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"PERMANENT SIZING OF WARPS"

by

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A report of research conducted at the Institute of Textile Technology under contract from the Office of The Quartermaster General through the National Research Council.

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With the large requirement for cotton duck that confronted the Quartermaster Corps during the war, it became necessary to turn to single yarn fabrics for Army tentage in place of plied yarn fabrics which had originally been specified. These single yarn fabrics had to be sized in order to weave properly, and then had to be desized in order to remove the starch which otherwise would form a ready nutrient for micro-organisms. It appeared desirable, as long as such large requirements for single yarn fabrics for tentage existed, to find a warp sizing material which would not support the growth of microorganisms and which could accordingly be left in the fabric, i. e., a warp sizing which would eliminate the need for desizing of the cloth and which would be compatible with the fire, water and weather resistant finish subsequently to be applied to the tentage fabric.

The Institute of Textile Technology was requested to investigate available materials for this purpose to determine what materials could be so used and the technology of their application.

At the end of the war with the cancellation of the tentage production program, it did not appear necessary to carry the work for the present beyond the preliminary findings which the staff of the Institute of Textile Technology had obtained. Their work has shown that there are svailable materials which could be used satisfactorily for the desired purpose in the event that a requirement was to develop for the production of fabrics of this type.

In releasing this report to the public it was recognized that to make it of general value it would be desirable to identify the products tested. The industrial concerns which supplied them have accordingly been contacted, and the listings by product name which are made herein are with the concurrence of the producers whose products are so identified.

It is believed that further research in this field should be productive of results having military as well as civilian applications. The concept of using a warp sizing to impart certain desired properties to a fabric which can be left in the fabric offers a line of research which could have important applications for many textile uses.

January 1947

S. J. KENNEDY Assistant Director For Textiles, Clothing and Footwear

4	Availability Codes		
Dist A-/	Avail and/or Special		
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### OBJECT :

The object of this research project has been to find a permanent, mildew-resistant material to replace starch as a warp size for tent twill.

## SULMARY :

The work has progressed to the point at which recommendations can be made for mill trials. The recommended sizes are Dow's Carboxymethocel A, Monsanto's Stymer, Rohm and Haas' RHoplex MR, and American Cyanamid's Aerotex 625. Indications are that the first two materials have considerable assurance of success; performance of the latter two cannot be predicted.

## CONCLUSIONS:

Of 43 materials, or combinations, studied for possible permanent warp sizing, 4 proved satisfactorily mildew-resistant, with qualities which might make them acceptable as warp sizes. Materials studied were in the following classes;

- 1. Cellulose and its derivatives;
- 2. Gums and resins;
- 3. Synthetic latexes.

The 4 recommended materials are Carboxymethoce' A, Stymer, RHoplex MR, and Aerotex 625. Carboxymethocel A and Stymer are applied from ammoniacal solutions. Permanency is acquired by evaporation of the water and ammonia. On the basis of previous work done on Stymer (4), and studies by Pacific Mills on the sodium derivative of Carboxymethocellulose S, as well as studies in this laboratory, these two materials should be successful warp sizes.

RHoplex MR and Aerotex 625 are applied from emulsions. Permanency is obtained by evaporation of the dispersing medium. Recommendations on these materials are based on studies in this laboratory alone.

Patents which might apply to this use of these materials are as follows:

 Carboxymethocel A:
 US 1,950,664; 1,978,785; 2,106,298, and

 Stymer:
 Patents applied for
 2,331,859

 RHoplex MR;
 US 2,368,948

 Aerotex 625;
 US 2,376,595.

## RECOMMENDATIONS:

It is recommended that extensive mill trials be conducted on Carboxymethodel A, Stymer, RHoplex MR, and Aerotex 625, starting with the operating conditions stated below. These recommended conditions are based on a speed of 25 yd./min.

- Carboxymethocel A: Dissolve a low viscosity grade of Carboxymethocel A in 0.35 lb. of NH<sub>3</sub> solution per pound of Carboxymethocel A. A concentration of 5% Carboxymethocel A will probably be found the maximum that can be used, because of the viscosity. An alkali-stable wetting agent should be used in the bath. Box temperature can be room temperature, or slightly higher. Elevated temperatures will drive off ammonia. Size pick-up should be about 5% of the weight of the dried yarn. Weaving should be at not over 65% relative humidity. Above this, the size may absorb water, becoming soft and swollen.
- Stymer: Dissolve in about 1 lb. of 28% NH3 solution per pound of Stymer. Instructions from the manufacturer called for 5% ammonia solution. However, runs have been made with as low as 0.65 lb. 28% solution per pound of Stymer. A concentration of 5% Stymer will probably be found the maximum that can be used, because of the viscosity. Stymer V-30 was used on this project. The box temperature may be up to about 50° C. Size pick-up should be about 5% of the weight of the dried yarn.
- RHoplex MR: Dilute the 50% emulsion to 10%, with water, while agitating. Box temperature can be from room temperature to about  $50^{\circ}$  C.
- Aerotex 625; Dilute to 10%, with water, while agitating. It is believed desirable to raise the pH from its original value of 4 to 6 or 7. This can be done by careful addition of ammonia solution, with agitation, just before using.
- Methods of size pick-up analysis; Carboxymethocel A and Stymer can be extracted from the yarn by heating about 30 minutes in 5% ammonia solution, at 80-90°C., and then rinsing in hot running water. After determining the original moisture in the sample, by conventional methods, the size is determined by difference. Because ammonia will extract matter from unsized yarn, blanks should be run, and corrections made on the dry basis.

No methods were found for analyzing RHoplex MR or Aerotex 625, nor was there time to discuss the problem with the manufacturers. If a solvent is found, analyses can be run on these materials just as for Stymer and Carboxymethocel A, running a blank at the same time.

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## EXPERIMENTAL VORK

Belection of materials for study as permanent warp sizes was made under the following classifications.

1. Cellulose and its derivatives

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No. 25
   No. 9
   "Cellosize"* WS-100 (water soluble hydroxyethylcellulose,
                         compounded to obtain permanency)
   Carboxymethocel A (aluminum solt of celluloseglycolic acid)
   Dow Emulsion X-258 (or X-259) (ethylcellulose emulsion)
2. Guns and resins
   No. 2 (phenolic and alkyd resins)
    No. 5 (maleic and alkyd resins)
    No. 8 (phenolic and alkyd resins)
    No. 6 (maleic and alkyd resins)
   No. 4 (alkyd resin)
    No. 48 (alkyd resin)
   Dimethylolurea (urea-formaldehyde) and an alkyd resin
   Dimethylolurea (urea-formaldehyde) and a pH6 alkyd resin
   Dimethylolures (urea-formaldehyde) and a pH4 alkyd resin
   Dimethylolurea (urea-formaldehyde) and Methocel (methyl
      cellulose)
    Dimethylolurea and starch (pH4) (urea-formaldehyde resin)
    Dimethylolurea pH8 (urea-formaldehyde)
    Dimethylolurea pH4 (urea-formaldehyde)
    No. 52 (urea formaldehyde)
    RHoplex ER (acrylic resin)
    RHoplex MR (acrylic resin)
    Rhoplex WA-5 (acrylic resin)
    RHoplex W-66 and RHoplex ER (acrylic resin)
    Neville R-17 (coumarone resin)
    Aerotex** Resin 7513 (type unknown)
    Aerotex** Resin 625 (type unknown)
    Elvacet (polyvinyl acetate)
    Methaorol BP (acrylic resin emulsion)
    TLF 223 E (vinyl acetate emulsion)
    No. 33
    No. 34
    Stymer (styrene derivative)
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Dow X-319 and X-276 (styrene polymers)

Trademarked name of Carbide and Carbon Chemicals Corporation
 Trade mark of American Cyanamid Company

## 3. Synthetic latexos

Dow Latex X-275 (ternary polymer comprising styrene, butadiene and vinylidene) Dow Latex X-337 (invert styraloy comprising styrene and butadiene) Experimental Latex X-512 (copolymer of styrene and butadiene) Neoprene Latex Type 571 (emulsion polymers of chloroprene with rosin soap) Neoprene Latex Type 572 (emulsion polymers of chloroprene with rosin soap) No. 39

Unclassified

Formula 1231-1 (Tennessee - Eastman Product) No. 15 Volclay (bentonite) Nopco EB (National Oil Products)

The first phase of the project was the mildew-testing of these materials. This phase was undertaken in conjunction with the Quartermaster Depot at Jeffersonville, Indiana. Fabric samples were prepared by the I.T.T. laboratory and tested by the Jeffersonville laboratory. The following samples were prepared:

- 1. Glass microscope slides coated with sizing.
- 2. Same as 1, but half of this coating covered with QMC finish 6-345.
- 3. Same as 1, but all of this coating covered with QMC finish 6-345.
- 4. Desized 12 oz. duck, impregnated with the size.
- 5. Same as 4, but treated with QMC finish 6-345.
- 6. Desized 12 oz. tent twill, impregnated with the size.
- 7. Same as 6, but treated with QMC finish 6-345.

The glass slides were immersed in an enriched soil suspension and incubated for 14 days. They were then examined visually for fungus growths. The cloth samples were cut into 5.5" ravelled strips. All were immersed in an enriched soil suspension and incubated. Those without QMC finish were incubated for 4 and 7 days: those with finish, for 7 and 14 days. All cloth samples were compared with desized and starch-sized samples. They were tested for tensile strength after incubation.

Testing of the glass slides showed little, if any, correlation with the cloth samples. Testing of glass slides was then stopped. Many of the cloth samples had to be repeated because of inadequate desizing of the goods received for sample preparation. All cloth for subsequent samples was then desized in the laboratory before use. Carboxymethocel A RHoplex MR Nopco EB Neville R-17 Aerotex 625 Polyvinyl acetate Dow Latex X-275 Dow Emulsion X-258 Dimethylolurea (urea-formaldehyde) and an alkyd resin (pH6) Dimethylolurea (urea-formaldehyde) and an alkyd resin (pH4) Dimethylolures and Methocel Dimethylolurea (pH8) Dimethylolurea (DII4) Methacrol BP No. 39 Stymer.

The second phase of the project was the preparation of yarns, sized with materials approved for mildew resistance, for single-end testing. Search of literature revealed no single-end tests which were truly indicative of "weavability". However, with the knowledge that starch is an accepted size for weaving, all other materials were to be compared to starch-sized yarns. Tests were to include breaking strength, elongation at break, stiffness, abrasion resistance, and fiber lay. Literature indicated that strength, stiffness, and abrasion resistence should be increased, but that an excessive increase was definitely harmful. Therefore, potential sizing materials were to be approved if they improved these yarn qualities as much as starch did.

Yarns were sized on a modification of the slasher designed by the Clemson laboratory of the USDA, (1) built by Pacific Mills laboratory in Newark, N.J. (see Fig. 1). Comparison with the description of the original design shows that Pacific Mills laboratory changed the location of the drier heaters and the fan for the drier. Studies on this project indicated that the new location of the heathers (below the drier chamber) was so close to the yarn on the bottom drier rolls that materials were melted off the yarn if their melting or softening point was as low as 125° C. Furthermore, the flyw of air was so low and unbaffled that the drier became a radiation drier, rather than a hotair drier.

The slasher was modified by the I.T.T. laboratory to provide extra driven rolls, and a constant yarn speed through the machine. Everything possible was done to prevent stretching. It was believed that stretching would be nonuniform among the materials studied, and, since no lease rods were used, it would be unnecessary. The rewind was a reel with 2 collapsible spokes, driven by a cord belt with as little power as possible. This low power prevented stretching of the yarn at this point. The slasher was run at 4-6 yd./min.; bath temperature 90-92° C. for starch, 50° C. for all other materials; drier temperature 65-95° C., varying for different materials.

The slasher was run with 20 ends spaced evenly over 4.5", condensed to 3" at the rewind. This was done to assure as much uniformity among the ends as possible. The yarns were divided into 3 sets in the drier, to obtain single-end drying. The sets were of different lengths. However, analyses showed moisture variations among the sets were within a total range of 1%.

Impossible operation or obvious lack of stiffness eliminated all materials except Carboxymethocel A, Stymer, RHoplex MR, and Aerotex 625. The following is a discussion of the tests run on these, with comparisons with starch, SR Gum, and Elvanol (polyvinyl alcohol).

Abrasicn testing of the sized yarns was attempted on a modified operation of the Walker abrader (6). The yarn was threaded over the pins of the testing machine, but not looped over itself. This gave a strict yarn-to-steel abrasion, without the yarn-to-yarn abrasion of the original method of operation. Preliminary work on this test, with known acceptable warp sizes, showed little, if any, correlation with "weavability". The test was then abandoned for this project. Abrasion tests which used carborundum paper (5) were not considered because experience of others indicated insufficient uniformity in the paper, and too much difference, between adjacent grades. Furthermore, this type of abrasion is not encountered in weaving. Because of lack of time, no further abrasion test was developed for this project.

Stiffness of the sized yarn was determined by forming a loop of the yarn, with the ends held together in a clamp, and an adjustable chain weight attached to the center (bottom) of the loop. As weight was applied, readings of the weight necessary to bring the sides of the loop together were taken when the sides were 2.0, 1.5, and 1.0 mm. apart at a constant distance below the clamp. The following results were obtained at a separation of 1 mm.

	Chain Length			
8-10 cm.	15 cm.	18 cm.		
Elvanol 3%	Starch 8%	SR Gum 10%		
RHoplex MR 5-10% Carboxymethocel A 5% Stymer 3%	Primer o%	Aerotex 625 5-10%		

Comparison of readings at all 3 points with visual judgment during slashing indicated that all of these materials were of the same degree of stiffness as starch.

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Tensile strengths and elongations at break are shown in the following table. Lack of stretch during slashing, of course, affects the elongation results. As can be seen, they are all within approximately the same range.

Size	Breaking Strength (grams)			Elongation at Break (%)		
	Max.	Min.	Aver.	Max.	Min.	Aver.
· ·	580	315	445	8.2	3.0	5.8
Unsized ·	700	385	535	8.0	5.0	6.3
Starch $\frac{\pi}{2}$	670	365	529	7.0	4.0	5.7
SR Gum	685	380	524	7.0	4.5	5.8
Elvanol 3%	540	385	458	7.0	5.0	5.7
Stymer 6%	640	420	473	7.0	4.0	5.8
Stymer 3%	540	335	463	8.5	5.5	6.6
Carboxymethocel A 5%	600	470	529	.7.5	5.0	6.2
Carboxymethocel A 2%	530	330	456	6.5	3.0	5.2
RHoplex MR	535	385	458	6.6	4.5	5.7
Aerotex 625	730	422	530	7.5	5.0	0.2

Fiber lay can be judged by the accompanying photographs (Figs. 2-11, inclusive). They are all as good as or better then starchsized yarns. These photographs were selected from groups developed with varying scales of gray, because they showed the fibers the most clearly, and agreed the best with visual judgment of the criginal samples.

In addition to the above tests, visual tests were made of stiffness during slashing, and of hand loop and hand break behavior (7). These all indicated that the 4 best materials were as good as or better than starch.

The third phase of this project was to have been mill trials. This phase has not been run because of the request of the Quartermaster Corps that the project be closed as of June 30, 1946. Work has been delayed by delayed mildew-tests from Jeffersonville, the difficulties in running the slasher on certain materials, and the failure of contemplated tests of sized yarns.

#### DISCUSSION:

A search of literature revealed no tests for warp sizing that would foretell their performance on the loom. Work by others (2,3,5) indicated general qualities required in a sized yarn. These are increased abrasion resistance, increased tensils strength, with maximum retention of elasticity, increased stiffness, and good fiber lay. There, were also indications that stiffness, abrasion resistance, and tensile strength should be increased only within limits. The sizing material, itself, must not dust off too badly; nor must it be scraped off in any form that would adhere to the heddles, etc., during weaving. On the basis of this literature study, work on this project was planned to study the above properties of the sizing materials considered, and to attempt to equal the values for these properties given to yarn by starch. Because of inadequecies found, to date, in the Walker abrader test method, and delays in obtaining other tests, judgment of weavability of the materials studied is supported by little data. However, those obtained on this project, and by Robinette and Pacific Mills, give good grounds for conducting extensive mill tests on the materials recommended.

The design of the experimental slasher limits its use to starch, cellulosic, and similar materials. Redesign of the slasher was underway when work was stopped.

The mildew-resistance test used at Jeffersonville was found to give comperable results for sized, but not finished, goods. Almost all samples with the QMC #6-345 finish gave good mildew resistance. Considering the results of field service of such finished febrics, the Jeffersonville test seems to be lacking in severity for finished goods.

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FIGURE I DIAGRAMATIC SKETCH OF SLASHER 3cale 2 : 1:0"

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Figure 5.

Starch Sized, 11% Size

(2X)



(2X)



