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Heterogeneous Hydrolysis of Cotton Cellulose Treated with Different Swelling Agents

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Introduction

The heterogeneous acid hydrolysis of cellulose has been studied extensively to elucidate the fine structure of cellulose.[1–7] The properties of hydrocelluloses obtained from celluloses of different origins or after different pretreatments have been studied.[8–11] In each case, it has been found that the degree of polymerization (DP) rapidly decreases in the initial period of hydrolysis and reaches a constant value which is not affected significantly even by prolonged hydrolysis. This leveling-off degree of polymerization (LODP) is different with different celluloses and is considered an index of the length of the crystallites obtained.[12,13] Values of crystallite lengths obtained by electron microscopy of the "limit hydrocelluloses" agree well with those derived from LODP measurements.[9,10] Recrystallization has been shown to occur under conditions of mild hydrolysis.[8,14–17] The extent of recrystallization is favored by the polar nature of hydrolysis media and by high temperatures.[18] If hydrolysis is continued after the amorphous portion is removed, there is no change in DP but the copper number continues to increase.[1] Degradation of the crystallites has been shown to occur through attack on the ends of the crystallites at a rate which is approximately inversely proportional to their mean lengths.[19,20]

The effect of swelling treatments on the crystallinity of cellulose has also been investigated by various authors. Hermans and Weidinger[21] found that the crystallinity of rayons increases from 40–50% after treatment with 18% sodium hydroxide followed by removal of alkali by boiling water. Mercerization of jute, hydrolyzed with increasing concentrations of sulfuric acid at 20°C., has shown a progressive increase in proportion to the hydrate modification with increasing severity of the acid treatment,[22] the transformation being complete when the acid concentration is 500 g./liter. Treatment with anhydrous ethylamine has been shown to produce rapid and extensive decrystallization of cellulose.[23] The extent of decrystallization is independent of the DP of cellulose. Crystallite orientation is essentially unaffected by the treatment. The physical properties of mercerized and decrystallized cottons have been compared by Orr et al.[24] The stability of the complex formed by ethylenediamine with cellulose has

been investigated by Segal and Loeb[25,26] who found that it is stable to non-polar solvents which do not contain oxygen. Polar oxygen-containing solvents cause a reconversion of the crystal structure to the cellulose I modification and also promote recrystallization. The changes in crystallite orientation of native cellulose produced by several swelling agents have been studied in this laboratory.[27]

Trivedi and Chitale[28] showed that when unmercerized and mercerized cotton yarns were hydrolyzed with 1*N* hydrochloric acid, the relation of fluidity to per cent strength retained was different in the two cases, the latter showing higher strength retention at the same fluidity. More recently, Colbran and Davidson[29,30] have compared hydrocelluloses from unmercerized and mercerized cotton with reference to loss of weight and change in copper number and carboxyl group content by treatments with 0.25*N* sodium hydroxide and 0.25*N* strontium hydroxide at 100°C. for 6 hr. They conclude from these results that hydrocellulose from mercerized cotton is less heterogeneous with respect to chain length.

The study reported here is an extension of the work of Trivedi and Chitale[28] on cotton yarns to fibers. The study was made in order that any complications due to changes in yarn structure produced by the swelling treatments and their possible effect on strength, may be avoided. The scope of the work has been enlarged to include other swelling agents besides sodium hydroxide.

Experimental

Egyptian cotton (karnak), in the form of combed slivers, scoured with 2% sodium carbonate, 1% sodium phosphate and 0.2% wetting agent [on the weight of the material, material to liquor (M:L) ratio 1:10] under a pressure of 20 psi for 4 hr. in a laboratory kier. Slivers were then washed with water. Approximately 60 g. of cotton fibers were treated in slack condition at 20°C. for 1 hr. with (*a*) 24% sodium hydroxide solution (*b*) 78% ethylenediamine (EDA), or (*c*) 70% zinc chloride solution. The swollen fibers were washed thoroughly to remove the swelling agent, complete removal being tested by appropriate methods.

All these samples as well as the untreated cotton were hydrolyzed with 1*N* hydrochloric acid at 35°C. for different periods ranging from 1 hr. to 15 days. These are referred to as treated samples. Some of the hydrocellulose samples from untreated cotton were treated with the swelling agents under conditions given earlier. The untreated and the three treated cellulose samples were also hydrolyzed under drastic conditions by refluxing with 2% hydrochloric acid solution in a boiling water bath for 14 hr., using a M:L ratio of 1:60. Loss in weight after drastic hydrolysis was 3.2, 9.3, 6.6, and 8.3% for the untreated and that treated with NaOH, EDA, and $ZnCl_2$, respectively.

Breaking strength of the various samples was measured at 0 and $^1/_8$ in. gauge length (G.L.) on a Stelometer. The breaking strength was calculated as gram/tex. Per cent loss in breaking strength was calculated on the

basis of the original strength of that sample as 100%. Strength determinations were not possible with the highly degraded samples. Fluidity of 0.5% solutions of the samples in cuprammonium hydroxide (15 ± 0.1 g./liter Cu and 200 ± 10 g./liter NH_3) was determined at 20°C. by the standard method, and intrinsic viscosity was calculated.[31] Degree of polymerization was obtained from intrinsic viscosity values according to the method recently published by Cumberbirch and Harland.[32]

Crystallinity and relative amounts of cellulose I and II were measured for selected samples. The crystallinity of different samples was determined by an x-ray diffraction technique based on the method of Wakelin et al.[33] This method was slightly modified to suit the present experimental conditions. The method was based on studying the radial intensity distribution scattered by (*a*) the sample, (*b*) a reference standard of crystalline cellulose I or II, and (*c*) a reference standard of amorphous cellulose. The crystalline standard of cellulose I was made available by courtesy of the Textile Research Institute, Princeton. The crystalline standard of cellulose II was obtained by repeated mercerization and hydrolysis of ramie fiber. The amorphous standard was obtained by pounding dry cellulose in a ball mill. Crystallinity was also obtained from accessibility measurements by iodine sorption according to the method of Hessler and Power.[34]

Results

Figure 1 shows the rates of hydrolysis of the different samples of cellulose. For the initial rapid hydrolysis and the subsequent slow hydrolysis, the rates given in Table I are obtained from these data.

TABLE I
Rates of Hydrolysis and Accessibility of Untreated and Treated Cotton Celluloses

	$K_{initial}$ × 10^2 hr.$^{-1}$	K_{slow} × 10^2 hr.$^{-1}$	Accessibility, % (iodine sorption)
Untreated	5.74	0.10	33
Treated with NaOH	8.88	0.15	62
Treated with EDA	7.20	0.14	51
Treated with $ZnCl_2$	6.57	0.11	45

The reactivity of native cotton cellulose has been increased considerably by all three swelling treatments, sodium hydroxide producing the largest and zinc chloride the smallest increase. Reactivity is almost linearly related to accessibility, as would be expected. The differences in the initial rates of hydrolysis persist during the later slow hydrolysis. The viscosity-average DP of the samples at the end of the rapid hydrolysis is: untreated,

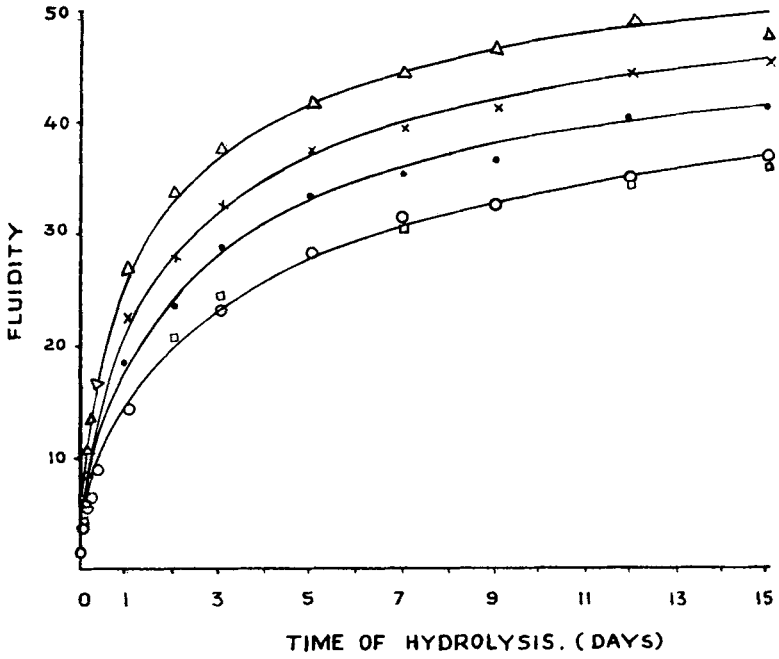

Fig. 1. Relation between fluidity and time of hydrolysis of untreated and treated cottons. (O) Untreated, hydrolyzed; (△) NaOH treated, hydrolyzed (×) EDA treated, hydrolyzed; (●) $ZnCl_2$ treated, hydrolyzed, (□) untreated, hydrolyzed, and NaOH treated.

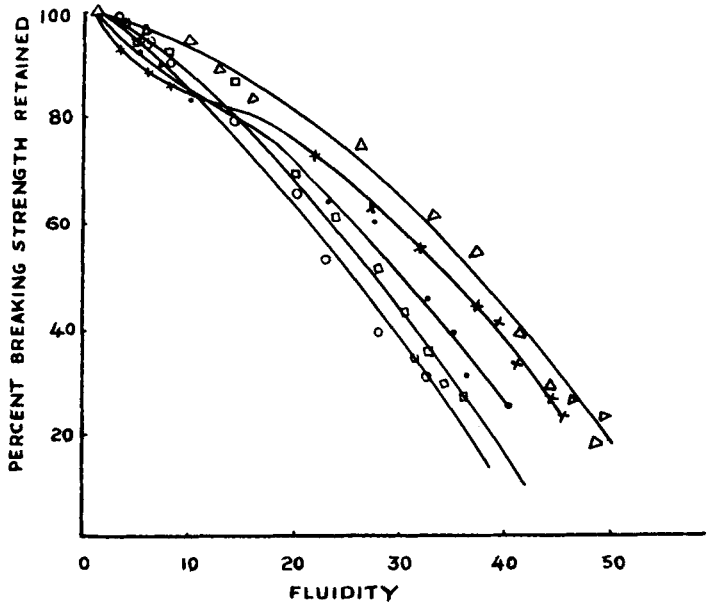

Fig. 2. Fluidity against per cent breaking strength retained at 0 in. G.L. (O) Untreated, hydrolyzed; (△) NaOH treated, hydrolyzed; (×) EDA treated, hydrolyzed; (●) $ZnCl_2$ treated, hydrolyzed; (□) untreated, hydrolyzed, and NaOH treated.

616; treated with NaOH, 247; treated with EDA, 335; treated with $ZnCl_2$, 448.

This is not LODP, and there is a steady decrease in DP during the further slow hydrolysis.

Figure 2 shows the relation between fluidity and per cent breaking strength retained after hydrolysis for various periods. All treated samples show a more favorable fluidity strength relation than untreated cotton. Mercerized cotton shows the highest strength retention after acid hydrolysis, at a given fluidity, followed by EDA-treated and zinc chloride-

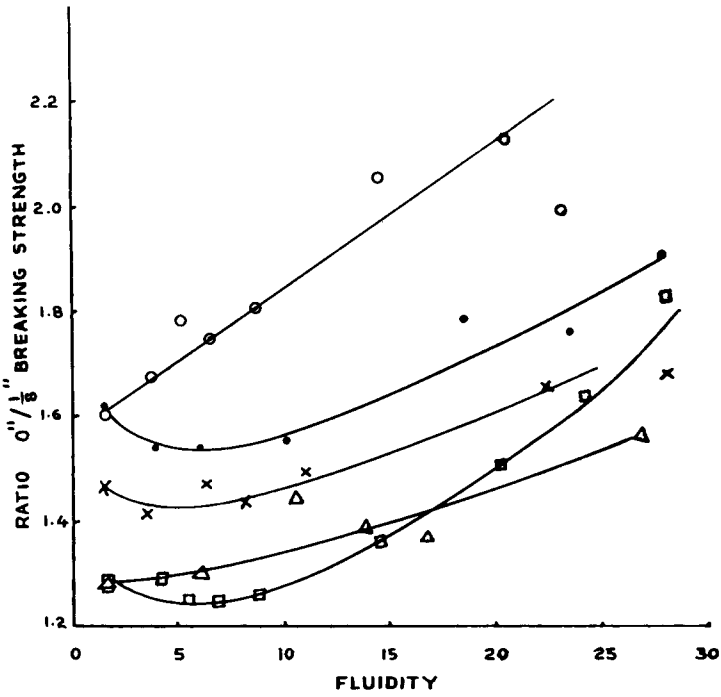

Fig. 3. Fluidity against ratio $0/^1/_8$ in. breaking strength. (O) untreated, hydrolyzed; (△) NaOH treated, hydrolyzed; (×) EDA treated, hydrolyzed; (●) $ZnCl_2$ treated, hydrolyzed; (□) untreated, hydrolyzed, and NaOH treated.

treated cottons. Improvement in strength retention follows the same order as increase in accessibility. Tripp et al.[35] have obtained a similar behavior on thermal degradation of cotton and viscose. Absolute strength of the fibers at 0 in. G.L. has been decreased, however, by all the swelling treatments as follows: untreated, 40.7; treated with NaOH, 36.1; treated with EDA, 40.4; and treated with $ZnCl_2$, 32.4

The fluidity of all these samples is less than 2, showing that no degradation of the chain molecules has occurred during the swelling treatments.

The ratio of breaking strength at 0 in. to that at $^1/_8$ in. G.L. has been plotted in Figure 3 against fluidity. With the idea of not complicating the

graph too much, results up to 30 fluidities have been plotted. It is seen that the samples treated with sodium hydroxide and ethylenediamine give lower values of this ratio initially as well as after acid hydrolysis. On the other hand, treatment with zinc chloride has not produced any change in the ratio for the unhydrolyzed sample though at all stages of hydrolysis, the value is lower than the corresponding one for the acid-hydrolyzed untreated cotton. Presence of weak places caused by structural reversals in the direction of fibril spirals in the cotton fiber has been shown by Wakeham and Spicer.[36] At the reversal, the cellulose is more highly oriented and probably more crystalline.[37] Due to the random presence of these weak

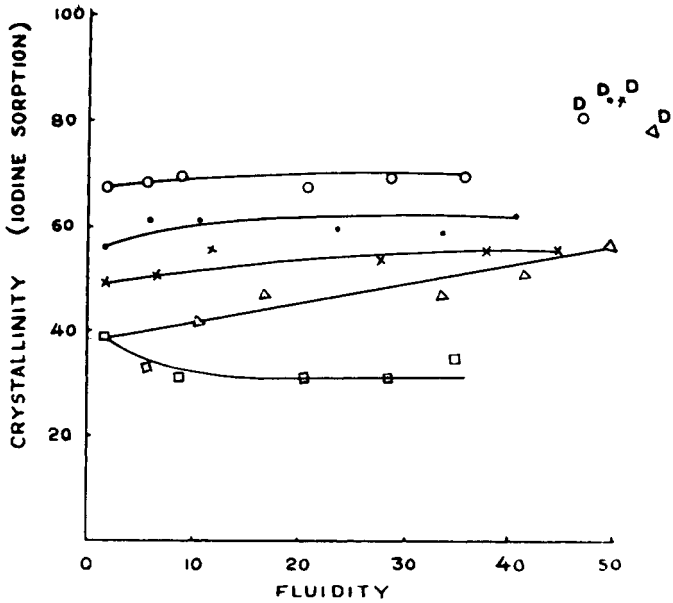

Fig. 4. Relation between fluidity and crystallinity (iodine sorption) of the acid-hydrolyzed samples. (O) Untreated, hydrolyzed; (△) NaOH treated, hydrolyzed; (×) EDA treated, hydrolyzed; (●) $ZnCl_2$ treated, hydrolyzed; (□) untreated, hydrolyzed, and NaOH treated; (D) samples after drastic hydrolysis.

places along the fiber length, fiber breaking strength is lower the greater the gage length. The ratio of 0 to $^1/_8$ in. gage length strengths is thus an index of uniformity of strength along the lengths of the fiber, lower values indicating greater uniformity. Mercerization improves uniformity of strength along the length of the fiber.[38] Present data show that ethylenediamine also produces a similar improvement though to a smaller extent. Swelling treatments with zinc chloride, however, do not improve strength uniformity. These differences are probably related to the mechanism of swelling which is different with the three agents. Sodium hydroxide penetrates into the crystalline regions and produces an irreversible change in the lattice structure from cellulose I to cellulose II. Ethylenediamine is also an

intracrystalline swelling agent but the change in lattice structure produced is reversed on treatments with polar oxygen-containing solvents. Zinc chloride is an intercrystalline swelling agent, does not penetrate into the crystallites, and consequently produces no change in the lattice structure.

Figure 3 shows another interesting feature. With each of the treated celluloses, there is a dip in the curve in the early stages of hydrolysis, indicating an increase in the strength uniformity of the fiber. Random acid hydrolysis of the fiber would be expected to decrease strength uniformity, as observed in the case of the untreated cellulose.

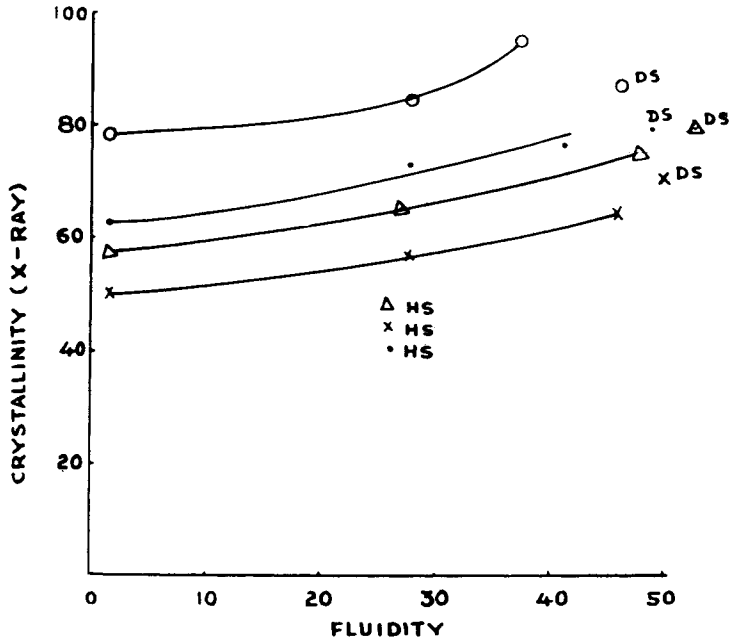

Fig. 5. Relation between fluidity and crystallinity (x-ray diffraction technique) of the acid-hydrolyzed samples. (O) Untreated, hydrolyzed; (△) NaOH treated, hydrolyzed; (×) EDA treated, hydrolyzed; (●) $ZnCl_2$ treated, hydrolyzed; (DS) samples after drastic hydrolysis; (HS) untreated, hydrolyzed, and treated with different swelling agents.

Changes in crystallinity produced by acid hydrolysis of the various samples have been determined by iodine sorption and by x-ray diffraction. These are shown respectively in Figures 4 and 5. The last point which is not joined in each curve represents the samples after drastic hydrolysis. Crystallinity of the initial unhydrolyzed cotton has been reduced by all the swelling treatments. However, only sodium hydroxide has produced a change in the lattice structure from cellulose I to cellulose II (Table II). Cotton treated with ethylenediamine and with zinc chloride shows only cellulose I structure. As zinc chloride does not penetrate into the crystallites, change in lattice structure of the cellulose would not be expected with a swelling agent of ths type. Ethylenediamine,

in the anhydrous condition, forms a complex with cellulose. This change, however, is reversed on treatment with polar, oxygen-containing solvents such as water. The treatment reported here is with ethylenediamine hydrate followed by washing in water. No change in lattice structure is therefore to be expected in this case, too. It is interesting to note that treatment with aqueous EDA has produced significant decrystallization under the conditions used.

TABLE II
Fluidity and Crystallinity of Cotton Cellulose Treated under Different Conditions

Treatment	Fluidity	DP	Crystallinity (x-ray method) Cell. I	Cell. II	Total
1. Untreated	1.5	8994	78	—	78
Hydrolyzed for 5 days	28.4	616	85	—	85
Hydrolyzed for 15 days	37.4	336	96	—	96
Drastic hydrolysis	46.2	177	88	—	88
2. Treated with NaOH	1.6	8550	7	50	57
Hydrolyzed for 24 hr.	27.0	676	7	59	66
Hydrolyzed for 15 days	48.1	146	10	66	76
Drastic hydrolysis	53.4	93	4	76	80
3. Treated with EDA	1.4	9226	50	—	50
Hydrolyzed for 2 days	28.0	639	57	—	57
Hydrolyzed for 15 days	45.9	181	65	—	65
Drastic hydrolysis	50.3	125	71	—	71
4. Treated with $ZnCl_2$	1.5	8994	62	—	62
Hydrolyzed for 3 days	28.1	626	73	—	73
Hydrolyzed for 15 days	41.5	254	77	—	77
Drastic hydrolysis	49.4	135	80	—	80
5. Hydrolyzed sample treated with					
(a) NaOH	24.4	800	—	50	50
(b) EDA	25.9	724	44	—	44
(c) $ZnCl_2$	25.7	733	36	—	36

Crystallinity measurements by the two methods show two outstanding differences. X-ray diffraction shows a lower crystallinity for the EDA-treated sample than for that treated with sodium hydroxide, while iodine sorption gives the opposite results. Also, iodine sorption does not show significant increases in crystallinity with progressive hydrolysis, except in the case of the sample treated with sodium hydroxide. Values of crystallinity obtained by this method for all the samples are lower. It has been reported that iodine sorption occurs not only in the accessible regions but also on the surface of crystallites.[39] This, together with the fact that different standards are used in the two methods, probably explains the dif-

ferences in values of crystallinity obtained. The contrary results for crystallinity from iodine sorption and x-ray diffraction obtained for the samples treated with sodium hydroxide and EDA, respectively, can be explained if one postulates that the crystallite dimensions are different in the two cases, favoring larger sorption of iodine on the crystallite surfaces for the former sample.

Treatment of hydrolyzed cotton with swelling agents gives some interesting results. This has been investigated more thoroughly with sodium hydroxide and results obtained for the hydrolyzed and subsequently mercerized cotton are shown in Figures 1 to 5. These data show that when hydrolyzed cellulose is treated with sodium hydroxide: (*a*) no significant change in cuprammonium fluidity is produced; (*b*) per cent breaking strength retained (as well as absolute strength) is increased significantly at any given fluidity; (*c*) there is a pronounced change in the ratio of 0 to $^1/_8$ in. gage length breaking strengths, markedly lower values being obtained after mercerization of the hydrolyzed samples; (*d*) a large decrease in crystallinity is obtained, the value of crystallinity being lower for the hydrolyzed, mercerized cellulose than the mercerized, hydrolyzed one at the same fluidity; (*e*) conversion of cellulose I to cellulose II is complete as judged from x-ray diffraction data (Table II). A similar result was obtained also with the other two swelling agents, and as low as 36% crystallinity was obtained by treating a hydrocellulose of about 25 fluidity with zinc chloride (cf. Fig. 5).

Treatment of acid-hydrolyzed cellulose with sodium hydroxide under conditions used here would remove, by solution, some of the low molecular weight fractions. This soluble fraction is probably too small to affect significantly the weight-average molecular weight as measured by fluidity. It can, however, explain the significant increase in strength obtained. Presence of a small amount of a low molecular weight fraction in a polymer is known to reduce its strength considerably.[40] Another probable explanation is the change in fine structure of the cellulose produced by the swelling treatment leading to considerably reduced crystallinity. This shift toward the viscose structure may also make breaking strength less dependent on the molecular chain length. The marked change in the ratio of 0 to $^1/_8$ in. gage length strengths produced by treatment of the acid-hydrolyzed cotton fibers with sodium hydroxide indicates that the weakness of cotton fibers due to structural reversals persists even after pronounced acid degradation

Summary

Treatment of cotton cellulose with swelling agents such as sodium hydroxide, ethylenediamine, and zinc chloride decreases crystallinity and increases reactivity of the cellulose. Increase in reactivity is approximately linearly related to the extent of decrystallization. Sodium hydroxide is the most effective and zinc chloride the least effective agent in producing these changes. Such treatments also improve the strength-fluidity relation of the cotton fibers so that for the same fluidity the per cent strength re-

tained is higher for the treated than for the untreated cotton. This improvement is due to the change in fine structure of cotton produced by the swelling treatments and follows the same order as the extent of decrystallization. Absolute strength of the fibers at 0 in. gage length has been reduced, however, by all the swelling treatments.

The ratio of fiber breaking strength at 0 and $^1/_8$ in. gage length is reduced by treatments with sodium hydroxide and ethylenediamine showing a greater strength uniformity along the length of the fiber due to removal of weak places caused by structural reversals. Again sodium hydroxide is more effective than ethylenediamine, while zinc chloride, an intercrystalline swelling agent produces no change at all in this ratio.

Significant differences are obtained in crystallinity values obtained by x-ray diffraction and by iodine sorption. Treatment of acid-hydrolyzed cotton cellulose with sodium hydroxide improves fiber strength considerably without a significant change in fluidity. This is also accompanied by marked decrystallization, the crystallinity obtained by swelling a hydrolyzed cellulose being much lower than that obtained by hydrolyzing a swollen cellulose at comparable fluidities.

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Synopsis

Acid hydrolyses of untreated cotton (scoured) and cottons treated in slack with three swelling agents were carried out for different periods. It was found that treatment with swelling agents decreases crystallinity and fiber strength and increases reactivity of cellulose. Such treatments also improve the strength-fluidity relation of the fibers. Swelling treatment with sodium hydroxide (NaOH) or ethylenediamine (EDA) improves strength uniformity along the fiber length. Both mild and drastic hydrolyses increase crystallinity of the untreated and the treated cottons. Crystallinity figures obtained by x-ray diffraction technique are different from those obtained by iodine sorption method. Treatment of acid-hydrolyzed cotton fibers with sodium hydroxide improves fiber strength considerably. When acid-hydrolyzed cotton is swollen with zinc chloride solution, crystallinity (x-ray) drops down to as low as 36%.

Résumé

Des hydrolyses acides de coton non-traité et des cotons traités en masse avec trois agents gonflants, ont été effectuées pendant différentes périodes. On a trouvé que le traitement avec des agents gonflants diminue la cristallinité et la force de la fibre et augmente la réactivité de la cellulose. De tels traitements améliorent aussi la relation fluidité–force des fibres. Le traitement de gonflement avec l'hydroxyde de sodium (NaOH) ou l'éthylène diamine (EDA) améliore l'uniformité de la force le long de toute la fibre. Des hydrolyses ménagées et drastiques augmentent la cristallinité des cotons traités et non-traités. Les figures de cristallinité obtenues par diffraction des rayons-X sont différentes de celles obtenues pa la méthode d'absorption d'iode. En traitant avec de l'hydroxyde de sodium les fibres de coton ayant subi l'hydrolyse acide, on améliore considérablement la force de la fibre. Lorsque le coton après une hydrolyse acide est gonflé avec une solution de chlorure de zinc, la cristallinité (rayons-X) tombe presqu'à 36%.

Zusammenfassung

Saure Hydrolysen von unbehandelter Baumwolle (gereinigt) und von Baumwolle, die im losen Zustand mit drei Quellungsmitteln verschieden lang behandelt wurde, wur-

den durchgeführt. Es wurde gefunden, dass die Behandlung mit Quellungsmitteln die Kristallinität und die Faserfestigkeit hertabsetzt und die Reaktivität der Cellulose steigert. Eine solche Behandlung verbessert auch die Festigkeits–Fluiditätsbeziehung der Fasern. Quellungsbehandlung mit Natriumhydroxyd (NaOH) oder Äthylendiamin (EDA) verbessert die Gleichmässigkeit der Festigkeit entlang der Faserlänge. Sowohl milde als auch starke Hydrolyse erhöht die Kristallinität von nichtbehandelter und vorbehandelter Baumwolle. Der durch Röntgenuntersuchungen erhaltene Betrag des kristallinen Anteils stimmt mit dem nach der Jodabsorptionsmethode erhaltenen nicht überein. Eine Behandlung der sauer hydrolysierten Baumwollefasern mit Natriumhydroxyd verbessert die Faserfestigkeit wesentlich. Bei Quellung von sauer hydrolysierter Baumwolle in Zinkchloridlösung fällt die Kristallinität (nach Röntgenuntersuchungen) bis auf 36% ab.

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