STUDIES ON BLENDING OF COTTON WITH POLYESTER

Part I. Improving the Properties of Cotton Component Via Chemical Modification.

BY

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INTRODUCTION:

Cellulose, in particular cotton, is the most widely used textile fibres all over the world. This preeminence is due to a happy combination of properties such as abundance, low cost, fine crossection, high strength and durability, high thermal stability and good mechanical properties, ability to absorb moisture, easy dyeability, wearing comfort, etc...

However, cellulonic fibres have some inherent drawbacks which can be briefly formulated as follows: (a) poor solubility in common solvents, (b) poor crease resistance, (c) Lack of thermoplasticity, and (d) poor dimensional stability /1, 2, 3/.

1. Improvements of cellulose properties via changing its physical structure:

The physical structure of cellulose can be changed by either swelling or regeneration; cellulose can be swollen in a suitable solution of swelling agent, such as caustic soda of mercerizing strength, then partially deswollen by removal of the swelling agent. Practically, there is no change in the chemical structure of the fibres /4,5/ but there are considerable changes in the physical fine structure resulting in enhancement of the fibre strength, lastre and reactivity.

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In regeneration, the native cellulose is treated to render it soluble and then by regenerating it from solution /6,7/. This process, which is the basis of rayon and cellophane structure from a cellulose I lattice to a cellulose II lattice and results in a corresponding modification of the physical structure.

2. Improvements of cellulose properties by chemical Modifications:

The chemical structure of cellulose can be changed in three ways /8/.

a) By substitution of the cellulose hydroxyls where by the cellulose molecules are altered through introducing bulky side groups either by an esterification or by an etherification reaction. The esterification reaction is exemplified below by acetylation and phosphorylation of cotton whereas the etherification reaction is exemplified by cyanoethylation and partial carboxymethylation of cotton.

b) By reacting cellulose with bi-or polyfunctional compounds which results in the production of crosslinks and/or resinsification products in the cellulose there by stabilizing its structure.

c) By preparing a branched cellulose or what is called copolymer of cellulose.

Chemical modification of cotton cellulose via partial carboxymethylation has been reported to exert considerable influence on the physical, mechanical and chemical properties of cotton /9, 10, 11, 12, 13/.

Partially carboxymethylated cotton may be simply prepared by treatment of cotton fibres, yarns, or Fabrics with chloroacetic acid and sodium hydroxide /14, 15, 16, 17, 18, 19,20,21/.
The process is carried-out by impregnating the cotton with an aqueous solution of chloroacetic acid followed by treatment with strong sodium hydroxide solution.

This research programme has three-Fold objective:

a) Increasing the extension of cotton fibres through slack mercerization and partial carboxymethylation,

b) Blending the modified cotton fibres and polyester fibres at different ratios and studying the relations between the fibre properties in the components and their blends and,

c) Spinning the blended fibres to yarns having the same count under similar processing conditions and studying the optimum conditions of blending the modified cotton with polyester.

The present work describes the chemical modification of cotton fibres via partial carboxymethylation and slack mercerization and the effect of these modification on the major properties of cotton fibres.

EXPERIMENTAL:

Materials:

Raw cotton (RC), alkali treated cotton (AC), Low partially carboxymethylated cotton (LC), medium partially carboxymethylated cotton (MC) and high partially carboxymethylated cotton (HC) were used throughout this investigation.

Slack Mercerization of Cotton Fibres:

Cotton fibres in the form of bulks (2Kg) were treated with an aqueous solution of caustic soda (12 N) for 2 minutes, then the fibres were squeezed so as to give 130% - 140% wet pick-up, and stored for 5 minutes. At this end, the fibres were washed with water, neutralized with 1% Hydrochloric acid, washed again thoroughly with water and air-dried.
These fibres will be referred to as "Alkali treated cotton" and will serve as a control for partially carboxymethylated cottons.

Preparation of partially carboxymethylated cotton:

Cotton fibres—in the form of stocks (2 Kg) were first treated with an aqueous solution of "mono-chloroacetic acid" (3N) for 5 minutes, using a material to liquor ratio 1:20. After this treatment the fibres were squeezed to a wet-pick up of 75–85%, then treated with aqueous solution of caustic soda (12 N) for 2 minutes. The fibres were then squeezed to a wet pick-up of 130% – 140% and stored in polyethylene cover for 30 minutes. Two other fibre stocks were treated in a similar way except that the storing periods after the caustic soda treatments were 60 minutes and 120 minutes.

After each period the fibres were washed with water, neutralized with dilute hydrochloric acid (1%), washed thoroughly again with water and air dried.

Measurements of Fibre Properties:

The properties of the fibres were measured according to the procedure described in A.S.T.M. These properties were:

1) Determination of carboxylic groups:
The calcium acetate method was used for the determination of carboxyl content /22/.

2) Fibre Fineness:
The fibre fineness was measured using sheffield Micronair tester, from each variety six samples were tested.

3) Fibre Length: Fibre length and length distribution were measured by:
a) Digital Fibrograph: From each variety 3 samples were tested. Fibre length at 2.5%, 12.5%, 50%, 66.7% span length and uniformity ratio were measured.

b) Sutter-web tester: from each variety 3 samples were tested. Upper quartile length, standard deviation and coefficient of variation.

4) Fibre Strength and Elongation: Fibre strength and elongation were measured using stelometer (1/8 inch gauge length).

5) Moisture Regain %. The moisture regain % was determined using the electric shirley moisture meter.

6) The microscopical Investigation: It is expected that chemical treatments would cause decrease in thickness of the primary wall of the cotton fibre. To check this, the fibre were examined under light microscope using a swelling solution as given below: 100 g. ofm chloride and 32 g. potassium iodide were dissolved in 34 ml distilled water, then iodine was added till saturation, the solution was then filtered through filter paper.

Results and Discussions:

1- Carboxyl content: To start with, the different cotton substrates were analyzed for their carboxyl-content with a view of determining the extent of chemical modification.

   The results obtained are shown in Table 1. It is seen that the raw cotton acquires carboxyl content which is appreciably higher than that of alkali treated cotton. This could be ascribed to the removal of carboxyl-containing materials such as pectin under the action of the strong caustic soda solution used in the treatment—substantial amount of carboxyl groups are present in partially carboxymethylated cottons.
Table (1): Carboxyl content of Raw, Alkali treated and Partially carboxymethylated cottons.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Substrate</th>
<th>Carboxyl content Milli eq. -COOH/100 g Cellulose</th>
</tr>
</thead>
<tbody>
<tr>
<td>RC</td>
<td>Raw cotton</td>
<td>9.7</td>
</tr>
<tr>
<td>AC</td>
<td>Alkali-treated cotton</td>
<td>5.82</td>
</tr>
<tr>
<td>LC</td>
<td>Low partially carboxymethylated cotton</td>
<td>16.68</td>
</tr>
<tr>
<td>MC</td>
<td>Medium partially carboxymethylated cotton</td>
<td>21.34</td>
</tr>
<tr>
<td>HC</td>
<td>High partially carboxymethylated cotton</td>
<td>24.4</td>
</tr>
</tbody>
</table>
This is rather expected since the carboxymethyl groups expressed as carboxyl groups. As already indicated in the experimental work, the magnitude of the carboxyl groups can be controlled by increasing the time of partial carboxymethylation.

2- Fibre Fineness: Table (2) shows the fibre fineness of raw cotton (RC), partially carboxymethylated cottons (LC, MC, and HC) and Alcali treated cotton (AC). It is evident that slack mercerization of raw cotton, by treating it in 12 N caustic soda for 2 minutes, brings about a significant change in fibre fineness expressed as Micronaire value. The increase in the latter after slack mercerization amounts to Ca. 25%. A more significant increase in the Micronaire value could be achieved via Partial carboxymethylation of the raw cotton particularly when this chemical modification was allowed to proceed for more than one hour. The Micronaire value in MC and HC is 40% higher than that of raw cotton.

The increase in Micronaire values due to the chemical treatments could be attributed to: (a) shrinkage in fibre length, (b) increase in fibre thickness, (c) increase in moisture regain, (d) removal of low molecular weight cellulose, and (e) change of fibre cross-section to be more circular.

3- Fibre Length: The measurements of fibre length and fibre length distribution for raw cotton before and after slack mercerization and partial carboxymethylation were carried out. The results of these measurements are shown in Table 2. It is seen that both slack mercerization and partial carboxymethylation cause: (1) a decrease in the mean fibre length, (2) an increase in the C.V.% of fibre length, and (3) an increase in the short fibre content.
Table (2): Physical and Mechanical Properties of the Fibres.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>RC</td>
</tr>
<tr>
<td>1- Fibre Fineness:</td>
<td></td>
</tr>
<tr>
<td>Micronaire value</td>
<td>4</td>
</tr>
<tr>
<td>2- Fibre Length:</td>
<td></td>
</tr>
<tr>
<td>a) Sutter-Webb</td>
<td></td>
</tr>
<tr>
<td>U.Q.L. mm</td>
<td>37.1</td>
</tr>
<tr>
<td>C.V.%</td>
<td>32.8</td>
</tr>
<tr>
<td>Short fibres</td>
<td></td>
</tr>
<tr>
<td>% in.%%</td>
<td>6.1</td>
</tr>
<tr>
<td>b) Digital Fibrograph</td>
<td></td>
</tr>
<tr>
<td>2.5% span length</td>
<td>33.78</td>
</tr>
<tr>
<td>12.5% span length</td>
<td>26.16</td>
</tr>
<tr>
<td>(mean length)</td>
<td></td>
</tr>
<tr>
<td>50% span length</td>
<td>15.0</td>
</tr>
<tr>
<td>Uniformity ratio</td>
<td>77.44</td>
</tr>
<tr>
<td>3- Fibre strength</td>
<td></td>
</tr>
<tr>
<td>and elongation:</td>
<td></td>
</tr>
<tr>
<td>Stelometer (1/8)</td>
<td></td>
</tr>
<tr>
<td>Strength g-wt/tex</td>
<td>37.94</td>
</tr>
<tr>
<td>Elongation %</td>
<td>6.46</td>
</tr>
<tr>
<td>4- Moisture Regain%</td>
<td>7.3</td>
</tr>
</tbody>
</table>
The decrease in fibre length and variation in parameters associated with it such as C.V.% and short fibre content are in accordance with previous reports/19, 20/ which attributed this to: (a) Fibre breakage, and (b) Shrinkage in length during caustic swelling.

4- Fibre Strength: Fibre strength of raw cotton before and after alkali treatment and partial carboxymethylation is shown in Table (2). It is clear that alkali treatment causes a substantial decrease in fibre strength. Since no tension was applied during the alkali treatment, the decrease in strength could be associated with:
a) increase in the amorphous regions in cotton on the expense of crystalline regions,
b) decrease in fibre orientation, and
c) reduction in the uniformity of the strength along the fibre.

As it will also be seen later, alkali treatment deteriorates seriously the fibre primary wall. The latter, has been reported to contribute in fibre strength /23/.

Partial carboxymethylation of cotton also brings about a decrease in fibre strength. This is rather the situation encountered with alkali treatment and could be explained on similar basis. It should be pointed out, however, that the decrease in strength caused by partial carboxymethylation tends to lessen as the modification was allowed to proceed further. That is, (HC) shows a strength which is considerably higher than (LC). This reflects the effect of increasing the magnitude of carboxymethyl groups on the strength of the fibre. Nevertheless, the strength of (HC) is still lower than the raw cotton, which again is due to the fact that no stretching of the fibre was made during the modification treatment.
5- Elongation: Data of elongation at break (%) of raw cotton before and after being slack mercerized or partially carboxymethylated to different degrees are shown in Table 2. It is seen that alkali treatment of raw cotton is accompanied by a significant increase in its elongation at break. An increment of 80% in elongation at break could be achieved.

A more significant increase in elongation at break could be obtained by partial carboxymethylation of raw cotton. Increments in elongation as high as 115% could be brought about by this chemical modification treatment. The significant enhancement in elongation at break observed with alkali treated cotton could be interpreted in terms of decreased crystallinity and orientation of the fibre as well as the deterioration of the primary wall under the influence of the strong caustic soda used in the treatment. However, the increased ability of the alkali treated cotton to absorb water cannot be ruled out. The same explanation is valid to partially carboxymethylated cotton fibres.

The higher increase in elongation of the partially carboxymethylated cotton fibres as compared with alkali treated cotton is possibly because of one or more of the following factors:

a) The introduction of carboxymethyl groups into the molecular structure of cotton cellulose.

b) The increased swellability of the fibre due to hydrophilic nature of these groups, and

c) Ionization and consequent repulsion between chemically bonded carboxymethyl groups which prevent fibrils and molecular chains becoming contiguous.
6- Moisture Regain %: The moisture regain of raw cotton before and after alkali treatment and partial carboxymethylation is shown in Table 2. It is evident that alkali treated cotton acquires much higher regain than raw cotton. The same holds true for partially carboxymethylated cottons. The latter even possess higher moisture regains than the alkalitreated cotton. The improvement in moisture regain for alkali treated cotton could be explained on the basis of swelling and the consequent increase in the amorphous region together with the serious deterioration in the primary wall of the fibre by the effect of strong caustic soda. Whereas the higher improvements in the regain found with partially carboxymethylated cottons could be attributed to the presence of carboxymethyl groups in the molecular structure of cotton. Presence of these groups may be expected to increase the hydrophilicity of the modified cotton by virtue of their polarity. In addition, the mere presence of these groups will spread cellulose chains thereby permitting easier access to water molecules.

7- Microscopical Investigation: Raw cotton fibres before and after being subjected to alkali treatment and partially carboxymethylation were examined microscopically in the swollen state using "Bargash solution" as described above. Longitudinal view and pre-determined length of the fibres (0.6 of the hand microtone cycle) was assessed and the mean number of beads calculated. Figures (1 and 2-a - 2.m) illustrate the effect of the said chemical treatments on raw cotton fibres. It is observed that alkali treatment has a serious adverse effect on the primary wall. The latter nearly disappeared after the alkali treatment.
Similar situation is encountered with the partial carboxymethylation treatment. Both the alkali treated and partially carboxymethylated fibres show much lower number of beads as compared with the raw cotton. Previous work \(^/23/\) has disclosed a relation between the number of beads and fibre strength indicating partial contribution of the primary wall to the strength. Hence the lower number of beads observed with the chemically treated cottons is indicative of a decrease in their strength when compared with that of raw cotton.

CONCLUSIONS:

From the previous discussions and investigations the following conclusions may be drawn:

1- Partially carboxymethylated cotton shows higher micronaire values as compared with raw cotton and alkali treated cotton particularly at higher level of chemical modification.

2- Both partial carboxymethylation and alkali treatment of cotton fibres are accompanied by a decrease in fibre length and length uniformity ratio.

3- There is a marginal decrease in fibre strength after partial carboxymethylation and alkali treatment.

4- Partial carboxymethylation of cotton fibres causes a significant increment in breaking elongation. It increases from about 6.5% for raw cotton to about 14% for partially carboxymethylation cotton while it is 11% for alkali treated cotton.

5- Moisture regain of partially carboxymethylated cotton is considerably higher as compared with raw cotton and alkali treated cotton. The moisture regain amounts to 7.3% for raw cotton and 7.6% for alkali treated cotton while it was as high as 9.7% for partially carboxymethylated cotton.
REFERENCES:


Fig. (1) Effect of Modification on The primary Wall of Cotton Fibres.
(2-a)

(RC) Without Swelling Solution.

(2-b)

(2-e)

(LC)

(2-d)
Effect of Modification on
The Carboxyl Content
Effect of Modification on The Micronaire Value
Effect of Modification on Fibre Type
Mean Fibre Length

PC  LC  MC  HC  AC  P

Mean Fibre Length (mm)

35
30
25
20
15
10
5
0
Effect of Modification on Fibre Bundle Strength (Stelometer $\frac{1}{g}$)
Effect of Modification on Fibre Bundle
Elongation % (Stiometer) \( \left( \frac{x}{y} \right) \)
Effect of Modification on Moisture Regain %

Fibre Type

RC  LC  MC  HC  AC  P

Moisture Regain %