
laundering for the temperature of 135°F. This increase was not observed at the eighth laundering.

Although a significant F ratio was obtained for the finish by temperature by laundering cycle interaction, no significantly different means were found using Newman Keul's method, beyond the fact that the difference in the wrinkle recovery of the untreated samples laundered at 105°F was more pronounced at the eighth laundering than the third.

**Abrasion Resistance**

Twelve of the fifteen effects for the univariate analysis of abrasion resistance were significant at the 5% level of significance. The results are summarized in Table 13. Multiple comparisons were made to determine the significance of the differences among the means in cases where more than two means were involved.

The fabric samples laundered in nonionic surfactant had a higher abrasion resistance than those laundered in anionic surfactant. The means are shown in Table 14. The untreated fabric exhibited a higher resistance to abrasion than the resin treated fabric. The samples that were laundered eight times also had a higher mean abrasion resistance than those laundered three times. The means are given in Table 15.
Table 13 (continued)

<table>
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*p = .05

**p = .001
Table 14

Adjusted Mean Abrasion Resistance for Surfactant

<table>
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<tr>
<th>Surfactant</th>
<th>No. of Samples</th>
<th>Mean Abrasion Resistance</th>
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<tr>
<td>Anionic</td>
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<td>114.9</td>
</tr>
<tr>
<td>Nonionic</td>
<td>24</td>
<td>140.8</td>
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</table>

Table 15

Adjusted Mean Abrasion Resistance for Laundering Cycle

<table>
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<tr>
<th>Laundering Cycle</th>
<th>No. of Samples</th>
<th>Mean Abrasion Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>24</td>
<td>117.83</td>
</tr>
<tr>
<td>8</td>
<td>24</td>
<td>137.91</td>
</tr>
</tbody>
</table>
A multiple comparison for the significant surfactant by temperature interaction showed that an increase in the laundering temperature caused the abrasion resistance of the samples laundered in anionic surfactant to increase. The reverse situation was obtained for the nonionic surfactant where the resistance to abrasion decreased as the temperature was raised from 105 F to 135 F.

The significant interaction between surfactant and laundering cycle showed, after comparisons were made, that the fabrics laundered in the nonionic surfactant showed an increase in abrasion resistance from three to eight laundering cycles. The increase for the samples laundered in the anionic surfactant was not as pronounced. The means are given in Figure 13.

The interaction between finish and temperature was significant. When the means were compared, it was found that the increase in temperature caused an increase in the abrasion resistance of the untreated fabric while the abrasion resistance of the resin-treated fabric was relatively unaffected by changes in temperature. A similar result was obtained for the interaction between finish and laundering cycle. The resistance of the untreated fabrics to abrasion increased with laundering cycle while that of the resin-treated fabric did not change significantly.
Resistance to abrasion also yielded different values for the different levels of temperature and laundering cycle. At the lower temperature, there was no significant increase from three to eight launderings. At 135 F however, abrasion resistance increased with the laundering cycles.
The same pattern was repeated in the interaction between finish, temperature and surfactant. The untreated samples laundered in anionic surfactant increased in abrasion resistance as temperature increased. No significant changes were observed for the resin-treated samples. No significant changes were observed in the fabrics laundered in the non-ionic surfactant. The means are shown in Figure 14.

![Graph showing adjusted mean abrasion resistance for surfactant by finish by temperature interaction](image)

**Figure 14**

Adjusted Mean Abrasion Resistance for Surfactant by Finish by Temperature Interaction (N=6)

**Key:**
- Anionic Surfactant, Unfinished
- Anionic Surfactant, Resin-Treated
- Nonionic Surfactant, Resin-Treated
- Nonionic Surfactant, Untreated
Comparisons were made to determine which of the means for the significant surfactant by laundering cycle by finish interaction differed. These means are shown in Figure 15. It was found that the abrasion resistance of the untreated fabric increased with laundering cycle, irrespective of the surfactant used, with the nonionic surfactant maintaining its superiority. With the resin-treated fabric, however, the fabrics laundered in both surfactants had comparable abrasion resistance at the third laundering cycle. At the eighth cycle, the abrasion resistance of the fabrics laundered in nonionic surfactant exceeded that of the fabrics laundered in anionic surfactant.

**Summary**

The multivariate analysis of covariance yielded significant results for 12 of the 15 effects at the .05 level of significance. The canonical correlations showed that surfactant content and abrasion resistance were responsible for most of the significance. However, surfactant content received the heavier weight of the two in most of the instances. The results of the univariate analysis of covariance were in agreement with the multivariate results.
Figure 15

Adjusted Mean Abrasion Resistance for Surfactant by Laundering Cycle by Finish Interaction (N=6)

Key:  
— Anionic Surfactant, Untreated
— Anionic Surfactant, Resin-Treated
— Nonionic Surfactant, Untreated
-x-x-x-x-x- Nonionic Surfactant, Resin-Treated
The univariate analysis for surfactant content indicated the significance of 12 of the 15 effects at the .02 and .01 levels of significance. Multiple comparisons of means showed that the fabrics retained more anionic than nonionic surfactant. The quantity of surfactant retained increased significantly with temperature and laundering cycle. On the whole, the untreated fabric retained more surfactant than the finished fabric but while the untreated fabric had a greater affinity for the nonionic surfactant than the resin-treated fabric, the position was reversed for the anionic surfactant. It was found that the anionic surfactant content of the fabrics was greater at 135 F than at 105 F. The rate of increase of the anionic surfactant as temperature and laundering cycle increased was greater than that of the nonionic surfactant. The rate of increase of the anionic surfactant content was also significantly higher for the resin-treated fabric than for the untreated fabric. The rate of increase of the nonionic surfactant was, however, higher for the untreated fabric than for the resin-treated fabric.

The univariate analysis for warp tensile strength gave three significant effects while that for the filling tensile strength showed only one significant effect at the
.05 level of confidence. The fabrics laundered in the anionic surfactant had a higher warp tensile strength than the fabrics laundered in the nonionic surfactant. The effect of surfactant was also more obvious for the untreated fabric than for the resin-treated fabric. The anionic surfactant gave a significantly higher warp wrinkle recovery than the nonionic surfactant. Three interaction effects were significant for the filling wrinkle recovery. The untreated fabric lost wrinkle recovery as the laundering cycle increased, and the temperature of 105 F contributed more to this decrease than the temperature of 135 F, especially for the fabrics laundered in the anionic surfactant. The filling wrinkle recovery of the fabrics laundered in the nonionic surfactant was stable to temperature and laundering cycle.

Twelve highly significant effects were obtained for the abrasion resistance. Multiple comparisons showed that the fabrics laundered in nonionic surfactant had a higher mean abrasion resistance than those laundered in anionic surfactant. The untreated fabric had the higher abrasion resistance of the two fabrics and abrasion resistance increased with laundering cycle. A highly significant interaction was obtained for surfactant and temperature.
Multiple comparisons showed that increases in temperature increased the abrasion resistance of the fabrics laundered in anionic surfactant. For the nonionic surfactant, the crucial factor was the laundering cycle, not temperature. The finish by laundering cycle interaction gave significant results. The untreated fabric was more susceptible to changes in laundering cycle than the finished fabric. The higher temperature also appeared to improve abrasion resistance as laundering cycle increased. The untreated fabric laundered in anionic surfactant showed increased abrasion resistance as temperature and laundering cycle increased while the resin-treated fabric laundered in anionic surfactant was stable to changes in temperature and laundering cycle. For the fabrics laundered in anionic surfactant, the beneficial effect of temperature on the abrasion resistance of the untreated fabric was more readily observed at the eighth laundering.

An examination of the correlation coefficients revealed only one significant correlation between surfactant content and the other dependent variables. This was a low correlation of .3 between surfactant content and warp wrinkle recovery. The correlation coefficients are shown in Appendix C.
CHAPTER V

SUMMARY, DISCUSSION AND RECOMMENDATIONS

SUMMARY

This study was concerned with the build-up of surfactant on untreated and resin-finished cotton fabrics. The investigation was prompted by a personal interest in cotton, a major product of Nigerian textile mills, and the fact that soil removal by an aqueous solution of surfactants is an important part of the wear life of a fabric. A search of the literature revealed that the build-up of surfactant on cotton had been studied. However, the conditions under which this build-up occurred had not been fully investigated nor were data found related to this phenomenon on resin-treated cotton.

Specifically, this study focused on the build-up of anionic and nonionic surfactants on untreated and resin-treated cotton fabrics under two temperature conditions and over eight laundering cycles. A secondary consideration was the effect of this build-up on the tensile strength, wrinkle recovery, and abrasion resistance of the fabrics.
The study was developed in the following sequence:

1. Formulation of hypotheses
2. Identification of the variables
3. Collection of data
4. Results of data analysis.

A summary of each aspect is given below, followed by the discussion of results and recommendations.

**Formulation of Hypotheses**

The following null hypotheses were developed for the study:

**Hypothesis 1.** There is no significant surfactant build-up attributable to the durable press finish.

**Hypothesis 2.** There is no significant surfactant build-up attributable to surfactant type.

**Hypothesis 3.** There is no significant surfactant build-up attributable to laundering temperature.

**Hypothesis 4.** There is no significant surfactant build-up attributable to any combination of fabric finish, laundering temperature and surfactant type.

**Hypothesis 5.** There is no significant difference in fabric properties attributable to surfactant build-up.
Identification of Variables

The variables identified for the study were the build-up of surfactant on untreated and resin-treated cotton fabrics under varying laundering conditions and the effect of the build-up on the tensile strength, wrinkle recovery, and abrasion resistance of the fabrics. The different laundering conditions were:

1. Surfactant - Anionic (Sodium Dodecylbenzene Sulphonate) and Nonionic (S1kyl Phenoxy Polyethoxy Ethanol)
2. Finish - Untreated and Resin-Treated
3. Temperature - 105 F and 135 F
4. Laundering Cycle - 3 and 8.

Collection of Data

Untreated and resin-treated cotton fabrics were obtained from the manufacturer and 54 samples were prepared. Three samples were assigned to each level of the factors and three samples of each fabric were left unlaundered and used as control. The fabric samples were laundered in an automatic home washer for 10 minutes and rinsed for 6 minutes. They were tumble dried for 30 minutes in an automatic home dryer at a high temperature setting.

The methylene blue indicator method was used to determine the anionic surfactant content of the samples and
the phosphomolybdic acid method was used for nonionic determinations. The surfactant content per gram of fabric was calculated using the formula:

\[
\frac{\text{mg surfactant}}{\text{gm of fabric}} = \frac{\% \text{conc.} \times \text{flask sol. vol.} \times 1000}{\% \text{cotton solids} \times \text{gm cotton}}
\]

ASTM and AATCC standard methods were used to test the physical properties of the fabrics. Multivariate analysis of covariance was used to determine the significance of the results. Multiple comparisons were made between the means of significant effects, using Newman Kuel's method.

**Results of Data Analysis**

The multivariate analysis of covariance yielded significant results for all the effects but the surfactant by finish interaction and the four-way interaction between the variables. On examination of the canonical correlations, it was found that the surfactant content and abrasion resistance accounted for most of the significance.

The univariate analysis of covariance for surfactant content yielded significant results for all but the finish by temperature, finish by laundering cycle, and temperature by laundering cycle interactions. Comparisons of individual

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Ginn, Kinney, and Harris, op. cit., p. 138.
means showed that surfactant content increased with temperature and laundering cycle. On the whole, more anionic than nonionic surfactant was retained. However, the finished fabric retained more of the anionic surfactant while the untreated fabric retained more of the nonionic surfactant. The surfactant content of the resin-finished fabric was higher than that of the untreated fabric. It was found that temperature was a crucial factor in the build-up of the anionic surfactant while laundering cycle was more important for the nonionic surfactant. Hypotheses one to four were rejected as a result of the evidence.

No strong correlations were found to exist between surfactant content and the measured fabric properties. There were no grounds for rejecting hypothesis 5.

The univariate analysis for abrasion resistance yielded 13 significant effects. Comparisons of the means showed that the nonionic surfactant caused a greater resistance to abrasion than the anionic surfactant. The abrasion resistance of the resin-treated fabric displayed a stability to changes in temperature and laundering cycle while the abrasion resistance of the untreated fabric increased with temperature and laundering cycle. Increases in temperature resulted in an increase in the resistance to
abrasion of the fabrics laundered in anionic surfactant
while for those laundered in nonionic surfactant, laundering
cycle was the important factor.

DISCUSSION

This investigation was designed to answer specific
questions namely:

1. Do fabric finish and surfactant type affect the
quantity of surfactant deposited on the fabric during laun-
dering?

2. Under what laundering conditions does surfactant
build-up occur?

3. Does surfactant build-up affect the physical
properties of the fabric?

To this end data were collected in a laboratory experiment.
The laundering procedure employed standard equipment and the
laundering conditions are replicable. The analytical method
used to determine the quantity of nonionic surfactant repre-
sents a method recommended by the AATCC for determining small
quantities of nonionic surfactant. The methylene blue method
for the anionic determination gives an end point, in a
titration, where colorometric changes are involved. In small
quantities of surfactant, the end point requires careful
interpretation. The procedure is used for assay purposes and here, the small changes in color on titration would not greatly affect the reliability of the results.

**Surfactant Content**

The univariate analysis of covariance showed a significant effect for the covariate, surfactant content. On examination of the original means, given in Appendix B, it was found that the fabrics contained more of the nonionic surfactant than the anionic surfactant. This was not surprising since if the anionic surfactant had been used in the scouring processes care would have been taken to ensure complete removal. Sodium salts of anionic materials cause yellowing on heating. The comparatively high nonionic surfactant content of the resin-treated fabric was also not surprising since nonionic wetting agents form a part of the resin system.

Hypothesis 1 stated that there was no significant surfactant build-up attributable to the durable press process. The univariate analysis of covariance yielded a significant finish effect. An examination of the means showed that the untreated fabric had a higher surfactant content than the resin-treated fabric. The quantity of
surfactant retained by both fabrics was on the low side and represented .23% and .19% of the fabric weights respectively. This is in keeping with the results of other investigators. Schwarz, Maple, and Aiken all found that cotton did not adsorb very large quantities of surfactant. Unlike wool and other amine-containing fibers, cotton contains no polar groups capable of forming chemical bonds. The surfactant molecules are then held by purely physical forces. This also accounts for the fact that the resin-treated fabric retained less surfactant than the untreated fabric. The resin treatment results in a reaction of the resin with the hydroxyl groups of cellulose to form cross-linkages. This loss of the hydroxyl groups results in a reduction of the free energy and Van Der Waals forces which reduced the affinity of the fibers for the surfactant molecules. The evidence provided by the data led to a rejection of hypothesis 1.

Hypothesis 2 stated that there was no significant surfactant build-up attributable to surfactant type. The univariate analysis gave a significant surfactant effect.

63 Schwarz et al., op. cit., p. 37, and Maple, op. cit., p. 505, and Aiken, op. cit., p. 60.
The means on Table 4 showed the retention of a larger quantity of the anionic than nonionic surfactant. This result is in keeping with market expectations of the nonionic surfactant. Zika stated that nonionic surfactants had a low substantivity. 64 Weatherburn and Bailey also found, in their investigation of the adsorption of the different classes of surfactant, that the nonionic surfactant was the least adsorbed. 65 This can be explained by their nonionic nature. Possessing no ionic groups, this class of surfactants has little attraction for the fabric and the hardness minerals in the wash bath. Hypothesis 2 was rejected on the basis of the above evidence.

Hypothesis 3 stated that there was no significant surfactant build-up attributable to the laundering temperature. A highly significant effect was obtained for temperature and the means, given in Table 6, showed that surfactant build-up increased with temperature. Investigators on the adsorption of surfactant on cotton found that adsorption increased with temperature. 66 A number of

64 Zika, op. cit., p. 26.
65 Weatherburn and Bailey, op. cit., p. 797.
66 Ibid.
factors are responsible for this behavior. At higher temperatures, the surfactant in the wash bath exists in the monomolecular form rather than the aggregated form and so penetrates the fibers more readily. This in effect, increases the quantity of molecules involved in adsorption. Aiken and other investigators have shown that increases in concentration result in increased adsorption. Further to this effect of temperature to the form of the surfactant in the wash bath, increases in temperature result in increased mobility of the fiber molecules. The opening up of the fiber that ensues makes the more remote areas of the fiber accessible to the hardness minerals in the wash water which are partly responsible for build-up. The overall result is increased adsorption and retention of surfactant. Hypothesis 3 was rejected as a result of the above evidence.

Hypothesis 4 stated that there was no significant surfactant build-up attributable to any combination of finish, surfactant and temperature. All but three of the interaction effects were found significant in the univariate analysis of covariance. A comparison of the means for the surfactant by finish interaction showed that the untreated

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67 Aiken, op. cit., p. 60.
fabric retained more nonionic surfactant that the resin-treated fabric while the resin-treated fabric retained more anionic surfactant than the untreated fabric. Weatherburn and Bailey came up with a result similar to the first portion of the interaction. They found that their cotton fabrics adsorbed more nonionic than any other category of surfactant.\(^{68}\) In this case, an explanation can be found in the nature of the resin-treated fabric. The build-up of surfactant on cotton has been attributed to the presence of waxes on the fiber surface, to hardness minerals in the wash water, and to free energy forces within the fiber. The durable press process involves an introduction of a reactive resin into the fiber. The resin reacts with the hydroxyl groups of cellulose to form crosslinkages. This reaction reduces the free energy forces of the crosslinked cellulose cutting down its affinity for several chemical compounds. It was therefore not surprising that the resin-treated fabric had a lower affinity for the nonionic surfactant which is not highly attracted to textile fibers in the first place. The second part of the interaction showed that the resin-treated fabric contained more anionic surfactant.

\(^{68}\) Weatherburn and Bailey, loc. cit.
surfactant than the untreated fabric. The resin system for the durable press finish contains polyethylene softeners which react to surfactants in a manner similar to wax. Ginn et al. found that wax-containing cotton adsorbed more anionic surfactant than dewaxed cotton. The softener then accounts for the higher retention of the anionic surfactant.

A highly significant interaction was obtained for the surfactant by temperature interaction. The comparison of the means shown in Figure 9, indicated that the rate of increase of the anionic surfactant as temperature increased was greater than that of the nonionic surfactant. It was observed in the discussion of the effect of temperature that increasing the temperature of the wash bath increased the single ion concentration as well as opening up the remote areas of the fiber, thus providing opportunity for increased retention of surfactant. It was also noted earlier in the discussion, that nonionic surfactant had a low affinity for the fibers. Increasing the opportunity would therefore have little effect if there is no affinity for the fibers. Thus, the anionic surfactant content increased as the opportunity was created by increasing the temperature. The increase for the nonionic was not as pronounced.

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69 Ginn, Kinney, and Harris, loc. cit.
The interaction between surfactant and laundering cycle was significant. The means in Figure 9 showed a reaction similar to what was observed in the preceding interaction. The rate of increase of the anionic surfactant as laundering cycle increased was greater than that of the nonionic surfactant. It was expected that more hardness minerals which are partly responsible for surfactant build-up would be deposited on the fabric as the number of launderings increased. As in the case of temperature, increasing the mineral deposits on the fabric would not greatly affect the nonionic surfactant content since it possesses little affinity for these materials.

The means for the interaction between surfactant, finish and laundering cycle showed that the anionic surfactant content of the two fabrics did not differ significantly at 105 F but at 135 F the resin-treated fabric accumulated more surfactant than the untreated fabric. For the non-ionic surfactant the untreated fabric retained more surfactant than the finished fabrics at both temperatures. This is a more complex illustration of the behavior already observed in the surfactant by finish and the surfactant by temperature interactions. The higher temperature increased the single ion concentration, making the fiber molecules
more accessible to the surfactant; this increased the quantity of anions retained by the softener.

A comparison of the means for the surfactant by temperature by laundering cycle interaction, illustrated in Figure 12, showed that increasing the temperature resulted in an increase in the quantity of anionic surfactant on the fabric. This held true for both laundering cycles. For the nonionic surfactant however, an increase in temperature resulted in increased build-up only at the third laundering cycle. At the eighth cycle, while the surfactant content increased for the fabrics laundered at 105 F, those laundered at 135 F remained essentially unchanged. The effect of temperature on the single ion concentration and the mobility of the cellulose molecule is evident in the anionic surfactant. The behavior of the nonionic surfactant is more difficult to explain. An examination of the individual components of this interaction suggested that the anomalous behavior was introduced by the resin-treated fabric which had a considerable drop in surfactant content when laundered eight times at 135 F. It is possible that a gradual removal of the softener was occurring at the higher temperature and this loss was neutralizing the increase that was occurring with laundering cycle.
In summary, several significant interactions were found to exist between surfactant type, fabric finish, and laundering temperature. The finished fabric built-up more anionic surfactant than the untreated fabric while the position was reversed for the nonionic surfactant. Increased temperature caused a greater increase in the build-up of anionic surfactant than the nonionic surfactant. In light of the above finding, hypothesis 4 was rejected.

Hypothesis 5 stated that there were no significant changes in fabric properties attributable to surfactant build-up. The correlation coefficients were examined for strong correlations between surfactant content and the variables. One correlation, between surfactant content and warp wrinkle recovery was significant. No explanation was found for this especially since the filling wrinkle recovery had a very low correlation with this variable. Enough evidence was not found to lead to a rejection of hypothesis 5.

Other Findings of Interest

It was found that the abrasion resistance of the fabrics was affected by the experimental variables. Some of these, like the effect of finish, were expected and so not of interest. The effect of surfactant and the
interactions between surfactants, temperature, and laundering cycles call for some mention.

The samples laundered in nonionic surfactant were found to possess an abrasion resistance superior to that of the fabrics laundered in anionic surfactant. Temperature had no effect on the abrasion resistance of these fabrics but laundering cycle caused appreciable increases. Three possible explanations can be advanced for this behavior.

1. Shrinkage occurred, resulting in increased abrasion resistance, and the nonionic surfactant caused a greater amount of shrinkage than the anionic surfactant.

2. The nonionic surfactant was causing a hydrolysis of the resin and this showed up as improved abrasion resistance.

3. The nonionic surfactant acted as a lubricant for fibers resulting in increased abrasion resistance.

It is known that higher temperatures cause greater shrinkage than the low temperature. If the nonionic surfactant was causing more shrinkage than the anionic surfactant it would have been observed in the surfactant by temperature interaction. Rather the reverse appeared to be the case. An examination of the surfactant by finish by temperature interaction showed that the increase in abrasion
resistance of the samples laundered in the anionic surfactant was changed only for the untreated fabric. This fabric was not treated for shrinkage control as was the resin-finished fabric. The abrasion resistance of the fabrics laundered in the nonionic surfactant increased with laundering cycle. Shrinkage would hardly continue to eight launderings. This then rules out the first explanation. Resin hydrolysis is known to increase with temperature. Yet, in the finish by temperature interaction, the abrasion resistance of the finished fabric did not increase with temperature. This also rules out hydrolysis of the resin, leaving lubrication of the fibers as the only plausible explanation.

In summary, the univariate analysis of covariance yielded twelve significant effects which led to a rejection of the first four hypotheses which stated that there was no significant surfactant build-up attributable to the durable press finish, surfactant type, laundering temperature, or any combination of these factors. An examination of the correlation between the dependent variables showed that there were no grounds for rejecting the fifth hypothesis which stated that there was no significant difference in fabric properties attributable to surfactant build-up.
From the results of this investigation it was concluded that untreated and resin-treated cotton fabrics do not retain large quantities of surfactants. For the little that is retained, temperature and surfactant type are important factors. No relationship exists between the quantity of surfactant on the fabric and the tensile strength, abrasion resistance, and wrinkle recovery of the fabrics; rather, the lubricating action of the surfactant is of more relevance to the abrasion resistance of the fabric.

**RECOMMENDATIONS**

The fabrics were laundered eight times which was less than one-fourth the number of launderings a fabric is expected to go through in a normal wear life. Future research should be conducted over a larger number of launderings to allow for pronounced changes in fabric properties.

The quantity of surfactant retained was small in quantity but fairly large if considered in terms of surface action. Research should be undertaken to determine the effect of this quantity of residual surfactant on the soiling property and the ease of soil removal of the fabrics.
Finally, the effects of concentration and builders were controlled for this study. Research should be undertaken to determine the effects of these on surfactant build-up.
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PERIODICALS (continued)


PERIODICALS (continued)


PERIODICALS (continued)


BOOKS


BOOKS (continued)


"Qualitative Analysis for Crosslinkage and Curing of Resin-Treated Cellulosic Fibers." *Cone Mills R & D Laboratory Microscopic Methods*, 1966.


APPENDIX A
## Canonical Correlations (N=48)

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Wkl. = Wrinkle
APPENDIX B
### MEAN SURFACTANT CONTENT FOR THE VARIABLE SURFACTANT

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**MEAN SURFACTANT CONTENT FOR THE VARIABLE LAUNDERING CYCLE**

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**MEAN SURFACTANT CONTENT FOR SURFACTANT BY TEMPERATURE INTERACTION**

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(N=48)

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*p = .05

Wkl. = wrinkle