

THE STUDY OF CREASE-RESISTANT FINISHING
TREATMENTS ON RAMIE FABRICS

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Siang Lin Ho
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THE STUDY OF CREASE-RESISTANT FINISHING

TREATMENTS ON RAMIE FABRICS

INTRODUCTION

Crease-resistance may be considered as the property of a fabric which causes it to recover from folding deformations that normally occur during its use. The crease-resistant finish is chiefly applied to rayon, cotton and linen fabrics, and occasionally to certain types of woolen and worsted fabrics. Cellulosic fibers have been notoriously known for their susceptibility to creasing, and the remedy for this defect may perhaps be considered as one of the greatest achievements in modern textile industry. The crease-resistant finishes now commonly employed are either urea-formaldehyde or melamine-formaldehyde resins. Other formaldehyde condensates, like thiourea-formaldehyde, phenol-formaldehyde, guanidine resins, etc., have been tried, but so far none has proved as satisfactory as urea- and melamine-formaldehyde resins.

Ramie, China grass or Rhea is obtained from *Boehmeria nivea* (or *Urtica nivea*) and *Boehmeria tenacissima*.¹ It has

¹Ott, E., Cellulose and Cellulose Derivatives, (New York: Interscience Publishers, 1946), pp. 17.

been known for many centuries and has many excellent properties that can qualify it for use in the textile field. However, until the last few years it had not attained much commercial importance because of the difficulties involved in the growing of the plant, the separation, and the purification of the fibers. The recent developments, which overcome or are well on the way to solve all these difficulties, give ramie a bright new future as a textile fiber. Many discussions and predictions found in the literature of the last decade apparently reveal its popularity among the scientists as well as the textile manufacturers. A brief review of the properties of ramie fibers may adequately indicate the reason for the revival of interest.

Ramie possesses many distinguishing advantages over other textile fibers, especially cotton. It has a high tensile strength, higher than cotton, silk, and even hemp.² It is more absorbent than cotton. It also has an increase in strength when wet. It dries rapidly. It has the additional advantage of being non-shrinkable and of being highly resistant to rot and mildew. When cotton and ramie materials are compared for coolness effect, the ramie material is known

²Sherman, J. V., and Sherman, S. L., The New Fibers, (New York: D. Van Nostrand Co., 1946), pp. 306.

to be more porous and cooler of the two.³ Its disadvantageous properties are its low torsional strength and inelasticity, which combination means brittleness and limits its use greatly. The fact that the fibrils of which the ramie fiber is composed are very highly orientated and parallel to the fiber axis and that it shows the most perfect crystallinity of all natural cellulose may account for its brittleness and also its susceptibility to creasing.

The object of this study is to investigate the possibility of imparting a crease-resistance to ramie fabrics by applying the urea-formaldehyde resins. There are many factors, such as the construction and size of yarn, thickness and construction of fabric, etc., which affect the property of crease-recovery of fabric. Furthermore, the results of the resin applications are also dependent upon many other factors, such as the relative amount of urea and formaldehyde, amount of catalyst, concentrations, padder pressures, padder speeds, tension on fabric during treatment, curing time and temperature, etc. However, in this study, the scope of investigation was limited to the following:

1. Determination of the amount of catalyst necessary while keeping other factors constant;

³Report No. 71829, U. S. Testing Co., Inc., (From Coss, H. T., and Taylor, J. L., "Ramie Today," Textile Industries, No. 14, Vol. 10, 1948).

2. Determination of the optimum molecular ratio of urea and formaldehyde;

3. Evaluation and analysis of the properties of the ramie fabrics before and after treatment.

THEORETICAL CONSIDERATIONS

Cellulosic fibers are now generally accepted as composed of long chain-like molecules in which some are packed closely together with their long axes parallel and have the properties of crystals, while the others are more or less randomly disposed. The groups of closely packed and aligned molecules are termed "crystallites," whereas regions of poor molecular alignment are termed "amorphous." Hence the fibers consist of alternating regions of crystalline and amorphous structure with some of the molecular chains running through both regions to give an irregular flexible network. The orientation of the crystallites with respect to the long axis of the fiber has an important influence on fiber properties. Cellulosic fibers which show high degree of crystallite alignment with the fiber axis will have high strength, low extensibility, and limited flexibility, and are very susceptible to creasing. This is why ramie having a high degree of orientation is more easily wrinkled than cotton, which has a lower degree of orientation.

The creasing or crushing of textile materials is a complex effect involving tensile, flexing, compressive and torsional stresses. Whenever a fabric is folded so sharply as to form a crease, the fibers at the crease are placed under strain. Those fibers on the outside of the fold will be under tension; while those at the inside will be under

compression. Furthermore, the fibers themselves will be bent, and the molecule chains on the outside of the fiber will be under tension and those on the inside may be under compression. In addition, there is some torsional strain in the fibers. However, it is probable that the tensile strain is the most important factor in creasing.

The extension of the fiber caused by stress results in a movement of the molecules within the fiber. With the disorientated fibers, some orientation will result, but they will recover their original positions when the strain is removed. With the orientated fibers, there may be a realignment of the molecules to a position of better orientation with the fiber axis or by a slipping of the molecular chains over each other. When the strain is released, there is no tendency or force to hold them to the original positions, hence the fiber is creased. Nevertheless, if the strain is not great enough to overcome the intermolecular forces of the cellulosic fibers, such as hydrogen bonding, the fibers may recover to their original forms.

The purpose of crease-resistant finishing is to produce a non-crush effect on the fabric without changing its properties as dress goods. It is extremely important that the product used for imparting this effect should be situated within the fiber of the textile material, for if it merely adheres to the surfaces of the fibers without penetration, the result will

be the loss of suppleness of the fabric without imparting crease-resistance.

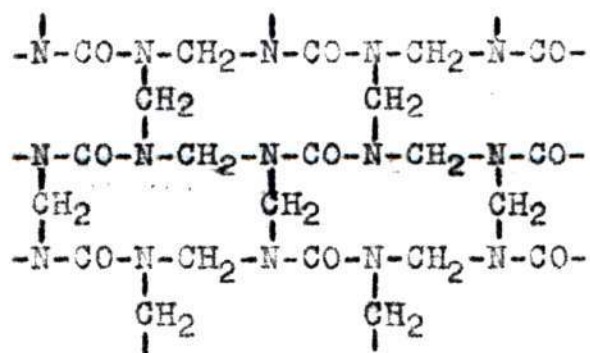
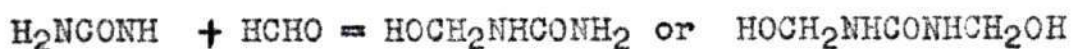
Many compounds and synthetic resins may be used for the crease-resistant finishing. In this study, the work was confined to the use of urea-formaldehyde resins. Under neutral or slightly alkaline conditions, urea and formaldehyde condense to give either mono- or dimethylol urea, according to the molecular proportions of the reagents. Both of them are crystalline substances, readily soluble in water and of low molecular weight. It is the aqueous solution of the compounds which impregnates the fabric and which penetrates into the fibers. The success of the process is largely dependent on the distribution of the pre-condensate of urea-formaldehyde resin throughout the cellulose. Hence, the fabric should have the greatest possible absorbency.

Foulds and Marsh⁴ suggested that the cellulose be dispersed or swollen beforehand by suitable swelling agents, such as caustic soda solution; and if necessary, the fabric may be treated in the wet swollen state after the removal of the swelling agent so that the pre-condensate will be accessible throughout the fiber. However, Nickerson⁵ suggested that the results obtained by using mercerized and unmercerized fabrics

⁴Tootal Broadhurst Lee Co., Foulds, R. P. and Marsh, J. T., "Treating With Liquids," B.P. 304,900, Jan. 9, 1928.

⁵Nickerson, R. F., "Investigations of Anticrease Treatments for Cotton," American Dyestuff Reporter, No. 2, Vol. 39, pp. 46-50, 62. 1950.

do not differ very much. Mechanical treatment, such as repeated impregnation with squeezing rollers is beneficial in assisting the penetration of the fiber by the reaction mixture. The pre-condensates, when heated under acid conditions, form the tri-dimensional network of urea-formaldehyde resin.



X-ray photographs have indicated that the resin is not situated within the micelle and must therefore lie between the "crystallites."⁶

Many postulations and theories have been advanced to account for the property of recovery from creasing shown by cellulosic materials after resin treatment. As the crease-resistance is affected by so many factors, a generally satisfactory theory has never been reached. Among the explanations

⁶Marsh, J. T., An Introduction to Textile Finishing, (London: Chapman and Hall Ltd., 1948), pp. 411.

suggested, some of them are subject to critical examination. For instance, the permanent-wave theory⁷ states that crease-recovery is associated with the permanence of the wavy form of the fibers, and that the resin fixes the fibers in the undulating position. In nearly all fabrics, the filling is much straighter than the warp, which is caused to take up an undulating path, yet measurements of crease-recovery in the treated fabrics very frequently show that the filling has a better crease-recovery. This theory also fails to explain the good crease-recovery possessed by the pile of treated velvets. However, waviness may be a contributory factor.

Another theory⁸ suggests that the improved crease-resistance is associated with the reduced swelling in water. It is true that cellulosic material which has been treated with crease-resistant finishes usually shows a reduced tendency to swell in water, but the two properties do not necessarily go together. Wool swells considerably in water and yet is regarded as the standard of crease-recovery. Linen, which has had crease-resistant finishing treatment, has a higher water-imbibition value and shows a greater swelling in water than the untreated product.

⁷Marsh, J. T., An Introduction to Textile Finishing, 1948, pp. 427.

⁸ibid., pp. 428.

One of the theories suggested that textile fibers are porous and contain a number of small cavities between the crystallites of which the fibers are composed; these cavities are assumed to act as air-cushions or air-pockets which are responsible in part for the elastic recovery of the compressed fibers. When the sub-microscopic cavities are very small, they are much less susceptible to crushing, and so cause an elastic pneumatic reaction to the folding of the fiber, which behaves like a porous rubber tube of very small dimensions. This theory depends solely on some data of Frey-Wyssling,⁹ from which it appears that the cavities in wool and silk are about one-half the size of those in cellulose, but reference to the original work shows that it depends on the impregnation of fibers with various metallic salts from aqueous solution, followed by deposition of the metal and the measurement of the metallic particles. Hence the data refer to the wet and swollen state and not to the dry state. The fact that the pore sizes of viscose rayon and silk are the same also shows there is no correlation between cavity size and crease-recovery.

Quehl¹⁰ suggested that creasing properties of fibers

⁹Marsh, J. T., An Introduction to Textile Finishing, 1948, pp. 428.

¹⁰ibid., pp. 428.

are determined by the amount of crystalline material which they contain, and that by increasing the proportion of amorphous material it is possible to improve the elastic recovery. The deposition of the insoluble condensation products of urea and formaldehyde, which is in amorphous form,¹¹ within the fiber naturally increases the proportion of amorphous matter and therefore increases the crease-resistance.

The hypothesis of the formation of cross-linkages between cellulose molecules give a better explanation for the crease-resistance phenomenon. It has been mentioned before that the cellulose fibers consist of alternating regions of crystalline and amorphous regions with some of the molecular chains running through regions to give an irregular flexible network.

Extension of the fiber inevitably involves a complex system of stresses and strains among the entangled mass of molecular chains in the amorphous region and this region probably is most responsible for the elasticity of the fiber.

It has also been mentioned that highly orientated filaments exhibit a relatively small total extension compared with that of disorientated filaments; the extension of the former is mainly due to micellar slippage, and that

¹¹ Schwarz, E. W. K., and Wengraf, P., "Modern Trends in Finishing," American Dyestuff Reporter, No. 22, Vol. 30, pp. 610-614, 618-619. 1941.

drain and rinse program would be called. If the system had not reached equilibrium, then the dyeing program would recycle. At the completion of the drain and rinse program, program control was returned to the main line program and the elapsed time was printed signifying the end of the dyeing process.

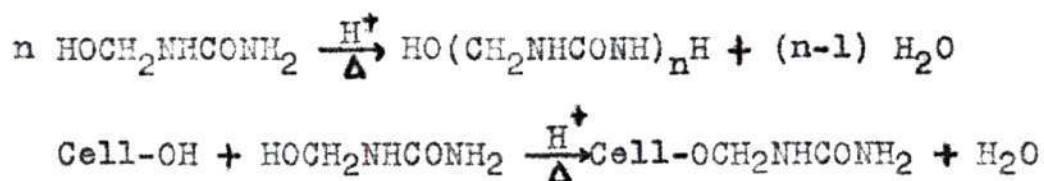
Dyeing Procedure

The dyeings were made to simulate industrial practice as much as possible. A shade was selected for which the dye formula was known. The carpet sample to be dyed was weighed and the amount of dyestuff needed to match the given formula was weighed. The dyestuff was then put into an aqueous solution so that it could be pumped to the dilution tank above the circulation pump. The dyestuffs used were of the type called acid levelling. These dyes are characterized as possessing high affinity for nylon, good fastness properties, bright colors, and, as a group, are sensitive to heat differences. If these dyes were applied from a bath that had uneven temperatures across the face of the material being dyed, then a greater amount of dye would be found on the material in the hotter part of the dye bath.

Phosphoric acid was used for pH control. From the beaker dyeings it was learned that 0.2 grams per liter of water resulted in a solution of pH = 3.0. This concentration was extended to the proper amount of acid to use in the larger operation. The beaker dyeings also showed that precise control of pH was not necessary. Dyeings made at pH = 2.5, pH = 3.0, and pH = 4.0 were found not to differ in color or bath exhaustion.

The carpet was sewn into a continuous loop inside the dye beck,

only possibilities are resin formation or simple cellulose products:



It is very possible both resin formation and cross-linking reactions may occur when the fabric is treated with the pre-condensate. As a whole, it seems quite logical to assume the formation of crosslinkages between the micelles by the urea-formaldehyde resin. Such linkages are not necessarily of the primary valence type, but also includes the hydrogen bondings between the cellulose hydroxyl groups and the bonds in the resin polymer.

APPARATUS

The apparatus used in these investigations are as follows:

Experimental three-roll Butterworth Padding Machine.

Central Scientific Company forced circulation, triple-walled, electrically heated and controlled oven (Model No. 95400).

Smith-Drum Monel Metal Rotary Dyeing Machine (Type 12-RD).

Monsanto Wrinkle-Recovery Tester.

Scott J-2 Combination Fabric Testing Machine.

Taber Abraser, Model E-4010.

Atlas Launder-Ometer, Type LHD-EF.

Atlas Fade-Ometer, Type FDA-R.

Ostwald Viscosimeter.

Beckman pH meter (Industrial Model M).

MATERIALS

Ramie Fabric -- Plain weave.

Warp: 16's.

Filling: 16's.

Construction: 50 ends
48 picks.

Width: 41 inches.

Formaldehyde (40 per cent by volume).

Urea (C. P.).

Ammonium Dihydrogen Phosphate (c.p.).

Other common finishing reagents.

GENERAL EXPERIMENTAL PROCEDURE

The method for the preparation of the pre-condensate of urea-formaldehyde, its impregnation on the fabric, drying curing and washing was kept the same throughout the investigations.

The ramie fabric to be tested in this study was prepared by the usual desizing, scouring and bleaching treatments.

The experimental phases of these investigations consisted of three main parts:

1. The determination of the optimum amount of catalyst, $\text{NH}_4\text{H}_2\text{PO}_4$, needed using a given molecular ratio of urea to formaldehyde (1.0 to 1.6).
2. The determination of the optimum molecular ratio of urea and formaldehyde using the previously determined amount of catalyst.
3. The evaluation and analysis of the properties of the fabrics before and after the crease-resistant treatment.

EXPERIMENTAL

1. General Method of Application:

The general method for the preparation of the pre-condensate of urea-formaldehyde,¹³ its impregnation on the fabric, drying, curing and washing was as follows:

The pH of the formaldehyde was adjusted to 7 with 0.1 N sodium hydroxide solution. The urea was added to the neutralized formaldehyde and dissolved in it. The solution was then adjusted to pH 8.9 ± 0.1 by addition of 0.1 N sodium hydroxide solution and was heated in a flask to 175°F in 20 minutes. It was kept at 175°F for five minutes, followed by rapid cooling to 70°F. The pre-condensate thus prepared was diluted to twice its volume by addition of water, the catalyst was added to urea-formaldehyde solution prior to impregnation of the cloth. The ramie fabric was impregnated with the solution on the padding machine by two dips and two nips. The rollers had a pressure of 60 pounds per square inch and a speed of 33 revolutions per minute (r.p.m.), thus giving a wet pick-up ranging from 70 to 85 per cent. The impregnated fabric was dried at room temperature, cured in electric oven at $300 \pm 2^\circ\text{F}$ for five minutes and followed

¹³ Creegan, H. F., "Control of Shrinkage and Crease-Resistance of Viscose Rayon Fabric," American Dyestuff Reporter, No. 22, Vol. 35, pp. 514-518, 1946.

by washing at 140°F for 45 minutes in a bath containing 2 per cent soap, 1 per cent soda ash and 1 per cent Santomerse, after which it was again rinsed with water and dried.

The wet pick-up was determined by the difference between the wet impregnated fabric and the conditioned untreated fabric. The dry pickup was determined by the difference between the dry conditioned treated and untreated fabrics.

2. Preparation of Fabric:

The ramie fabric to be tested was desized by a 3 per cent solution of Exsize for one hour at $120-130^{\circ}\text{F}$ with a pH of 6.0-6.5. The desized fabric was scoured by boiling in a bath containing 3 per cent sodium hydroxide for one hour. After which it was bleached at 185°F for one and one-half hours with 0.1 per cent solution of Textone containing:

0.1 per cent Santomerse,

0.6 per cent of 28 per cent Acetic Acid, and

0.125 per cent of Trisodium Phosphate.

3. Determination of Optimum Amount of Catalyst:

A pre-condensate of urea-formaldehyde was prepared by the method described above using a molecular ratio of urea to formaldehyde at 1.0 to 1.6. This solution was divided into five portions. Into each portion, five different amounts of $\text{NH}_4\text{H}_2\text{PO}_4$, ranging from 0.06 to 0.30 per cent (with an increment of 0.06 per cent) of the weight of the pre-condensate, were added respectively. Finally, each

portion was diluted to twice its volume by water and was applied to the fabric by the method described previously. Thus, five sets of samples of treated fabrics were prepared. Results of crease-recovery and breaking strength tests are shown in Table I, page 43, and Figure II, page 30.

4. Determination of Optimum Molecular Ratio of Urea and Formaldehyde:

Six pre-condensates of urea-formaldehyde were prepared by using the following molecular ratios of urea to formaldehyde:

Urea	Formaldehyde
1.0	1.0
1.0	1.2
1.0	1.4
1.0	1.6
1.0	1.8
1.0	2.0

Each pre-condensate was diluted to twice its volume and 0.18 per cent of $\text{NH}_4\text{H}_2\text{PO}_4$ based on the weight of the pre-condensate was added. A sample fabric, 8 inches by 72 inches, was impregnated with each bath, dried, cured and washed. Crease-recovery and breaking strength tests were performed on each treated fabric. Results of these tests are shown in Table II (page 44) and Figure III (page 32).

5. Evaluation and Analysis of the Properties of Treated Fabrics:

The following tests were made on crease-resistant treated (Urea : Formaldehyde = 1.0 : 1.6; catalyst = 0.18 per cent) and untreated fabrics:

- A. Crease-Recovery
- B. Moisture Content
- C. Breaking Strength and Stretch (Dry and Wet)
- D. Resistance to Abrasion.
- E. Wash Fastness of Resin Finish
- F. Effect on Dyeing Properties
 - (1) Colorfastness to Light
 - (2) Colorfastness to Washing

All the physical tests were conducted under the standard conditions (65 per cent R. H. and 70°F).

A. Crease-Recovery

A Monsanto Wrinkle Recovery Tester¹⁴ was employed for the crease-recovery measurements. The fabric to be tested was conditioned for at least four hours. Test specimens 1.5 cm. wide and 4 cm long were cut from the fabric in both warp and filling directions. The longer dimension represented the direction of the test. Two sets of five specimens each

¹⁴Bulletin No. T-7, "Monsanto Wrinkle Recovery Tester," Monsanto Chemical Co., Boston, Mass., Dec. 1, 1947.

were tested for both directions. The test specimen was placed between the metal leaves of the specimen holder of the tester. The exposed end of the specimen was folded over the shorter metal strip. The metallic specimen holder with the folded specimen was inserted into the plastic press. A load of one and one-half pounds was applied to the press for five minutes. Then the press was unloaded, the holder was taken out and mounted on the tester. This tester, Figure 1, consists of a movable disc with a rigidly supported platform. The folder was placed on the platform and was locked in position. one side of fold of the specimen was supported while the other was free to recover. The free side of the fold was brought to a vertical position, in alignment with the vertical guide line on the back stationary panel, by periodical manual rotation of the disc. After five minutes, the angle of recovery was read directly from the degree scale on the back panel by the reading index. The angles of recovery of each set of specimens were recorded and averaged.

B. Moisture Content

Moisture content of each fabric was determined after the fabric had been conditioned in the testing laboratory. The procedure consisted in weighing conditioned specimens, drying in oven and weighing again. The loss of weight was

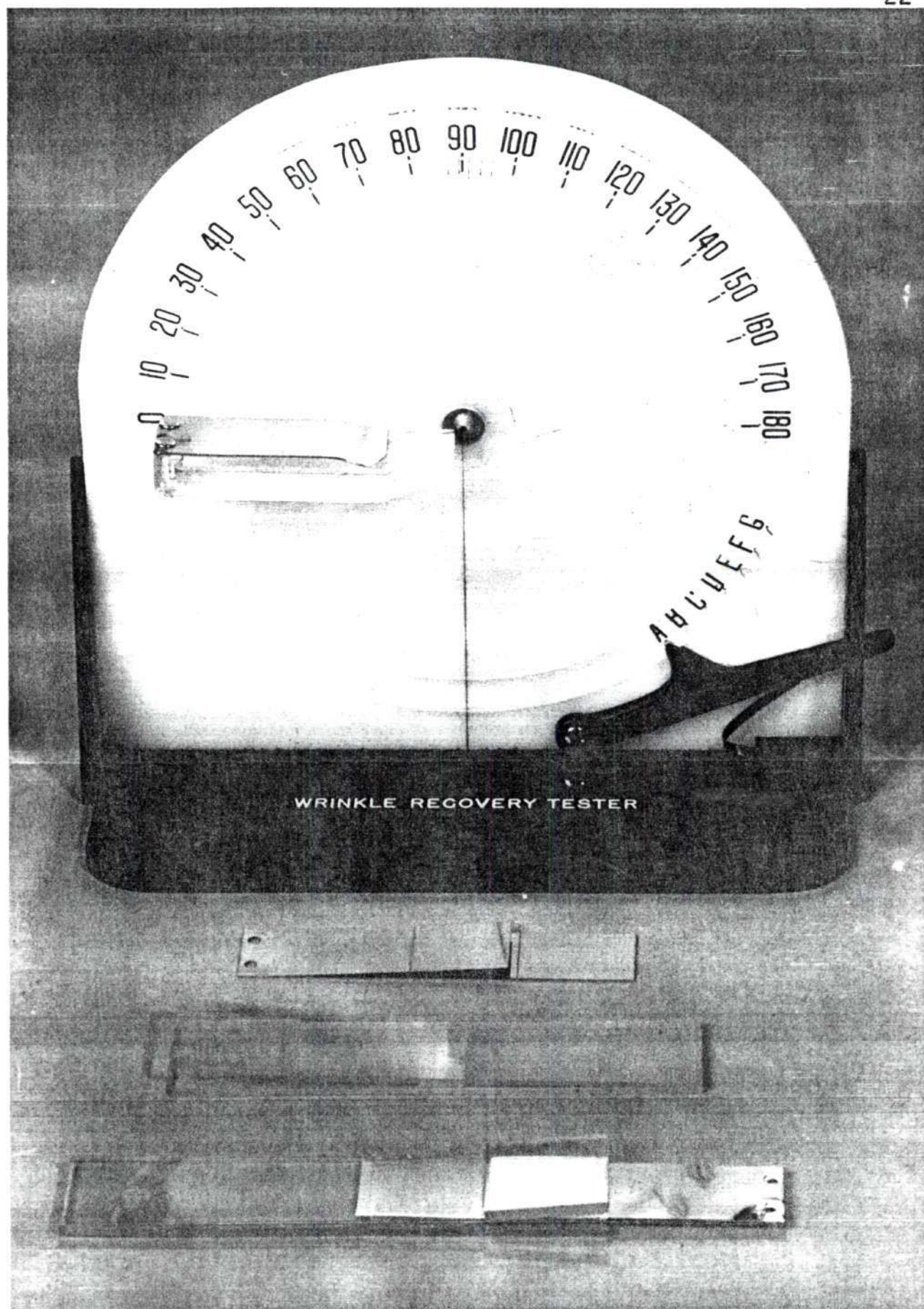


FIGURE I

MONSANTO WRINKLE RECOVERY TESTER

the moisture content. Three specimens each of treated and untreated fabrics were tested and their moisture contents averaged respectively, Table IV, page 46. The moisture content, expressed in percentage, was calculated by using the formula:

$$\text{Moisture Content, per cent} = \frac{C - D}{C} \times 100$$

where: C = weight of conditioned specimen, and
D = weight of bone dry specimen.

C. Breaking Strength & Stretch (Dry & Wet)

The breaking strength and stretch tests were performed according to A.S.T.M. cut strip method¹⁵ on a Henry L. Scott Company Combination Fabric Testing Machine. This machine was equipped with flat jaws for strip specimens and an autographic recorder to measure the stretch. Test specimens one inch wide and six inches long were cut from the fabric. For each fabric, two sets of five dry specimens each were tested for both warp and filling directions; and the same for two sets of five wet specimens. The longer dimension represented the direction of test. Both the upper and lower jaws are of two inches by one inch in size, and a three inches gauge was maintained between them. Five determinations

¹⁵A.S.T.M. Designation: D39-39, A.S.T.M. Standards on Textile Materials, (Philadelphia, A.S.T.M. Committee D-13, 1948), pp. 104.

of each set were tested and averaged. Results are shown in Table V, page 47.

D. Resistance to Abrasion

Comparative abrasion tests were made by using a Taber Abraser on the treated and untreated fabrics. The specimen, mounted on a horizontal rotating circular plate running at 68 r.p.m., was abraded by dual abrasive wheels, CS-10, which rub back and forth and criss-cross their path so that the abrasive action was at all angles to the weave. A constant pressure of 1000 grams was maintained on each of the abrasive wheels. An automatic counter which records the number of abrasion cycles is actuated directly by the rotating specimen holder. The number of cycles to form the first hole on the fabric was recorded for comparison. Three specimens of each of treated and untreated fabric were tested and their readings averaged respectively. Results are shown in Table VI, page 48.

E. Wash Fastness of Resin Finish

The fastness of resin finish to commercial laundering and domestic washing was determined by using an Atlas Launder-Ometer. The speed rotor of the machine is driven at a speed of 42 r.p.m. by a $\frac{1}{4}$ H.P. motor. Although colorfastness is not concerned in this test, eight specimens were tested according

to Test No. 3 of A.A.T.C.C. method for testing colorfastness of cotton and linen textiles,¹⁶ that is, each specimen was washed in a glass pint jar with ten $\frac{1}{4}$ inch stainless steel balls and a bath containing 0.5 per cent soap and 0.2 per cent sodium carbonate at 160°F for 45 minutes. Each of the eight specimen had a different number of washings, ranging from one to eight washings. After each washing, the specimens were rinsed twice with water at 105°F, then with a 0.05 per cent solution of acetic acid at 80°F and followed by another water-rinse at 80°F. All the specimens were tested for crease-recovery, see Table VII (page 49) for data on crease-recovery.

F. Effect on Dyeing Properties

Six pieces of ramie fabric were dyed with the following dyes:

Direct Dyes: Pontamine Fast Brown 4RL

Pontamine Fast Pink BL

Pontamine Fast Blue 2GL

Developed Dyes: Pontamine Diazo Blue 6G (with beta-Naphthol)

Vat Dyes: Sulfanthrene Blue GR Paste

Sulfur Dyes: Sulfogene Brown RBNCF

These dyed fabrics were divided into two sets. One set was treated with the crease-resistant finish (urea : formaldehyde = 1.0 : 1.6; $\text{NH}_4\text{H}_2\text{PO}_4$ = 0.18 per cent) and the other set left

(1) Colorfastness to Light: The colorfastness to light of the dyed fabrics was determined by the A.A.T.C.C. method.¹⁷ The apparatus used was the Atlas FDA-R Fade-Ometer. One specimen of 2½ inches by 3 inches was cut from each color (treated and untreated). The specimens were exposed simultaneously in the Fade-Ometer until one sample showed fading. The fading specimens were taken out, the time of exposure recorded and the remaining sample were further exposed to light. The amount of fading of each specimen was judged by the comparison of the exposed and the unexposed fabric. Results are shown in Table VIII, page 50.

(2) Colorfastness to Washing: The colorfastness to washing was determined by the A.A.T.C.C. method¹⁸ on a Launder-Ometer. The specimen of each color (treated and untreated) having a size of 2 inches by 4 inches, was attached with a white cotton cloth of 2 inches square. Specimens of the cotton standards for colorfastness to washing, furnished by the American Association of Textile Chemists and Colorists, were treated together so that the results of the test can be determined by comparing the change in color and staining

¹⁷"Colorfastness to Light" A.A.T.C.C. 1949 Year Book, pp. 105-106.

¹⁸"Colorfastness to Commercial Laundering and Domestic Washing," ibid., pp. 91-92.

of the test cloth produced by the same test on the corresponding standard dyeing. Results are shown in Table IX, page 51.

DISCUSSION OF EXPERIMENTAL PROCEDURE AND RESULTS

There are many factors which may affect the results of resin finish application. In this study precautions were taken to keep the controlling factors throughout the different processes as uniform as possible.

The measurement of crease-recovery angle and breaking strength were the principal tests used in this study to evaluate the results of various experiments.

The preparation of the urea-formaldehyde pre-condensate can be effected in many ways. For instance, for a laboratory scale, it can be made by boiling under a reflux condenser for three minutes and cooling rapidly. This method had been tried, but the condensation reaction was so rapid that it was found to be difficult to control for the desired viscosity, which is around 6 centipoises in this study. The pre-condensate can also be made by allowing the mixture to react at room temperature. This method is safer and more releable, but it takes too much time to get the desired viscosity. The method, which consists of heating the reagents to 175°F in 20 minutes and maintaining at that temperature for five minutes, had been found to be excellent, and, consequently, was adopted in this study.

The impregnated fabric was pinned on a wooden frame and dried at room temperature. High temperatures were

avoided in the initial stages of drying because of the possibility of bringing about migration of resin to the surface of the fabric to form surface resin which will give a dry and harsh feel to the fabric. Even after drying at room temperature, the treated fabric has a slightly harsh feel.

The resinification of urea-formaldehyde pre-condensate requires an acidic medium. The ideal catalyst is the one in which the acidity will not develop until heated. Hence, $\text{NH}_4\text{H}_2\text{PO}_4$ was used. Other ammonium salts of mineral acids and compounds, like polysulfones,¹⁹ borotartaric acid,²⁰ etc., can also be used. The amount of catalyst employed is critical for the best effects. If too much catalyst is used, there is a danger of the resin being hydrolyzed by the excess of acid and even of some hydrolytic attack on the cellulose itself. If too little catalyst is used, the condensate is not properly resinified and is soluble in mild alkali. From the results on Table I (page 43) and Figure II (page 30), it is evident that fabric treated with the pre-condensate containing 0.18 per cent of $\text{NH}_4\text{H}_2\text{PO}_4$ has the highest dry pick-up and crease-recovery angle, although the breaking strength is slightly lower than those of the fabrics using smaller

¹⁹Sylvania Industrial Corp., "Crease-Resistant Finishes -- Polysulfones as curing Agents," U.S.P. 2,410,395, October, 1946

²⁰Cameron, W. C., and Morton, T. H., "Permanent Finishes on Viscose Rayon Depending on Cross-Bonding," American Dyestuff Reporter, No. 16, Vol. 38, pp. 575-581, 1949.

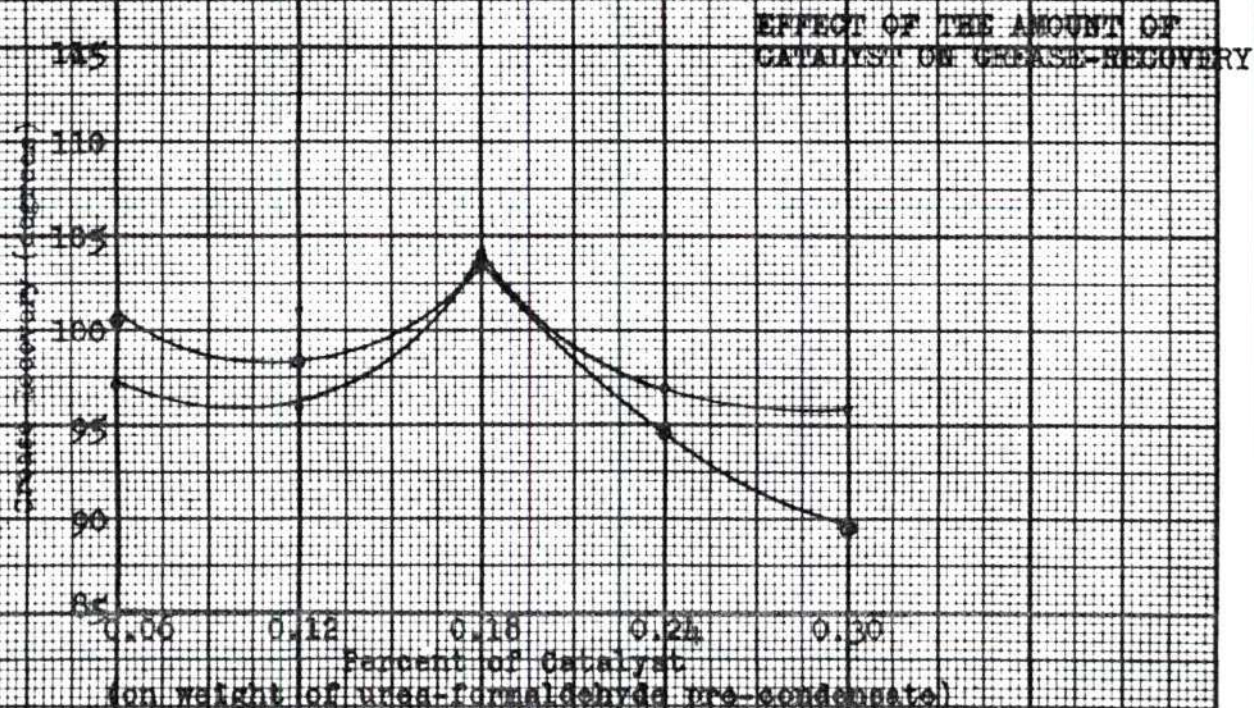
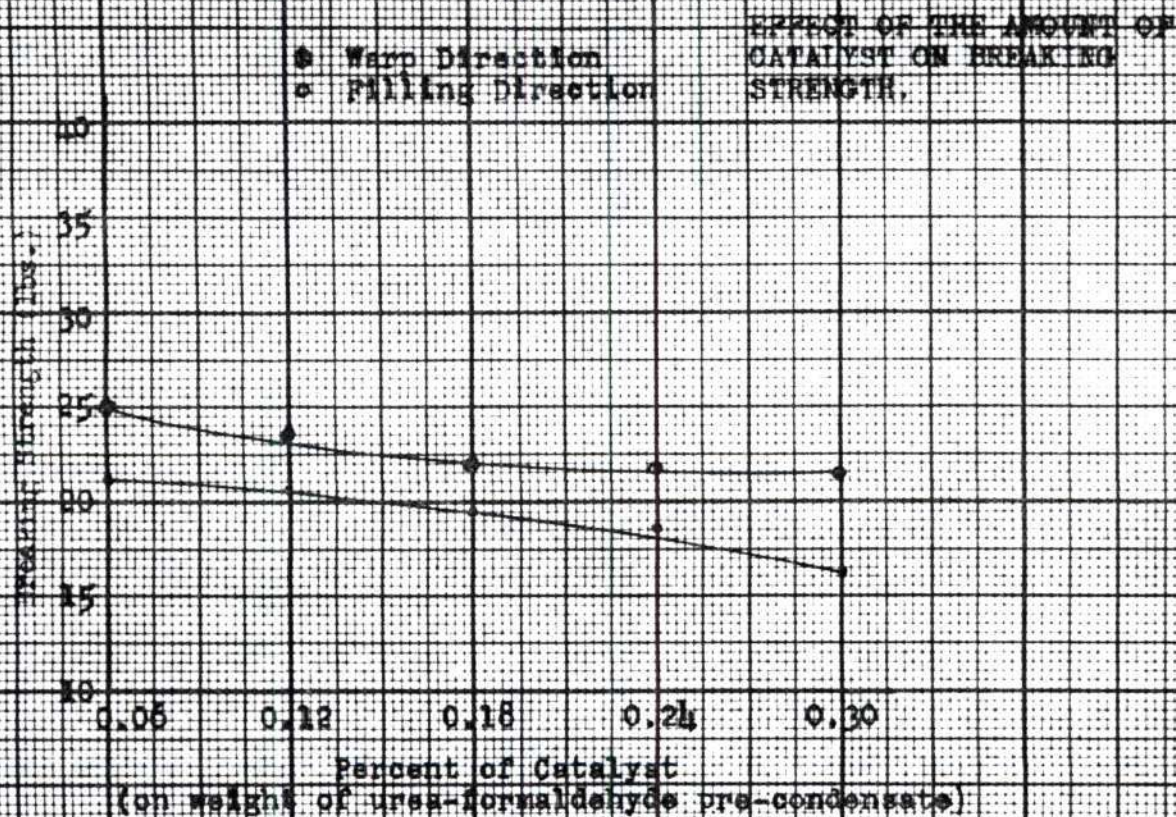
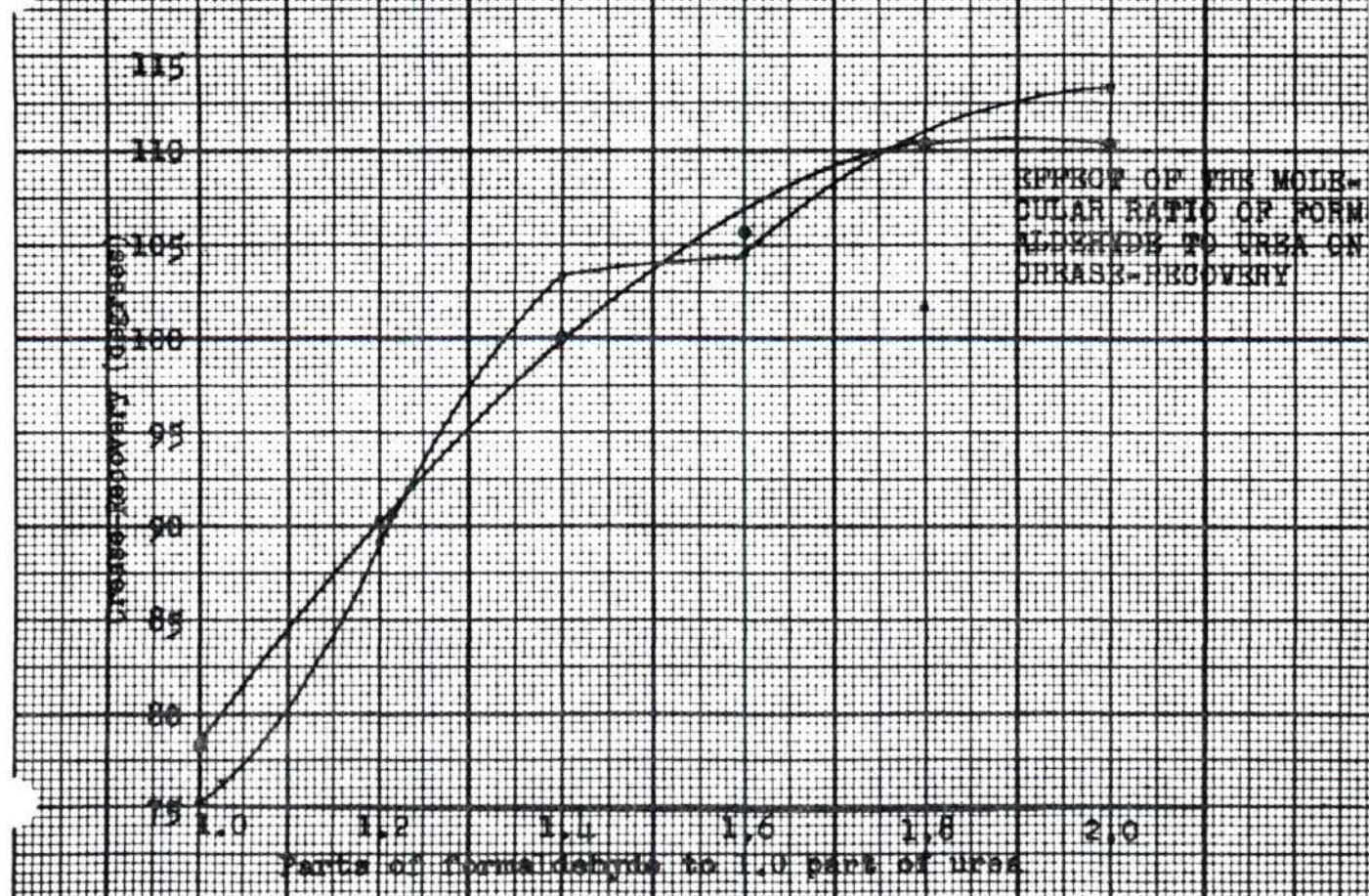
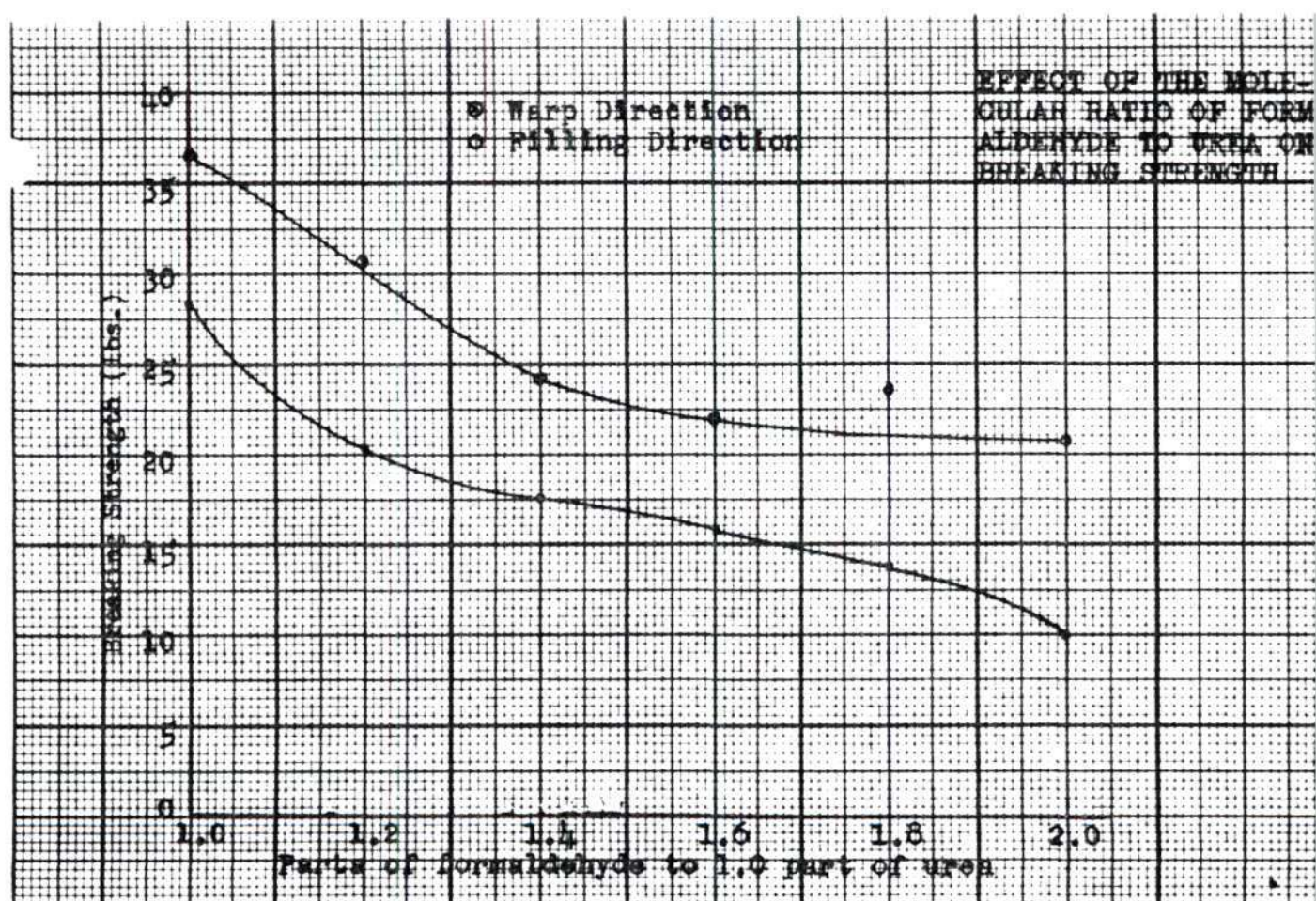


FIGURE II

amounts of catalyst. It is probably due to the fact that a smaller amount of the condensate was properly resinified by using insufficient quantity of catalyst and, consequently, less amount of resin was picked up in the cloth.

The molecular ratio of formaldehyde to urea tested ranged from 1.0:1.0 to 2.0:1.0. The higher ratios of formaldehyde to urea were more effective for the crease-resistance and shrinkage control, but also caused greater embrittlement of fibers which was reflected in the loss of breaking strength as shown in Table II (page 44) and Figure III (page 32). The molecular ratio of formaldehyde at 1.6:1.0 was chosen as the optimum ratio in this work. This ratio is not necessarily the best ratio for all purposes, but it gives fairly good crease-resistance without losing too much breaking strength.

It will be noticed that the improvement on crease-resistance is at the expense of the breaking strength (Tables II and III, pages 44 and 45). For the 95.92 per cent increase in crease-recovery angle in warp direction, the breaking strength was decreased by 53.19 per cent (Table V, page 47). For the 91.24 per cent increase in crease-recovery angle in the filling direction, the breaking strength was decreased by 65.87 per cent. The excessive loss in breaking strength is believed to be due to the inadequate tension applied during drying and curing. The pin frame and oven used in curing are probably not suitable for resinification.



Usually the resinification process will not impair the cellulose, because it is found that under proper control the properties of cellulose can be recovered unchanged by stripping off the resin.²¹ It is believed that breaking strength loss can be reduced to less than 20 per cent,²² if proper stretching, drying and curing chambers are employed. The greater decrease in breaking strength and smaller decrease in stretch in the filling direction are possibly associated with the method of impregnation where only the warp yarns are under tension.

The breaking strength of both treated and untreated fabrics were increased when wet (Table V, page 47). The increase in breaking strength of treated fabric was considerably greater than that of untreated fabric. The same is true with the increase in stretch.

The moisture content of fabric was decreased by 18.98 per cent after urea-formaldehyde resin treatment (Table IV, page 46). However, if the moisture content of the treated was calculated with reference to cellulose and not to the gross weight of cellulose and resin, the moisture content of the treated fabric is very close to the untreated (page 46).

The resistance to abrasion after treatment was reduced

²¹Nickerson, R.F., "Investigation of Anticrease Treatments For Cotton," American Dyestuff Reporter, No.2, Vol.39, pp.46-49, 51. 1950.

²²Buck, G.S., and McCord, T. H., "Crease-Resistance and Cotton," Textile Research Journal, No. 4, Vol.19, pp. 216-247. 19

by 35.44 per cent (Table VI, page 48). This is a natural result when the fibers become more brittle by the treatment.

The urea-formaldehyde resin is not fast to boiling alkaline liquors, but if the treated fabric is washed at a lower temperature, the crease-resistant effect will persist. From the experiment, it has been shown that the effect is only slightly reduced after eight severe washings (Table VII, page 49).

The fastness of dyes to light and washing is usually improved by the crease-resistant treatment, although exceptions are not rare. It has been found that the reduction in fiber swelling resulting from the treatment makes fabrics more difficult to dye, and for this reason the crease-resistant treatment is applied after the goods have been dyed.

In most cases, the dyeings will change in shade as a result of treatment. The changes in shade are of more importance when pale shades are concerned; the changes are usually more noticeable with brown colors; while with the heavy shades, the changes do not arise to any appreciable extent. Since the effects on the shade and fastness vary with the dye, it was not possible to test all dyes. However, in this study, six dyes of four classes, which are commonly employed on ramie fabrics, were selected for testing.

From the experiments (Table VIII, page 50), it has been found that the light fastness of Pontamine Fast Brown

4RL and Pontamine Fast Pink 5L was improved considerably, whereas that of Pontamine Fast Blue 2GL was reduced greatly. The colorfastness to washing of all three was improved, but to a different extent, Table IX (page 51). The shade of brown dye was changed into a reddish shade; that of the pink dye into a dull tone and that of the blue dye into a slightly paler shade.

With the azoic dyes, as illustrated by Pontamine Diazo Blue 6G in the experiment, the shade did not change to an appreciable extent. The colorfastness to light and washing was improved.

Vat dyes were not affected in shade by the crease-resistant treatment. The fastness to light and washing, already of high order, was not impaired and often improved as in the case of Sulfanthrene Blue GR Paste.

Sulfur dyes were seldom affected in shade by the crease-resistant treatment. The fastness to light and washing was not impaired. In this experiment, Sulfogene Brown BENCF showed no change in shade but improvement in fastness to light and washing.

CONCLUSIONS

From the results of this study, the following conclusions have been reached:

1. For a plain weave ramie fabric, the best crease-resistant treatment was obtained by using a molecular ratio 1.0 : 1.6 (urea : formaldehyde) to make the pre-condensate, and 0.18 per cent of $\text{NH}_4\text{H}_2\text{PO}_4$ of the weight of pre-condensate as catalyst.
2. The harsh feel of the treated fabric was reduced to a minimum by careful drying and washing in the crease-resistant finishing process.
3. The crease-resistance of ramie fabric resulting from the urea-formaldehyde resin treatment varied inversely with the breaking strength and stretch.
4. The breaking strength and stretch of ramie fabrics were increased when wet. The increase was greater in crease-resistant finishing treated fabrics.
5. The moisture contents of both treated and untreated fabrics were practically the same, provided the moisture content was based on the weight of the cellulose only.
6. The resistance to abrasion was decreased by 38.14 per cent due to the crease-resistant finishing treatment.
7. The crease-resistant effect on fabric by the urea-formaldehyde resin treatment was only slightly reduced

after eight severe washings, thus indicating that the treatment is not easily removed by ordinary laundering methods.

8. Most of the dyes used on ramie fabrics are altered in shade by the crease-resistant treatment, however, colorfastness to light and washing are generally improved.

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APPENDIX

Tables

TABLE I

BREAKING STRENGTH AND CREASE-RECOVERY OF RAMIE FABRICS
FOR THE SELECTION OF AMOUNT OF CATALYST

Catalyst (%)	WET Pickup (%)	DRY Pickup (%)	WARP		FILLING	
			Breaking Strength (lbs.)	Crease- Recovery (degrees)	Breaking Strength (lbs.)	Crease- Recovery (degrees)
0.06	77.2	14.6	25.0	100.6	21.4	97.2
0.12	79.6	15.7	23.6	98.4	20.8	96.0
0.18	78.7	17.6	22.0	103.6	19.6	104.0
0.24	78.7	17.4	21.9	94.6	18.7	97.0
0.30	79.1	17.0	21.4	89.6	16.2	96.0

Note: Urea : Formaldehyde = 1.0 : 1.6 (Molecular Ratio).

Viscosity of Pre-condensate = 5.69 centipoise.

Percentages of catalyst ($\text{NH}_4\text{H}_2\text{PO}_4$) expressed in the table were based on the weight of the pre-condensate.

Each breaking strength and crease-recovery test is the average of five tests.

TABLE II

BREAKING STRENGTH AND CREASE-RECOVERY OF RAMIE FABRICS FOR THE
SELECTION OF MOLECULAR RATIO OF UREA AND FORMALDEHYDE

Mol. Ratio U : F	Viscosity (centi- poise)	Wet Pickup (%)	Dry Pickup (%)	WARP		FILLING	
				Breaking Strength (lbs.)	Crease- Recovery (degrees)	Breaking Strength (lbs.)	Crease- Recovery (degrees)
1.0 : 1.0	6.41	75.5	18.7	36.8	78.2	28.4	75.2
1.0 : 1.2	5.96	75.9	19.0	30.7	90.2	20.4	89.2
1.0 : 1.4	5.47	74.1	17.6	24.3	100.0	17.8	103.4
1.0 : 1.6	5.98	73.6	16.9	22.0	105.8	15.8	104.8
1.0 : 1.8	6.28	76.8	16.5	23.8	110.4	13.9	101.6
1.0 : 2.0	5.10	74.6	12.5	20.7	110.4	10.0	113.4

Note: Amount of catalyst = 0.18 per cent on weight of pre-condensate.
Each breaking strength and crease-recovery test is the average
of five tests.

TABLE III

COMPARISON OF CREASE-RECOVERY ANGLES OF
TREATED AND UNTREATED RAMIE FABRICS

Test Number	Treated		Untreated	
	Warp	Filling	Warp	Filling
	Crease- Recovery (degrees)	Crease- Recovery (degrees)	Crease- Recovery (degrees)	Crease- Recovery (degrees)
1	106	107	57	62
2	106	102	54	57
3	106	99	53	58
4	104	108	54	53
5	107	108	52	58
average	105.8	104.8	54.0	57.6

Note: The fabric was treated with

Urea : Formaldehyde = 1.0 : 1.6

Catalyst = 0.18 per cent

The increase in Crease-recovery by treatment:

Warp direction = 95.92 %

Filling direction = 91.24 %.

TABLE IV

COMPARISON OF THE MOISTURE CONTENTS OF THE
TREATED AND UNTREATED FABRICS

Test Number	Treated			Untreated		
	C (gm.)	D (gm.)	% Moist.	C (gm.)	D (gm.)	% Moist.
1	2.12	1.98	6.69	1.68	1.54	8.34
2	2.11	1.97	6.63	1.72	1.58	8.14
3	2.04	1.90	6.86	1.80	1.65	8.33
Average			6.70			8.27

Note: Moisture Content, per cent = $\frac{C-D}{C} \times 100$

where: C = Conditioned weight of specimen, and

D = Bone dry weight of specimen.

Decrease in Moisture Content after crease-resistant treatment
= 18.98 per cent.

If the moisture content of the treated fabric was calculated
with reference to cellulose and not to the gross weight of
cellulose and resin, then

Dry pickup of fabric = 16.9 (Table II, page 44).

Moisture Content, per cent = $\frac{6.70}{(1 - 0.169)} = 8.07$

The value obtained, 8.07, is very close to the moisture
content of untreated fabric, 8.27.

TABLE V

COMPARISON OF BREAKING STRENGTH AND PERCENT STRETCH OF
TREATED AND UNTREATED RAMIE FABRICS (DRY AND WET)

Test Number	Treated				Untreated			
	Warp		Filling		Warp		Filling	
	Breaking Strength (lbs.)	Stretch (%)	Breaking Strength (lbs.)	Stretch (%)	Breaking Strength (lbs.)	Stretch (%)	Breaking Strength (lbs.)	Stretch (%)
DRY:								
1	22.5	2.08	13.5	4.17	46.0	6.77	47.5	5.74
2	21.5	2.08	15.0	4.17	54.0	7.28	42.0	5.74
3	22.0	2.62	17.0	3.12	47.0	5.74	48.5	5.74
4	23.0	2.62	15.0	4.17	43.0	5.74	42.0	5.74
5	21.0	2.62	18.5	3.12	45.0	7.28	51.5	6.77
Average	22.0	2.40	15.8	3.75	47.0	6.56	46.3	5.95
WET:								
1	27.5	3.64	23.0	6.77	63.0	9.38	56.0	8.34
2	29.5	4.17	22.0	6.77	56.0	10.42	59.0	8.86
3	29.0	3.64	22.5	6.77	67.0	9.38	50.0	8.34
4	30.0	3.64	22.0	6.77	55.0	9.90	56.0	8.34
5	29.0	4.17	21.0	7.28	61.0	10.42	54.0	10.42
Average	29.0	3.85	22.1	6.87	60.4	9.90	55.0	8.86
Note:								
					Warp	Filling		
Decrease in Breaking Strength (dry) by crease-resistant treatment.....					53.19 %	65.87 %		
Decrease in Percent Stretch (dry) by crease-resistant treatment.....					63.41 %	36.98 %		
Increase in Breaking Strength when wet:								
Treated Fabrics.....					31.87 %	33.13 %		
Untreated Fabrics.....					28.51 %	18.76 %		
Increase in Percent Stretch when wet:								
Treated Fabrics.....					60.41 %	83.20 %		
Untreated Fabrics.....					50.91 %	65.71 %		

TABLE VI

COMPARISON OF RESULTS OF ABRASION TEST

Test Number	Treated	Untreated
	Abrasion cycles	Abrasion cycles
1	155	243
2	148	240
3	148	237
4	147	251
Average	149.5	242.8

Note: Decrease in Resistance to Abrasion
after crease-resistant treatment
= 38.44 per cent.

TABLE VII

EFFECT OF WASHING ON CREASE-RECOVERY ANGLES

Test Number	Crease-Recovery (degrees)							
	1st Washing	2nd Washing	3rd Washing	4th Washing	5th Washing	6th Washing	7th Washing	8th Washing
WARP:								
1	100	117	97	101	97	97	100	97
2	108	104	108	103	103	94	99	95
3	106	95	105	100	97	98	100	90
4	104	106	99	103	100	96	96	96
5	111	104	98	98	99	97	93	100
Average	105.8	105.2	101.4	101.2	99.0	96.4	97.2	95.6
FILLING:								
1	100	101	103	100	102	106	90	92
2	112	104	111	92	103	97	98	97
3	102	107	95	107	98	97	93	97
4	95	100	96	103	94	93	105	94
5	108	103	103	107	99	100	97	90
Average	103.4	103.0	101.6	101.8	99.0	98.6	96.6	94.0
Note: Washing Bath: 0.5 % soap and 0.2 % sodium carbonate. Temperature: 160°F. Time: 45 minutes for each washing. Decrease in Crease-Recovery Angle (degrees) after 8 washings:								
					Warp	Filling		
	Before washing (Table III).....				105.8	104.8		
	After 8 washings.....				95.6	94.0		
	Decrease in Crease-Recovery (%)..				9.64 %	10.30 %		

TABLE VIII

COMPARISON OF COLORFASTNESS OF DYED TREATED AND
UNTREATED FABRICS TO LIGHT

Name of Dye	Treated		Untreated	
	Hours	Class.	Hours	Class.
Pontamine Fast Brown 4RL	35	5	25	5
Pontamine Fast Pink BL	15	4	10	3-4
Pontamine Fast Blue 2GL	10	3-4	20	4-5
Pontamine Diazo Blue 6G (developed with beta- Naphthol)	20	4-5	10	3
Sulfanthrene Blue GR Paste	40	5-6	40	5-6
Sulfogene Brown RBNCF	15	4	10	3

Note: Units were expressed in numbers of hours which show the first signs of fading.

Classification of fastness to light was made according to A.A.T.C.C. method.*

* "Colorfastness to Light," A. A. T. C. C. 1949 Year Book, pp. 105-106.

TABLE IX

COMPARISON OF COLORFASTNESS OF DYED TREATED AND
UNTREATED FABRICS TO WASHING

Name of Dye	Treated	Untreated
	Colorfastness to Washing (Class.)	Colorfastness to Washing (Class.)
Pontamine Fast Brown 4RL	2	1
Pontamine Fast Pink BL	3	1
Pontamine Fast Blue 2GL	3	2
Pontamine Diazo Blue 6G (developed with beta- Naphthol)	3	2
Sulfanthrene Blue GR Paste	3	3
Sulfogene Brown RBNCF	3	3

Note: The test was made according to A.A.T.C.C. method (Colorfastness of cotton and linen textiles to commercial laundering and to domestic washing, A.A.T.C.C. 1949 Year Book, pp. 91-92).

Class 1 -- Specimens which show no appreciable change in color and no appreciable staining of the attached test cloth in Test No. 1 (105°F, 0.5 % soap solution, 30 minutes washing) shall be reported as having Class 1 colorfastness to commercial laundering and domestic washing.

Class 2 -- Specimens which show no appreciable change in color and no appreciable staining of the attached test cloth in Test No. 2 (120°F, 0.5 % soap solution, 30 minutes washing) shall be reported as having Class 2 colorfastness to commercial laundering and domestic washing.

Class 3 -- Specimens which show no appreciable change in color and no appreciable staining of the attached test cloth in Test No. 3 (160°F, 0.5 % soap solution, 0.2 % solution of sodium carbonate, 45 minutes washing) shall be reported as having Class 3 colorfastness to commercial laundering and domestic washing.