INVESTIGATION OF METHODS FOR REMOVAL OF SILICONES FROM NYLON AND ACETATE FABRICS

A THESIS

Presented to
the Faculty of the Graduate Division
Georgia Institute of Technology

In Partial Fulfillment
of the Requirements for the Degree
Master of Science in Textiles

By

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September 1954

INVESTIGATION OF METHODS
FOR REMOVAL OF SILICONES FROM
NYLON AND ACETATE FABRICS

APPROVED: _____

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Date Approved by Chairman: Sept. 10, 1954

ACKNOWLEDGMENTS

In due recognition of the assistance received which contributed to the successful completion of this thesis, a sincere expression of gratitude is offered to the Dow Corning Corporation, whose financial assistance and suggestions made this investigation possible; to Dr. James L. Taylor of the Georgia Institute of Technology A. French Textile School who directed and guided the work performed in this thesis; to the J. P. Stevens Company, Inc., who provided the fabrics used in this work; and to the personnel of the Atlanta Research Laboratory of the E. I. duPont de Nemours and Company, Inc., who provided advice and materials used in the laboratory research phase of this investigation.

TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	v
LIST OF ILLUSTRATIONS	vii
ABSTRACT	viii
CHAPTER	
I. INTRODUCTION	1
II. SPECIAL MATERIAL AND EQUIPMENT	6
III. EXPERIMENTAL PROCEDURE	7
Method of Approach Scouring Strength Tests for Scoured Goods Application of Silicone Water-Repellent Strength Tests for Silicone Treated Goods Resistance of Silicone Water-Repellent to Washing Silicone Removal Treatments Strength Tests for Goods Treated for Silicone Removal Water-Repellency Tests Application of Dyes Evaluation of Dyeing Characteristics Determination of Silicone Content	
IV. RESULTS AND DISCUSSION OF RESULTS OF VARIOUS TREATMENTS FOR SILICONE REMOVAL	24
Soaps Acetic Acid Hydrochloric Acid Sulfuric Acid Phenol Formic Acid	

TABLE OF CONTENTS (Continued)

Phosphoric Acid
Sodium Hydroxide
Aniline
Sodium Carbonate
Tables of Results

V. CONCLUSIONS 47

VI. RECOMMENDATIONS 49

APPENDIX 50

BIBLIOGRAPHY 55

LIST OF TABLES

Table		Page
1.	Numerical Designation of Nylon Samples	29
2.	Numerical Designation of Acetate Samples	30
3.	Effect of Treatments on Silicon Content of Nylon Fabrics	31
4.	Effect of Treatments on Silicon Content of Acetate Fabrics	32
5.	Effect of Treatments on Water-Repellency	33
6.	Effect of Treatments on the Tensile Strength of Nylon Fabrics	34
7.	Effect of Treatments on the Tensile Strength of Acetate Fabrics	35
8.	Effect of Treatments on Shade Characteristics of Nylon using Scoured Sample as Control	36
9.	Effect of Treatments on Shade Characteristics of Acetate using Scoured Sample as Control . . .	37
10.	Light-Fastness of Acetamine Yellow N	38
11.	Light-Fastness of duPont Anthraquinone Violet 3R	39
12.	Light-Fastness of Pontamine Fast Red	40
13.	Light-Fastness of Celanthrene Brilliant Blue FFS	41
14.	Light-Fastness of Acetamine Diazo Black BGD . .	42
15.	Wash-Fastness of Dyes on Nylon	43
16.	Wash-Fastness of Dyes on Acetate	44
17.	Crock-Fastness of Dyed Nylon Fabrics	45
18.	Crock-Fastness of Dyed Acetate Fabrics	46

LIST OF TABLES (Continued)

Table		Page
19.	Determination of Moisture Pickup of Fabrics . . .	51
20.	Calibration of Spectrophotometer using Standard Sample	52
21.	Analysis of Nylon Fabrics for Silicon	53
22.	Analysis of Acetate Fabrics for Silicon	54

LIST OF ILLUSTRATIONS

Figure Page

1. Concentration of Water-Repellent Needed to Apply 2% Silicon at Various Moisture Pickups . . . 11

2. Spectrophotometric Absorption Curve Obtained with Standard Silicon Sample 20

INVESTIGATION OF METHODS FOR REMOVAL OF SILICONES FROM NYLON AND ACETATE FABRICS

ABSTRACT

The purpose of this investigation was twofold: first, to determine whether a Silicone water-repellent could be removed from certain synthetic fabrics; and second, to determine what changes in dyeing characteristics of these fabrics would result from the following treatments: (a) Silicone treated; (b) Silicone partially removed; and (c) Silicone completely removed.

In the first phase of this work two methods were used in attempting to remove the Silicone from the fabric. The Silicone fabrics were treated with reagents that were known to exert swelling action on the particular type fiber. The aim was to swell the fiber and in so doing to crack the Silicone film which surrounded the fiber and thereby to break the film and remove it. The second approach used was to subject the Silicone fabrics to chemical reagents such as hydrochloric or sulfuric acid, sodium hydroxide, or sodium carbonate and to evaluate their effectiveness in removing the Silicone.

After treatments, the fabrics were tested for tensile strength and dyeing characteristics; the results were compared with those obtained on scoured samples. This was done to determine whether the treatment, if successful, had adversely affected any properties of the fabric which might make it unacceptable to the industry.

The Silicone content of the fabric was determined by a colorimetric analysis of siliconolybic acid that had been reduced with sodium sulfite. The siliconolybic acid was formed by fusing the sample in a Parr peroxide bomb and treating the fused residue with hydrochloric acid and ammonium molybdate.

The investigation was successful in discovering a method for completely removing the Silicone from nylon fabrics. Sodium hydroxide effected complete removal of the water-repellent; of the properties studied, only a trace change in the shade characteristics resulted. For the acetate fabrics, the investigation was not entirely successful as the Silicone was only partially removed.

The swelling agents used on the fabrics removed the Silicone partially and the acids were ineffective as removal agents. However, in the preliminary stages of the investigation, it was discovered that a hot alkaline soap wash accompanied with severe mechanical action would remove the Silicone completely from nylon and almost completely from

acetate. These tests were not evaluated for changes in properties other than that of water-repellency.

The conclusions drawn from this work were; that the Silicone was resistant to most acids and not resistant to alkalis; that swelling agents, without the aid of mechanical action, were only partially effective in removing the film; that the process of application and complete removal of a Silicone would not affect the properties or dyeing characteristics of a fabric, provided the reagent used would not affect the fabric; and that the presence of a Silicone film adversely affects the dyeing characteristics of a fabric, if present in a sufficient percentage.

INVESTIGATION OF METHODS FOR REMOVAL OF SILICONES FROM NYLON AND ACETATE FABRICS

CHAPTER I

INTRODUCTION

In this investigation a study was made to determine whether a Silicone water-repellent could be removed from nylon and acetate fabrics without adversely affecting the tensile strength of the fabrics. An additional study was made to ascertain the changes in the dyeing characteristics of nylon and acetate fabrics which had the following treatments: (a) Silicone treated; (b) Silicone partially removed; (c) Silicone completely removed.

From a practical point of view, if a method for the removal of the Silicone water-repellent could be found, which would not adversely affect other properties of the fabric, then the usefulness of the water-repellent would be increased. The Silicone water-repellent might be used as a warp size or as an addition to a warp size, and the industry would be less hesitant in using the water-repellent, for any goods to which the Silicone had been incorrectly applied could be refinished with no loss in money on second rated goods.

The possibility of using the Silicone water-repellent in the warp sizing operation of the textile industry exists because besides imparting water-repellency the product also gives other desirable properties to most synthetic fabrics, such as more resiliency, smoother surface (1), and increased tensile strength for acetate fabrics. It is reported also that the Silicone water-repellent increases the tear strength of most fabrics, some as much as 50 per cent perpendicular to the warp. (2). Studies have shown that in the application of the Silicone water-repellent, a film is formed which completely surrounds the fibers (3) and this film increases the abrasion resistance of these fibers (4) and should protect the filaments during the manufacturing processes.

Literature.--The history of commercial Silicones does not date beyond the early 1940's, while their use as a textile finish began in 1948 when it was discovered that an oil-in-water emulsion of linear methyl silicone polymers which have labile hydrogen-silicon bonds could be prepared. The emulsion could be padded on the fabrics and then through the use of moderate heat, the hydrogen-silicon bonds could be broken with the hydrogen being liberated and the compound cross linking and thereby becoming insoluble (5). This discovery led to the Silicone water-repellents as known today for use on textile fabrics.

The early work with Silicone water-repellents for textile fabrics began in 1940 when Patnode (6) (7) discovered that the vapor phase of methylchlorosilanes would react with many surfaces, among these being cellulosic material, to impart permanent water-repellency which was resistant to all but strong reagents, such as hydrofluoric acid. While this discovery increased the usefulness of Silicones, it was not readily acceptable to the textile industry because of the method of application and the hydrogen chloride which was liberated during the reaction of the methylchlorosilane. This acid had to be neutralized with gaseous ammonia.

Schuyten, et al., (8) in discussing Patnode's discovery, suggested the following type reaction of the methylchlorosilane with water:

$$3R_2SiCl_2 + 6H_2O \longrightarrow 3R_2Si(OH)_2 + 6HCl \longrightarrow \quad (1)$$
$$HOSiR_2OSiR_2OSiR_2OH + 2H_2O$$

They stated that it was reasonable to expect the above reaction could take place with the absorbed moisture carried in the fibers. They went further and suggested that the Silicone compound as described by Patnode could react with the hydroxyl groups of cellulose or the amino groups of animal fibers in the following manner:

$$R_3SiCl + HO\text{-cellulose} \longrightarrow R_3SiO\text{-cellulose} + HCl \quad (2)$$

$$R_3SiCl + NH_2\text{-wool} \longrightarrow R_3SiNH\text{-wool} + HCl \quad (3)$$

However, Pingree (9) in his article on water-repellency of textiles, stated that Patnode's reaction had not proved practical to the industry.

The work showed that Silicones had a future in the textile finishing industry if a more suitable method of application could be devised and undoubtably led to the Silicone emulsion products now available.

Fortess and Wood (10) and Harding (11) have discussed the preparation of these products and the method of application of the end products to fabrics. Briefly, the manufacture consists in the production of silicon tetrachloride from sand coke, and brine, then replacing one or more of the chlorine atoms with organic radicals by use of the Grignard Reaction.

$$SiCl_4 + 2RMgCl \longrightarrow R_2SiCl_2 + 2MgCl_2 \qquad (4)$$

This is hydrolyzed to a silanol which is unstable and condenses to form siloxanes.

$$nR_2SiCl_2 + 2nH_2O \rightarrow nR_2Si(OH)_2 \rightarrow \left[\begin{matrix} R \\ -Si\,O \\ R \end{matrix}\right]_n + 2nH_2O \qquad (5)$$

By use of the Grignard Reaction the following can be prepared:

$(CH_3)HSiCl_2$ --- (methylhydrogendichlorosilane) (6)

$(CH_3)_2SiCl_2$ --- (dimethyldichlorosilane) (7)

$(CH_3)_3SiCl$ --- (trimethylchlorosilane) (8)

By use of the above hydrolysis-condensation reaction (5) on equal parts of compounds (6) and (7) with a small amount of the chain stopping compound (8), the following linear polymer is prepared:

$$(CH_3)_3Si-\left(\begin{matrix}CH_3\\OSi\\H\end{matrix}-\right)_n\left(\begin{matrix}CH_3\\OSi\\CH_3\end{matrix}-\right)_n OSi(CH_3)_3 \qquad (9)$$

This polymer (9) contains the hydrogen-silicon bonds which can be oxidized at elevated temperatures or in the presence of a catalyst to produce an insoluble, three dimensional polymer.

A survey of the literature has revealed that no attempts have been made to use Silicone water-repellent for any use other than imparting water-repellency.

CHAPTER II

SPECIAL MATERIAL AND EQUIPMENT

A. Fabrics.--

1. Nylon, 110 x 80, plain weave, (filament)
2. Acetate, 172 x 60, plain weave, (filament)

B. Equipment.--

1. Smith-Drum Hosiery Dyeing Machine, Model 12-RD, capacity 15 pounds.
2. Butterworth Laboratory Padder
3. Constant Temperature Oven
4. Wiley Laboratory Mill, Intermediate Model, No. 4376M.
5. Parr Flame Ignition Peroxide Bomb.
6. Beckmann Spectrophotometer, Model B.

CHAPTER III

EXPERIMENTAL PROCEDURE

For this investigation a polymer of the above type (9), Dow Corning's DeCotex 104 Emulsion, was used in conjunction with the Catalyst XEY-16.

Method of Approach

The Silicone water-repellent was applied to the nylon and acetate fabrics and tests were made to check the durability of the Silicone to washing. The samples were given spray tests after each washing. These tests showed the spray rating of the acetate fabrics decreased more rapidly than those of the nylon fabrics. Since acetate swells more easily than nylon it was indicated that the Silicone water-repellent was either being removed or the fibers were swelling and the film was being cracked during the washing process. Therefore, one of the approaches used in attempting to remove the Silicone was to treat the fabrics with known swelling agents and to determine what loss, if any, in Silicone resulted. The other approach was to treat the fabrics with various chemicals which are widely used in the textile finishing industry and to measure the Silicone content after each treatment. All samples were then dyed to determine the changes in dyeing characteristics; a scoured untreated sample was used as a control.

Scouring

The fabrics were received in the grey state and prior to scouring were cut into strips eight and one-half inches wide (a suitable width for passing through the padding machine), and approximately five yards long.

The scouring operation was carried out in a Smith-Drum Rotary Hosiery dyeing machine that revolved at the rate of eight revolutions per minute and reversed direction every two and one-half revolutions. The bath contained two per cent Du Ponol WA flakes, two per cent neutral soap and one per cent tri-sodium phosphate, based on the weight of the goods, and a volume of 23 gallons of water. The scouring was carried out at 150°F for 30 minutes.

After scouring, the goods were given three five-minute rinses with fresh water at 150°F and one ten-minute rinse with running water at 100-105°F. The goods were then hydro-extracted and pressed with an electric iron. This was followed by a 24-hour conditioning at 65 per cent relative humidity and 70°F.

Strength Test for Scoured Goods

After conditioning, samples of the scoured goods were given tests for physical strength. The testing was carried out in accordance with the Grab Method of the American Society for Testing Materials, Standard Test D39-49. (12)

Application of Silicone Water-Repellent

The Silicone emulsion, as recommended by the Dow-Corning Corporation (13), was prepared by mixing five parts by weight of DeCetex 104 Emulsion with one part by weight of Catalyst XRY-16. The mixture was then diluted to the necessary concentration so that a two per cent application of the water-repellent would be deposited on the fabric. (See Fig. 1). The necessary concentration was determined from the moisture pickup by assuming that the water-repellent had no affinity for the fabric but was merely carried along with the water.

The moisture pickup of the fabrics was determined by conditioning samples of the fabrics at 65 per cent relative humidity and 70°F for 24 hours, weighing them accurately on an analytical balance and wetting them thoroughly with water. The samples were then passed through a laboratory padder to remove all excess moisture and were immediately reweighed on the same balance with the increase in weight being considered as the moisture pickup. This procedure was repeated once for each type fabric and the results were averaged. (See Table 19 in Appendix.)

Prior to treating the cloth with the Silicone emulsion, each type fabric was sewed end to end so that a long continuous run through the padder could be made. A leader of cotton cloth was used to thread the sample through the padder.

The Silicone emulsion was applied at a temperature of 76°F and pH of 5 to the fabric by passing the sample through the padder at a constant speed and at a pressure of 59 p.s.i. The pad was filled with the emulsion in a concentration calculated to give two per cent pickup (See Fig. 1). The per cent was calculated as weight of silicon on conditioned weight of fabric. Two passes were made with two dips and two nips per pass to insure maximum and uniform penetration of the Silicone. After application the samples were air dried and placed on a rack in an oven in a loop fashion and were cured for eight minutes at 300-303°F.

Strength Test for Silicone Treated Goods

After the curing operation was completed, samples of the Silicone treated goods were conditioned at 65 per cent relative humidity and at 70°F for 24 hours for breaking strength test. Again the Grab Method was used with the recommended five breaks being made and averaged. The results were compared with those obtained for the scoured goods and the effect of the Silicone water-repellent on the physical strength of the material noted. (See Tables 6 and 7.)

Resistance of the Silicone Water-Repellent to Washing

Samples of the Silicone treated goods were given wash fastness test in accordance with American Association of Textile Chemists and Colorists, (AATCC), Standard Test Method 36-45 (14), Test No. 3, so that the durability of

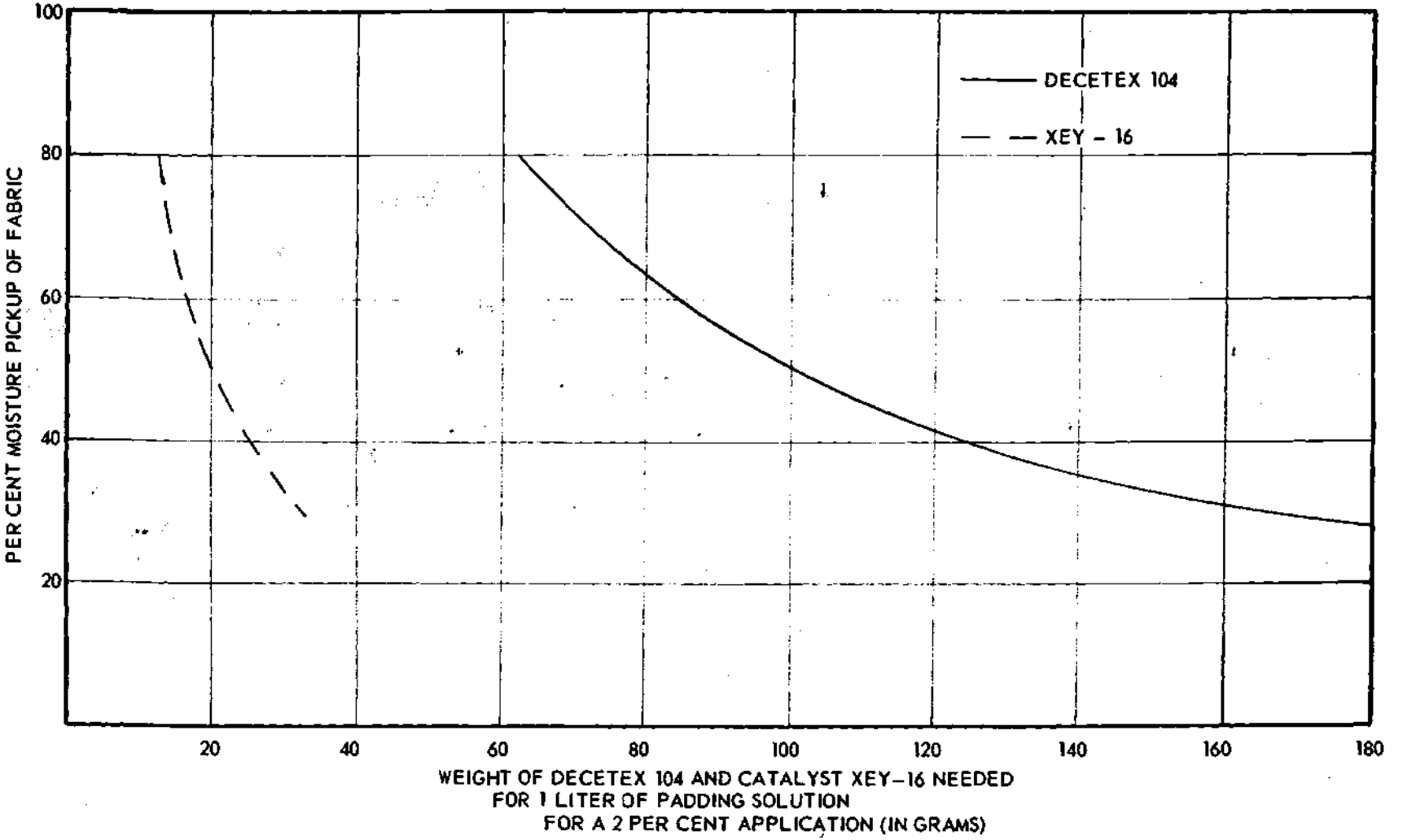

Figure 1. Concentration of Water-Repellent Needed to Apply 2% Silicon at Various Moisture Pickups.

the water-repellent to washing could be established. Five washes were made in testing the durability, with each wash consisting of treating samples for 45 minutes at 160°F with a solution of 0.5 per cent neutral soap and 0.2 per cent sodium carbonate. After each washing, two rinses with water at 105°F, followed by a sour with a solution of 0.05 per cent acetic acid at 80°F and a final rinse with water at 80°F were made. Each rinse and sour were accompanied with vigorous shaking for one minute. Deviations in the test procedure were made in that samples eight by eight inches instead of recommended size of two by four inches, were washed and all recommended volumes (200 ml. for each step) were doubled. The increase in sample size was necessary in order to be able later to subject the samples to spray test.

Silicone Removal Treatments

The reagents for these treatments may be divided into two classes, those which are normally thought of as swelling agents for the particular fiber, and those which are normally thought of merely as chemical reagents.

The swelling agents and chemical reagents used for nylon were some of those listed by the DuPont Company in their Technical Manual on Nylon (15).

The swelling agents used were:

1. Formic acid
2. Acetic acid
3. Phenol
4. Aqueous phosphoric acid
5. Phosphoric acid in methanol

The chemical reagents used were:

1. Sodium hydroxide
2. Sulfuric acid
3. Hydrochloric acid

(For a listing of concentration, time, and temperature, see Table 1.)

The swelling agents used for Silicone treated acetate were some of those suggested in work done by Marsden and Urquhart (16), in which they studied the per cent swelling caused by certain phenolic compounds on cellulose acetate sheets. The swelling agents used in this investigation in attempting to remove the Silicones were:

1. Phenol
2. Phenol and sodium sulfate
3. Phenol and sodium peroxide
4. Phenol and hydrochloric acid
5. Aniline

The chemical reagents used were:

1. Acetic acid
2. Hydrochloric acid
3. Sulfuric acid
4. Sodium carbonate

(For listing of concentration, time, and temperature, see Table 2.)

Treatments were carried out by immersing a sample, 8 by 72 inches, in one liter of solution. The sample was turned slowly through the solution so that all surfaces

would come in contact with the reagent and a uniform treatment would result. For acetate treatments, a wetting agent was used in addition to the reagent as it was discovered the reagents were not wetting the surface of the fabric. (See Table 2.)

The goods were then treated either with a two per cent solution of acetic acid or a two per cent solution of sodium carbonate, depending on the type of solution the fabric was treated with in the above step. The goods were given a thorough warm and cold water rinse and pressed with an electric iron.

Strength Tests for Goods Treated for Silicone Removal.

The same procedure as previously mentioned on page 10 was followed.

Water Repellency Tests

Upon completion of the Silicone removal treatment, samples were conditioned at 65 per cent relative humidity and 70°F for 24 hours. They were then given water-repellency tests in accordance with AATCC Standard Test Procedure 22-41 (17), and the samples were rated accordingly.

Application of Dyes

In order to properly evaluate the effect of the various treatments on the dyeing characteristics of the fabrics, it was thought advisable to use different classes of dyes in different colors on each type fabric. The dyes used for each material were:

Material	Class	Color	Manufacturer's Trade Name
Nylon	Dispersed Acetate	Yellow	Acetamine Yellow N
Nylon	Acid	Violet	DuPont Anthraquinone Violet 3R
Nylon	Direct	Red	Pontamine Fast Red 150% conc.
Acetate	Dispersed Acetate	Blue	Celanthrene Brilliant Blue FFS, 200% conc.
Acetate	Diazo	Black	Acetamine Diazo Black BGD

The procedures used were those recommended by the dye manufacturer (18), (19), and varied with the type dye used. In all cases, samples were cut to eight by eight inches in size, accurately weighed and were dyed in a bath volume of 300 ml. After dyeings were completed, the goods were rinsed in cold and warm water, followed by a wash with neutral soap and a final warm and cold water rinse. After rinsing, the goods were pressed with an electric iron. The individual procedure used for each dye varied in the following manner.

Acetamine Yellow N.--A 1.0 per cent dye dispersion, based on the weight of the fabric (owf), was prepared with 1.0 per cent DuPonol WA flakes, 2.0 per cent neutral soap, and 0.5 per cent tri-sodium phosphate. The weights of dispersing agents were based on weight of dye. The goods were entered at 140°F, the temperature was raised to 180°F over a period of 30 minutes, and dyeing was continued for 45 minutes at 180-185°F.

DuPont Anthraquinone Violet 3R and Pontamine Fast Red.--For these dyeings, 2.0 per cent dye (owf) was applied to the fabrics, with the dye being dispersed with 0.5 per cent DuPonol WA flakes, based on the weight of dye. The goods were entered at 140°F and the temperature was raised to the boil in 15 minutes, where it was maintained for 35 minutes for the Pontamine Fast Red and for 45 minutes for the Anthraquinone Violet 3R. For the Pontamine Fast Red, 5.0 ml. of 2.0 per cent acetic acid solution were added 5, 15, and 30 minutes after the temperature reached the boil and for the Anthraquinone Violet 3R, 5.0 ml. of 2.0 per cent acetic acid were added 10, 20, and 35 minutes after the bath temperature reached the boil.

Celanthrene Brilliant Blue FFS.--The dye dispersion was prepared by pasting the dye with hot water in a ratio of one to sixteen and 0.006 per cent of neutral soap and 0.004 per cent of DuPonol WA flakes. A 2.0 per cent dye, based on the weight of the goods, was applied by entering the goods at a temperature of 112°F, raising the temperature to 180°F in 15 minutes, and continuing the dyeing process for 60 minutes at 180-185°F.

Acetamine Diazo Black BGB.--The dye dispersion was prepared in the same manner as with the Celanthrene Brilliant Blue FFS. The goods were entered at a bath temperature of 102°F, and the temperature was raised to 180°F in 15 minutes, and dyeing was continued for 60 minutes. The samples were

rinsed and treated for 30 minutes at 70°F in a bath containing 5.0 per cent sodium nitrite and 10.0 per cent of 32°Tw hydrochloric acid (owf). After the diazotization treatment, the goods were thoroughly rinsed and developed with 3.0 per cent Acetamine Developer AD Extra. The developer was dispersed with a small amount of hot water and 2.5 per cent of 28.0 per cent acetic acid with the same amount of acetic acid being added to the development bath. The goods were entered in the bath after the temperature had been raised to 104°F and the bath temperature was then raised to 140°F in 15 minutes where development was continued for 30 minutes. The goods were then given the usual rinse and soaping operations.

Evaluation of Dyeing Characteristics

For an evaluation of the dyeing characteristics of the treated samples, four properties were studied; shade differences, light-fastness, wash-fastness, and crock-fastness. These properties were compared with those obtained on the scoured sample which was considered as the control sample.

Shade Differences.--For an evaluation of the shade differences, the services of the Technical Laboratory, Atlanta Service Division of the DuPont Company were used so that an unbiased and professional opinion could be obtained. The shades were compared visually using a natural northern light. (See Tables 8 and 9).

Light-Fastness.--The dyed samples were exposed in a FDA-R Fade-Ometer for a period of 12, 24, 36, and 48 hours in accordance with the AATCC Tentative Test Method 16-45, with the temperature set at 105°F (20). Each sample was rated individually and the results were compared with the control sample. (See Tables 10-14.)

Wash-Fastness.--For testing fastness of the dyes and samples to washing, the procedure recommended by the AATCC Standard Test Method 36-45, Test No. 3, was used. This process used a soap solution of 0.5 per cent neutral soap and 0.2 per cent sodium carbonate at 160°F for 45 minutes per washing. The results were recorded and compared with those obtained on the control sample. (See Tables 15 and 16.)

Crock-Fastness.--The dye samples were tested for crock-fastness by using the AATCC Standard Test Method 8-45 (21). For evaluation of the crock-fastness of the samples, the results were compared with the control sample and rated. (See Tables 17 and 18.)

Determination of Silicon Content

The determination of Silicon content of the fabrics was accomplished by using methods described by McHard, et al., (22), the Parr Instrument Company, (23), and the Dow Corning Corporation (24). The equipment used for this phase of the investigation was a 22 ml. flame ignition macro peroxide bomb, Parr type, in which all samples were fused. Firing of the bomb was accomplished using an oxygen, air, and natural

gas burner. The flame was adjusted so that the bottom of the fusion cup became a dull red 30-35 seconds after the flame was applied to the bomb.

In order to determine the Silicon content of the samples, it was necessary to fuse a sample of known Silicon content. The sample used was Bureau of Standards' Feldspar sample No. 99, which contained 68.66 per cent Silicon oxide (SiO_2). After fusion, known concentrations were analyzed.

Analysis was accomplished colorimetrically by measuring the blue color produced when silicomolybic acid was reduced with sodium sulfite. The developed color was measured with a Beckmann Model B Spectrophotometer and at a wave length of 715 millimicrons. A calibration curve was obtained by using samples of varying but known concentrations and the per cent light transmitted was plotted against milligrams of Silicon. (See Fig. 2.)

Calibration.--The Feldspar sample was accurately weighed to 0.0146 grams, an amount which would give a final concentration of 0.01 milligrams per milliliter after the sample was fused and diluted to one liter. This sample was mixed in the bottom of the fusion cup with 2.0 grams of sodium peroxide and 0.07 grams of confectioners sugar, and then one dipper, with a capacity of 15 grams, of sodium peroxide was added to the fusion cup. After securing the cover, the bomb was placed in an ignition housing and fired as mentioned above. The flame was removed after 35 seconds and the reaction allowed to continue for 20 seconds. The bomb was

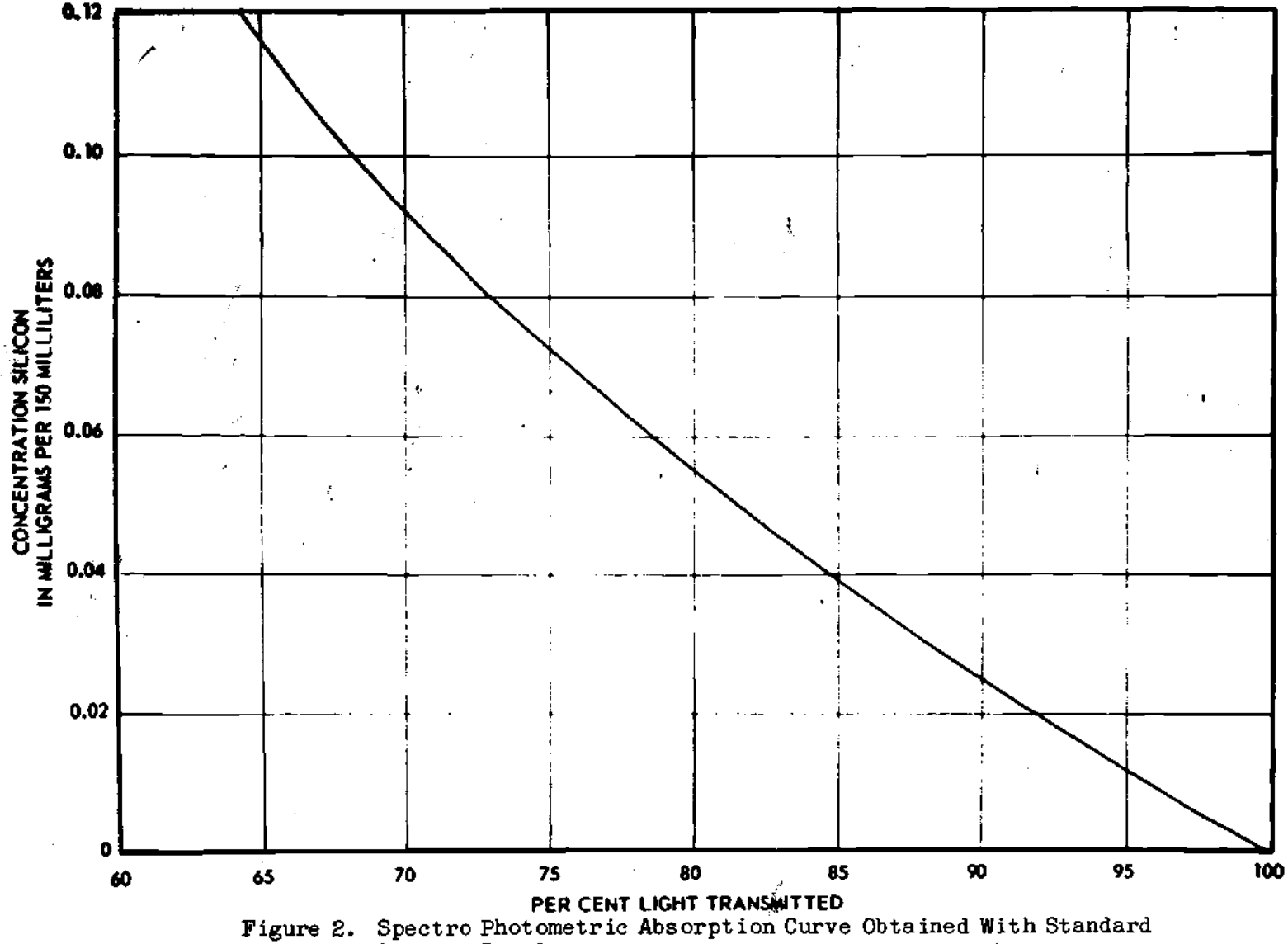

Figure 2. Spectro Photometric Absorption Curve Obtained With Standard Silicon Sample.

then placed in a bucket of cold water after which the entire outer area of the bomb was rinsed with distilled water to remove any impurities present, and the screw cap and cover were removed. The bomb cover was rinsed with 100 ml. of distilled water and the rinse water was collected in a 500 ml. monel beaker. The fusion cup was then dropped into the beaker and a stainless steel cover was placed over the beaker while the fused mass dissolved. After the peroxide was dissolved, the cup was removed from the beaker with tongs and all surfaces were rinsed with distilled water with the rinse water being collected in the monel beaker. The inside of the fusion cup was rinsed with 10 ml. of concentrated hydrochloric acid and all the inside surfaces of the fusion cup were carefully brought into contact with the acid. The acid was added to the monel beaker and the solution was cooled to 50°F. After the solution was cooled, 40 ml. of concentrated hydrochloric acid were added. Again the solution was cooled. At this point a test was made with litmus paper to check for the acidity of the solution. (The test should be distinctly positive.) The solution was transferred to a 1000 ml. volumetric flask, the volume was increased to approximately 975 ml. with distilled water, and the solution was allowed to stand for two hours before increasing the volume to 1000 ml. The solution was then thoroughly mixed.

Portions of the solution containing 0.02, 0.04, 0.06, 0.08, 0.10, and 0.12 milligrams of silicon were transferred to 250 ml. Erlenmeyer flasks and diluted to 50 ml. with distilled water. To each solution 25.0 ml. of 1.0 N hydrochloric acid were added, mixed, and then 25.0 ml. of 10.0 per cent ammonium molybdate, freshly prepared and stored in a monel beaker, were added and mixed. Two minutes after the start of the addition of the ammonium molybdate, 50.0 ml. of 17.0 per cent sodium sulfite were added, mixed thoroughly, and then transferred to a 40 ml. photometer cell. Seven minutes after the start of the addition of sodium sulfite transmission readings were made with the spectrophotometer. (For this investigation the instrument was adjusted to 715 millimicrons and the adjustment was not changed at any time during use.) Just prior to reading the per cent transmission of light through the sample, the spectrophotometer was adjusted to read 100 per cent transmission for a blank sample that had been prepared in exactly the same manner as the standard sample and contained the same size aliquot. The blank sample contained no silicon. Two samples of the standard sample were fused and the results compared. (See Table 20.)

<u>Analysis of Samples</u>.--In the analysis of the fabrics for silicon content, each sample was first ground in a Wiley Laboratory Mill and passed through a 20 mesh screen. This step was necessary to insure that an adequate surface would come into contact with the sodium peroxide and thereby fuse

properly. The samples were accurately weighed to approximately 0.23 grams. Thereafter, the procedure used for fusing the standard sample was followed.

In determining the silicon content, a 10.0 ml. aliquot was withdrawn from the 1000 ml. volumetric flask and the procedure above for the standard sample was used.

For calculating the per cent silicon content, the following formula supplied by the Dow Corning Corporation was used: (25)

$$\text{Per cent Silicon} = \frac{\frac{(\text{mg. Si})}{(\text{ml. Aliquot})}}{\text{Sample weight in grams}} \times 100$$

To convert the per cent silicon to per cent Silicone applied, a multiplication factor of 2.34 is used. The factor varies with each type Silicone and the factor 2.34 applies only to DeCetex 104 Emulsion.

These data were also obtained for the scoured samples of both nylon and acetate so that the silicon originally contained in the fabrics could be subtracted from the amount determined for each of the treated samples and thereby the true amount of silicon applied or removed could be determined.

CHAPTER IV

RESULTS AND DISCUSSION OF RESULTS OF VARIOUS TREATMENTS FOR SILICONE REMOVAL

The results of this investigation are presented according to the reagent used to remove the Silicone water-repellent. All comparisons of fabric properties were made by using the scoured untreated samples as the control samples.

Soap.--The washing test showed that when the Silicone treated fabrics were washed in an alkaline soap bath at a high (160°F) temperature and severe mechanical action (constant tumbling of 10 steel balls), the Silicone water-repellent is not permanent. These wash tests are considered to be in excess of the normal type washing that would be given these fabrics in everyday use. The Silicone was more durable to acetate than to nylon, as determined in the analysis for silicon content. However, the spray ratings of the nylon remained consistently higher than those for acetate. This apparent contradiction to what would be expected may be due to residual wetting agents that adhered to the acetate fabrics and thereby caused re-wetting when the spray tests were conducted. (See Tables 2, 3, and 5.)

These samples were not evaluated for strength or dyeing properties.

Acetic Acid.—The use of acetic acid in a concentration of three per cent and at a temperature of 150°F for acetate and at the boil for nylon was found to be ineffective in removing the Silicone. The 100 per cent acetic acid at 77°F on nylon removed 79.4 per cent of the silicon.

The dyeing characteristics of the samples treated with acetic acid were generally found to have noticeable shade differences with the three per cent treatments and only trace differences with the 100 per cent acetic acid treatment on nylon. The fastness properties of these samples were found to be less than the control sample. (See Tables 8-18.)

This treatment caused no apparent loss in tensile strength. (See Tables 6 and 7.)

Sulfuric Acid.—The sulfuric acid when used at a 10 per cent concentration and at a temperature of 77°F removed 59.1 per cent of the silicon from nylon and only 8.4 per cent from the acetate.

The acid caused approximately 25 per cent loss in strength of the fabrics, except for the acetate filling which showed no apparent change in strength. (See Tables 6 and 7.)

The shade differences were noticeable on both fabrics and the fastness properties less on the nylon and slightly more on the acetate. (See Tables 8-18.)

Phenol.—This reagent at a two per cent concentration and at a temperature of 77°F removed 67.8 per cent of the silicon from nylon and was relatively ineffective on the acetate, even when sodium sulfite, sodium hydroxide, or hydrochloric acid in equivalent amounts were used in combination with the phenol. However, when the temperature was increased while treating the acetate the per cent silicon removed increased to as much as 36.2 per cent. (See Tables 3 and 4.)

The phenol had no effect on the strength of either fabric when used at 77°F, but at 112°F on acetate caused a small loss in tensile strength of the filling and at 140°F caused a great loss, both for warp and filling. (See Tables 6 and 7.)

The dyeing characteristics of the samples treated with phenol showed noticeable shade differences on the nylon and trace differences on the acetate, with the treatment at 112°F on acetate showing no shade differences. (See Tables 8 and 9.)

The fastness properties of dyed nylon after treatment with phenol were generally less to light, the same to washing, and weaker to crocking. For acetate, this reagent adversely affected the light-fastness of the dyes only slightly; it improved the wash-fastness of the dyed fabric, and slightly impaired the crock-fastness. (See Tables 13, 14, 16, and 18.)

Formic Acid.--This acid was used on nylon fabrics at concentrations of three and 20 per cent. It proved to be ineffective as a Silicone removal agent.

The samples showed noticeable shade differences in dyeing and had weaker light-, wash-, and crock-fastness properties. (See Tables 8, 10-12, 15, and 17.)

The acid had no apparent effect on the tensile strength of the nylon. (See Table 6.)

Phosphoric Acid.--Nylon was treated with this acid at a concentration of 20 per cent and at a temperature of 77°F in water and methanol solutions. When used in a water solution, it removed 46.1 per cent of the silicon and in methanol, 75.6 per cent.

This treatment on nylon showed appreciable to noticeable shade differences. The fastness properties of the fabric were generally less on all tests for both methods of treatment. (See Tables 8, 10-12, 15, and 17.)

The acid caused no apparent loss in strength filling-wise and a slight loss warp-wise. (See Table 6.)

Sodium Hydroxide.--Nylon was treated with 10 per cent sodium hydroxide at 180°F for periods of 15, 30, and 60 minutes. In all cases the sodium hydroxide completely removed the Silicone water-repellent. When treated for 60 minutes, the sodium hydroxide caused a slight loss in tensile strength and no change in dyeing characteristics. When treated for 15 and 30 minutes, the nylon showed no change in tensile

strength, only trace changes in shade characteristics, and trace or no changes in fastness properties. (See Tables 6, 8, 10-12, 15, and 17.)

Aniline.--A 1.0 per cent solution of this reagent at a temperature of 77°F was used on acetate fabrics and removed only 19.5 per cent of the silicon.

The samples treated with aniline showed slight loss in tensile strength, trace changes in shade characteristics, and trace changes in wash- and light-fastness properties with the dispersed acetate dye. The crock-fastness properties were appreciably weaker. With the black dye, the samples showed stronger light-fastness properties. The wash-fastness properties for this dye were as good or better than the control sample, and the crock-fastness properties were noticeably less. (See Tables 7, 9, 13, 14, 16, and 18.)

Sodium Carbonate.--This reagent at a 10 per cent concentration and at 150°F was used on the acetate fabrics and removed only 14.8 per cent of the silicon. However, this reagent reduced the spray rating to zero.

The sample dyed with the dispersed acetate dye showed less light-fastness properties and with the diazo dye showed more light-fastness properties than the control sample. There was no change in wash-fastness properties. The samples showed less crock-fastness properties. This reagent caused no apparent loss in tensile strength. (See Tables 7, 9, 13, 14, 16, and 18.)

Table 1. Numerical Designation of Nylon Samples

Sample Number	Material (Sample number) Treated	Treatment	Concentration* %	Time (min)	Temperature (°F)
101	-	Scoured	-	-	-
102	101	Silicone**	-	-	-
103	101	Silicone**	-	-	-
104	102	Soap (one washing)	-	45	160
105	102	Soap (two washings)	-	45	160
106	102	Soap (three washings)	-	45	160
107	102	Soap (four washings)	-	45	160
108	102	Soap (five washings)	-	45	160
109	102	Formic acid	3.0	30	212
110	102	Acetic acid	3.0	30	212
111	102	Hydrochloric acid	10.0	10	77
112	102	Sulfuric acid	10.0	10	77
113	102	Sodium hydroxide	10.0	60	180
114	102	Sodium hydroxide	10.0	15	180
115	102	Sodium hydroxide	10.0	30	180
116	103	Formic acid	20.0	30	77
117	103	Acetic acid	100.0	30	77
118	103	Phenol	2.0	30	77
119	103	Phosphoric acid in water	20.0	30	77
120	103	Phosphoric acid in methanol	20.0	30	77

* All percentages are based on solutions.
** See Table 3 for amount of silicon applied.

Table 2. Numerical Designation of Acetate Fabrics

Sample Number	Material (sample number) Treated	Treatment*	Concentration** (%)	Time (min)	Temperature (°F)
201	-	Scoured	-	-	-
202	201	Silicone***	-	-	-
203	201	Silicone***	-	-	-
204	202	Soap (one washing)	-	45	160
205	202	Soap (two washings)	-	45	160
206	202	Soap (three washings)	-	45	160
207	202	Soap (four washings)	-	45	160
208	202	Soap (five washings)	-	45	160
209	202	Aniline	1.0	30	77
210	202	Hydrochloric acid	10.0	10	77
211	202	Sulfuric acid	10.0	10	77
212	203	Acetic acid	3.0	30	150
213	203	Phenol	2.0	60	77
214	203	Phenol and	2.0	60	77
		sodium sulfate	1.5		
215	203	Phenol and	2.0	60	77
		sodium hydroxide	0.8		
216	203	Phenol and	2.0	60	77
		hydrochloric acid	0.8		
217	203	Phenol	2.0	60	77
218	203	Phenol	2.0	60	77
219	203	Sodium carbonate	10.0	60	77

* 25 ml. of 1 per cent Merpol C added as wetting agent for samples 209 through 219
** All percentages are based on solutions
*** See Table 4 for amounts of silicon applied

Table 3. Effect of Treatments on Silicon Content of Nylon Fabrics

Sample number	Silicon applied (mg.)	Silicon applied (%)	Silicon content after treatment (mg.)	Silicon content after treatment (%)	Corrected silicon content after treatment* (%)	Silicon removed (%)
101	0.40	0.17	-	-	-	-
102	4.75	2.10	-	-	1.93	-
103	4.60	1.97	-	-	1.80	-
104	4.75	2.10	2.40	1.03	0.86	45.5
105	4.75	2.10	0	0	0	100.0
106	4.75	2.10	0	0	0	100.0
107	4.75	2.10	0	0	0	100.0
108	4.75	2.10	0	0	0	100.0
109	4.75	2.10	9.35	3.72**	3.55	0
110	4.75	2.10	5.30	2.20**	2.03	0
111	4.75	2.10	5.18	2.26	2.09	0
112	4.75	2.10	2.30	0.96	0.79	59.1
113	4.75	2.10	0	0	0	100.0
114	4.75	2.10	0	0	0	100.0
115	4.75	2.10	0	0	0	100.0
116	4.60	1.97	4.00	1.75	1.58	12.2
117	4.60	1.97	1.25	0.54	0.37	79.4
118	4.60	1.97	1.80	0.75	0.58	67.8
119	4.60	1.97	2.45	1.04	0.97	46.1
120	4.60	1.97	1.45	0.61	0.44	75.6

* Corrected for the amount (0.17%) of silicon present in the original fabric
** The source of this discrepancy was not determined.

Table 4. Effect of Treatments on Silicon Content of Acetate Fabrics

Sample number	Silicon applied (mg.)	Silicon applied (%)	Silicon content after treatments (mg.)	Silicon content after treatments (%)	Silicon removed (%)
201	-	-	-	-	-
202	4.45	1.91	-	-	-
203	4.70	2.10	-	-	-
204	4.45	1.91	2.82	1.23	35.0
205	4.45	1.91	2.28	0.97	49.3
206	4.45	1.91	1.64	0.69	63.8
207	4.45	1.91	0.80	0.34	82.2
208	4.45	1.91	1.30	0.55	71.2
209	4.45	1.91	3.85	1.54	19.5
210	4.45	1.91	3.70	1.64	14.1
211	4.45	1.91	4.38	1.75	8.4
212	4.70	2.10	6.31	2.82*	0
213	4.70	2.10	3.62	1.57	25.3
214	4.70	2.10	4.00	1.73	16.3
215	4.70	2.10	4.45	1.91	8.5
216	4.70	2.10	5.05	2.14	0
217	4.70	2.10	3.25	1.37	36.8
218	4.70	2.10	3.40	1.46	30.5
219	4.70	2.10	2.98	1.79	14.8

* The source of this discrepancy was not determined.

Table 5. Effect of Treatments on Water Repellency

Nylon		Acetate	
Sample number	Spray rating	Sample number	Spray rating
101	0	201	0
102	100	202	100
103	100	203	100
104	90	204	70
105	90	205	50
106	70	206	50
107	50	207	0
108	50	208	0
109	80	209	70
110	90	210	80
111	90	211	70
112	80	212	80
113	0	213	90
114	0	214	90
115	0	215	70
116	100	216	80
117	90	217	90
118	70	218	90
119	80	219	0
120	70		

Classification.-- 100 No sticking or wetting of upper surface
90 Slight random sticking or wetting of upper surface
80 Wetting of upper surface at spray points
70 Partial wetting of whole of upper surface
50 Complete wetting of whole of upper surface
0 Complete wetting of whole of upper and lower surfaces

Table 6. Effect of Treatments on the Tensile Strength of Nylon Fabrics

Sample number	Tensile Strength. Average Results of Five Breaks			
	Warp (lbs.)	Warp strength remaining (%)	Filling (lbs.)	Filling strength remaining (%)
101	92.5*	-	77.9*	-
102	93.4	100.9	77.4	99.4
109	94.8	102.5	86.4	111.0
110	92.4	99.9	84.8	109.0
111	95.2	102.8	81.4	104.4
112	69.6	75.3	58.8	75.5
113	90.2	97.5	70.6	90.6
114	92.8	100.3	77.6	99.6
115	92.2	99.6	83.4	103.0
116	100.6	108.8	83.4	107.0
117	95.8	103.7	89.2	114.5
118	102.0	110.2	83.2	106.9
119	89.2	96.5	79.4	101.9
120	85.8	92.8	81.4	104.4

Note.--Method; ASTM, D39-49, Grab method.

* Total of 16 breaks made for average of control samples.

Table 7. Effect of Treatments on the Tensile Strength of Acetate Fabrics

Sample number	Tensile Strength. Average Results of Five Breaks			
	Warp (lbs.)	Warp strength remaining (%)	Filling (lbs.)	Filling strength remaining (%)
201	23.5*	-	25.0*	-
202	29.0	123.3	30.0	120.0
209	20.2	86.0	23.2	92.8
210	23.0	97.0	24.2	96.8
211	20.0	85.0	26.8	107.2
212	24.4	103.9	26.6	106.3
213	24.6	104.8	26.6	106.3
214	25.0	106.4	24.4	37.5
215	27.8	118.2	25.0	100.0
216	23.6	100.6	25.2	100.8
217	25.4	108.2	19.2	76.4
218	6.0	25.4	4.6	18.4
219	24.4	103.9	27.2	108.9

Note.—Method: ASTM, D39-49, Grab method.

* Total of 10 breaks made for average on control samples.

Table 8. Effect of Treatments on Shade Characteristics of Nylon Using Scoured Sample as Control

Sample number	Dye: Acetamine Yellow N	Dye: DuPont Anthraquinone Violet 3R	Dye: Pontamine Fast Red
103	3 W, G	5-4 Bl	4 W
109	3 W	5	4 W
110	3 W	5	4,3 W
111	3 W	5	4 W
112	3 W	5-4 R	4, 3 W
113	5	-	-
114	5-4 R	5-4 Bl	5-4 D
115	5-4 R	5-4 Bl	5-4 D
116	4 W	5	3 W
117	5-4 W, 4 G	5	5-4 Y, B
118	4 W	5	3 Y
119	3 W, G	5	3 W
120	3 W	5-4 Bl	4 W

Classification.— 5 Very good - No shade difference
5-4 Very good - Trace shade difference
4 Good - Noticeable shade difference
3 Fairly good - Appreciable shade difference
2 Moderate - Considerable shade difference
1 Poor - Very much shade difference

B - Brighter
Bl- Bluer
D - Duller
G - Greener
J - Jetter
R - Redder
W - Weaker
Y - Yellower

Table 9. Effect of Treatments on Shade Characteristics of Acetate Using Scoured Sample as Control

Sample number	Dye: Celanthrene Brilliant Blue FFS	Dye: Acetamine Diazo Black BGD
203	5-4, Bl, D	4 J
209	5-4, Bl, D	4 J
210	4 R, D	4 J
211	4 R, 5-4, D	4 J
212	4 R, 5-4, D	4 J
213	5-4 D	4 J
214	5-4 D, R	4 J
215	5-4 R	4 J
216	5-4 R	4 J
217	5	5
218	4 W	5-4 W
219	5-4 D	4 J

Classification.— 5 Very good - No trace difference
5-4 Very good - Trace shade difference
4 Good - Noticeable shade difference
3 Fairly good - Appreciable shade difference
2 Moderate - Considerable shade difference
1 Poor - Very much shade difference

R - Brighter
Bl- Bluer
D - Duller
G - Greener
J - Jetter
R - Redder
W - Weaker
Y - Yellower

Table 10. Light-Fastness of Acetamine Yellow N

Sample number	Hours Exposed			
	12	24	36	48
101	8	7	6	5
103	7	6	5	4
109	8	7	6	4
110	7	6	5	3
111	8	6	5	3
112	7	5	4	3
113	8	7	6	5
114	8	7	6	5
115	8	7	6	5
116	7	6	5	3
117	7	6	5	4
118	7	5	4	3
119	7	6	5	4
120	8	6	5	4

Classification.--8 Maximum
7 Excellent
6 Very good
5 Good
4 Fairly good
3 Fair
2 Poor
1 Very poor

Table 11. Light-Fastness of DuPont Anthraquinone Violet 3R

Sample number	Hours Exposed			
	12	24	36	48
101	7	6	5	5
103	7	6	5	5
109	7	5	4	4
110	7	6	5	5
111	6	6	5	5
112	7	5	5	5
113	-	-	-	-
114	7	5	5	5
115	7	6	5	5
116	7	6	5	5
117	7	6	6	5
118	6	5	4	4
119	6	5	4	4
120	6	5	4	4

Classification.--8 Maximum
7 Excellent
6 Very good
5 Good
4 Fairly good
3 Fair
2 Poor
1 Very poor

Table 12. Light-Fastness of Pontamine Fast Red

Sample number	Hours Exposed			
	12	24	36	48
101	8	8	7	7
103	5	4	3	3
109	7	6	4	4
110	7	6	5	5
111	7	6	4	2
112	4	3	2	1
113	-	-	-	-
114	8	8	7	6
115	8	7	7	6
116	5	4	3	2
117	6	5	4	3
118	6	4	3	2
119	6	4	2	1
120	6	6	4	3

Classification.--8 Maximum
7 Excellent
6 Very good
5 Good
4 Fairly good
3 Fair
2 Poor
1 Very poor

Table 13. Light-Fastness of Celanthrene Brilliant Blue FFB

Sample number	Hours Exposed			
	12	24	36	48
201	7	7	7	7
203	7	6	6	6
209	6	6	6	6
210	6	6	5	5
211	8	7	6	6
212	7	7	6	6
213	7	7	6	6
214	7	6	6	5
215	7	7	6	6
216	7	7	6	6
217	7	7	6	6
218	8	7	6	5
219	7	6	5	4

Classification.--8 Maximum
7 Excellent
6 Very good
5 Good
4 Fairly good
3 Fair
2 Poor
1 Very poor

Table 14. Light-Fastness of Acetamine Diamo Black BGD

Sample number	Hours Exposed			
	12	24	36	48
201	6	4	2	1
203	7	5	3	2
209	7	5	3	2
210	7	6	3	1
211	7	5	3	1
212	7	5	3	2
213	7	5	3	1
214	6	5	3	1
215	7	5	3	1
216	7	5	3	1
217	6	5	3	2
218	6	4	3	1
219	7	5	3	2

Classification.—8 Maximum
7 Excellent
6 Very good
5 Good
4 Fairly good
3 Fair
2 Poor
1 Very poor

Table 15. Wash-Fastness of Dyes on Nylon

	Dyes					
	Acetamine* Yellow N		DuPont** Anthraquinone Violet 3R		Pontamine* Fast Red	
Sample number	Color fastness	Staining effect	Color fastness	Staining effect	Color fastness	Staining effect
101	2	1	4	5	3	3
103	2	1	3	5	2	4
109	2	2	2	5	2	3
110	2	1	2	5	3	4
111	2	2	2	5	3	3
112	3	2	2	5	2	3
113	2	1	-	-	-	-
114	2	1	4	5	3	3
115	2	1	4	5	3	2
116	2	1	3	5	3	4
117	2	1	2	5	3	3
118	2	2	2	5	3	4
119	3	1	3	5	3	4
120	3	1	4	5	3	4

Classification.—

Color Fastness	Staining Effect
5 Excellent	5 Virtually unstained
4 Very good	4 Slightly stained
3 Good	3 Considerably stained
2 Fair	2 Heavily stained
1 Poor	1 Dyed

Note: Method, AATCC Standard Test Method 36-45, Test No. 3

* One washing
** Two washings

Table 16. Wash-Fastness of Dyes on Acetate

	Dyes			
	Celanthrene* Brilliant Blue FFS		Acetamine** Diazo Black BGD	
Sample number	Color fastness	Staining effect	Color fastness	Staining effect
201	2	1	2	3
203	2	1	2	3
209	2	1	2	4
210	2	1	3	4
211	2	1	2	4
212	2	1	3	4
213	2	1	3	3
214	2	1	2	3
215	2	1	3	3
216	2	1	2	4
217	2	1	2	3
218	2	1	2	4
219	2	1	2	3

Classification.—

Color Fastness	Staining Effect
5 Excellent	5 Virtually unstained
4 Very good	4 Slightly stained
3 Good	3 Considerably stained
2 Fair	2 Heavily stained
1 Poor	1 Dyed

Note: Method, AATCC Standard Test Method 36-45, Test No. 2.

* One washing
** Two washings

Table 17. Crock-Fastness of Dyes to Nylon Fabrics

Sample number	Dyes		
	Acetamine Yellow N	DuPont Anthraquinone Violet 3R	Pontamine Fast Red
101	4	4	4
103	3	4	3
109	3	4	1
110	2	4	2
111	4	4	3
112	4	5	3
113	4	-	-
114	4	4	5
115	4	4	5
116	2	4	3
117	2	5	2
118	2	4	1
119	2	4	2
120	2	4	3

Classification.— 5 Excellent
4 Very good
3 Good
2 Fair
1 Poor

Table 18. Crock-Fastness of Dyes to Acetate Fabrics

Sample number	Dyes	
	Celanthrene Brilliant Blue FFS	Acetamine Diamo Black BGD
201	5	5
203	4	4
209	3	4
210	3	5
211	1	4
212	3	3
213	4	4
214	5	4
215	4	4
216	5	4
217	5	5
218	3	4
219	4	3

Classification.— 5 Excellent
4 Very good
3 Good
2 Fair
1 Poor

CHAPTER V

CONCLUSIONS

From data obtained in this investigation, the following conclusions may be drawn:

1. The Silicone water-repellent was not resistant to 10 per cent sodium hydroxide when treated at 180°F. This reagent may be used in removing the Silicone from nylon and the dyed fabric will show only trace changes in shade characteristics, no change in fastness properties, and no change in tensile strength on treatments of 15 to 30 minutes.

2. The Silicone water-repellent on nylon was not resistant to repeated washings with a soap solution of 0.5 per cent neutral soap and 0.2 per cent sodium carbonate when the washings were conducted at 160°F and accompanied with severe mechanical action. When applied to acetate the Silicone water-repellent was partially removed by washing with the above soap solution and conditions.

3. The Silicone water-repellent was resistant to mineral acid treatments.

4. The Silicone water-repellent was partially removed by swelling agents such as phenol, formic acid, and acetic acid.

5. In all cases, when the same reagents were used on both nylon and acetate, more of the Silicone water-repellent was removed from the nylon than from the acetate, which may indicate that the Silicone was more strongly bonded to the acetate than to the nylon. (26)

6. Exclusive of washing treatments, the two per cent phenol at 112°F removed the highest percentage of the Silicone; and this sample showed no changes in dyeing characteristics, which was an indication that the Silicone, if completely removed, would have no effect on the dyeing properties of the acetate.

7. A Silicone treated fabric will show appreciable shade differences when dyed. A sample with the Silicone partially removed will show noticeable to trace changes in shade when dyed. A sample with the Silicone completely removed will show trace to no change in shade when dyed. The degree change will depend on the percentage Silicone present and the type dye used.

CHAPTER VI

RECOMMENDATIONS

This investigation has shown the possibilities of using the Silicone water-repellent, DeCotex 104 Emulsion, as a sizing agent for nylon since it can be easily removed by sodium hydroxide, with only trace to no change in dyeing characteristics resulting. It is recommended that work be undertaken evaluating the suggested use of Silicone as a sizing agent or as an addition to a size agent.

The investigation has indicated that if the Silicone is completely removed from acetate the dyeing characteristics of the fabric will be unaltered. It is recommended that this investigation be continued on acetate fabrics, and it is suggested that various type soaps and detergents be studied and evaluated. This study should include the effect of mechanical action used in conjunction with soaps and detergents.

APPENDIX

Table 19. Determination of Moisture Pickup of Fabrics

Acetate		
Wet weight in grams	28.7763	29.0404
Conditioned weight in grams	19.6094	19.9146
Moisture pickup in grams	9.1669	9.1258
Moisture pickup (%)	46.6	45.7
Average moisture pickup (%)	46.2	
Nylon		
Wet weight in grams	12.7733	13.8531
Conditioned weight in grams	9.9577	10.8755
Moisture pickup in grams	2.8156	2.9776
Moisture pickup (%)	28.2	27.4
Average moisture pickup (%)	27.8	

Table 20. Calibration of Spectrophotometer Using Standard Sample

Silicon (mg./150 ml.)	Transmission (%)
0.02	-
0.02	93.2
0.04	84.2
0.04	85.5
0.06	79.0
0.06	79.0
0.08	73.5
0.08	73.2
0.10	68.0
0.10	70.4
0.12	60.0
0.12	64.2

Instrument	Beckmann Spectrophotometer, Model B
Wave length	715 millimicrons
Phototube	Blue-sensitive
Sensitivity setting	4 (maximum)

Table 21. Analysis of Nylon Fabrics for Silicon

Sample number	Weight sample fused (grams)	Transmission (%)	Mg. of Silicon* in 10 ml. of aliquot
101	0.2407	98.5	0.0040
102	0.2325	83.0	0.0475
103	0.2339	83.5	0.0460
104	0.2341	91.0	0.0240
105	0.2283	106.0	0
106	0.2351	108.0	0
107	0.2311	102.0	0
108	0.2318	110.0	0
109	0.2515	70.0	0.0935
110	0.2405	81.2	0.0530
111	0.2303	81.6	0.0518
112	0.2390	91.4	0.0230
113	0.2332	109.0	0
114	0.2163	102.0	0
115	0.2369	106.0	0
116	0.2289	85.5	0.0400
117	0.2320	95.2	0.0125
118	0.2394	93.0	0.0180
119	0.2359	90.8	0.0245
120	0.2383	94.5	0.0145

Instrument	Beckmann Spectrophotometer, Model B
Wave length	715 millimicrons
Phototube	Blue-sensitive
Sensitivity setting	4 (maximum)

* Read directly from Fig. 2.

Table 22. Analysis of Acetate Fabrics for Silicon

Sample number	Weight sample fused (grams)	Transmission (%)	Mg. of Silicon* in 10 ml. of aliquot
201	0.2412	101.0	0
202	0.2330	84.0	0.0445
203	0.2327	83.2	0.0470
204	0.2300	89.5	0.0262
205	0.2353	91.5	0.0228
206	0.2381	93.5	0.0164
207	0.2338	97.0	0.0080
208	0.2338	95.0	0.0130
209	0.2507	86.0	0.0365
210	0.2260	86.5	0.0370
211	0.2504	84.2	0.0438
212	0.2278	78.5	0.0631
213	0.2303	86.8	0.0362
214	0.2314	85.6	0.0400
215	0.2318	84.0	0.0445
216	0.2359	82.0	0.0505
217	0.2365	88.0	0.0325
218	0.2327	87.5	0.0340
219	0.1667	89.0	0.0298

Instrument	Beckmann Spectrophotometer, Model B
Wave length	715 millimicrons
Phototube	Blue-sensitive
Sensitivity setting	4 (maximum)

* Read directly from Fig. 2.

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