

Carboxyethylation of Cotton by Treatment with Acrylamide*

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Synopsis

New methods for the treatment of cotton with acrylamide have been studied which permit the preparation of modified fabrics with relatively high degrees of carboxyethyl substitution. Wet treatment with acrylamide and sodium hydroxide can be used to produce cottons bearing both carboxyethyl and carbamoylethyl ether substituents. Adjustment of reaction conditions controls the amount and ratio of these substituents. The effects of varying the concentrations of the reactants, the time and the temperature of reaction, and the solvent media employed have been determined. Some elucidation of the chemical mechanisms is provided. Previous work has shown that dry heat treatments of cotton impregnated with acrylamide and alkali can be used to produce high carbamoylethyl substitutions with little or no carboxyethyl substitution. The present work provides an extension of the cotton-acrylamide treatment whereby fabrics can be produced with only carboxyethyl or carbamoylethyl groups, or with mixtures of the two.

INTRODUCTION

The treatment of cotton with acrylamide has been investigated as a means of introducing carboxyethyl ether substituents along the cellulosic chains. It has been found that this amide can be used to prepare etherified cottons with carboxyethyl and carbamoylethyl groups, and that, by adjusting reaction conditions, the amount and ratio of the substituents can be controlled.

In earlier work at this laboratory, acrylamide was used with 2-5% sodium hydroxide to carbamoylethylate cotton.¹⁻³ A pad-bake technique yielded fabrics with high degrees of carbamoylethyl substitution and little or no carboxyethyl substitution. It was also shown that boiling carbamoylethylated fabric in alkali solution converted a few of the substituents to carboxyethyl groups, but the principal reaction was cleavage at the ether linkages.

Soluble, nontextile cellulosic products have been prepared by treatment of alkali cellulose, suspended in organic solvent, with acrylamide at 50-

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140°C.⁴ and by treatment of cellulose with an aqueous solution containing acrylamide and 10–40% of a strongly alkaline material at 0–40°C.⁵ The product of the former treatment is reported to be carboxyethyl cellulose, while that of the latter contained both carbamoylethyl and carboxyethyl groups. Touzinsky⁶ studied the distribution of substituent groups among the three reactive sites of the anhydroglucose unit in products from this type of treatment. He found that the ratio of carbamoylethyl to carboxyethyl groups was 1.6 when cellulose was treated with aqueous acrylamide at 4°C. in the presence of sodium hydroxide, and that the relative equilibrium constants for reaction at the three alcohol groups of cellulose were $K_2:K_3:K_6 = 9:1:19$ for this treatment.

Other methods for producing carboxyethylated cellulose include the hydrolysis of cyanoethylated cotton,⁷ the saponification of β -propiolactone-treated cotton,⁸ and the reaction of β -chloropropionic acid,⁹ or esters of acrylic acid with cellulose.¹⁰

In this paper are described several types of acrylamide treatments of cotton and the effect of variables on the course and extent of the reaction. Additional information on the mechanism of the reaction of acrylamide with cellulose and on the stability of the products is provided. One-step methods for the preparation of fibrous cotton textile materials with relatively high degrees of carboxyethylation are demonstrated.

MATERIALS, TESTS, AND ANALYSES

The fabric used was an 80 × 80 cotton print cloth, 3.2 oz./sq. yd., which had been desized, caustic-boiled, and bleached. Samples also were prepared from a 7/2 mercerized cotton yarn.

Acrylamide was used as received from commercial sources without additional purification. Other chemicals used were laboratory reagent grade.

Strength tests were carried out by standard procedures of the American Society for Testing Materials.¹¹

Nitrogen content was determined by the Kjeldahl method; carboxyl by a back-titration technique.¹² The degrees of substitution (D.S., the average number of substituent groups per anhydroglucose unit of the cellulose molecules) of treated samples were estimated from these analyses. The individual D.S. calculated is low if there are substantial amounts of each of the two substituent groups. However, the values reported here would be little affected by correction for the presence of the other group.

EXPERIMENTAL AND RESULTS

Pad-Bake Treatments

Samples of cotton fabric were impregnated with 5–30% acrylamide solution, dried, then padded with 12% sodium hydroxide solution, baked at 140°C. for 5 min., neutralized, washed, and dried. Chemical analyses of the treated samples indicated that all the samples had been carboxy-

TABLE I
Pad-Bake Treatment of Cotton with
Acrylamide-Sodium Hydroxide Solutions

Pad bath concentrations ^a		Product analyses				
Acrylamide, %	Sodium hydroxide, %	COOH, %	D.S. carboxyethyl	N, %	D.S. carbamoyl-ethyl	Ratio CE/CA ^b
7	5	0.29	0.01	0.56	0.06	0.17
7	10	0.42	0.02	0.44	0.05	0.40
5	12	0.56	0.02	0.25	0.03	0.67
10	12	0.59	0.02	0.53	0.06	0.33
7	20	0.96	0.04	0.15	0.02	2.00

^a Samples of cotton print cloth were immersed in solutions containing the indicated concentrations of acrylamide and sodium hydroxide, padded to remove excess (100–120% pickup), baked at 140°C. for 5 min., neutralized, washed, and dried.

^b Approximate ratio of the number of carboxyethyl groups to carbamoylethyl groups.

ethylated to about the same extent, D.S. 0.01–0.02, and that carbamoyl-ethyl substitution varied from D.S. 0.01–0.06 as a function of the acrylamide concentration used.

Application of acrylamide and sodium hydroxide together from a mixed pad bath followed by baking at 140°C. for 5 min., neutralizing, and washing produced the results shown in Table I. A higher degree of carboxyethylation was achieved in the last example of Table I, but the method was unsatisfactory for producing a relatively wide range of carboxyethyl substitutions. This technique was essentially that of Reeves and co-workers^{2,3} but with the utilization of pad baths with higher ratios of sodium hydroxide to acrylamide concentration.

Wet Treatments

The reaction of acrylamide and sodium hydroxide with cotton in the wet state was studied and found to yield fabrics with relatively high carboxyethyl substitutions and little or no carbamoylethyl substitution.

Pad baths containing acrylamide and sodium hydroxide were prepared by mixing together cold aqueous solutions of each reagent in amounts calculated to give the desired concentrations. The baths were used immediately to avoid concentration changes due to hydrolysis of the amide which takes place slowly in cold aqueous alkaline solution.

Fabric was immersed in the solution and the pickup adjusted to about 85% by passing through pad rolls. The fabric was then rolled on a glass tube, wrapped in impermeable plastic film, and allowed to react for the time and at the temperature noted. After the reaction period, the fabric was neutralized with dilute acid, thoroughly washed, and dried.

Acrylamide Concentration. The effect of changes in the concentration of acrylamide with 12% sodium hydroxide in the pad bath is shown in Figure 1. The degrees of etherification obtained after 1 hr. at 70°C. are

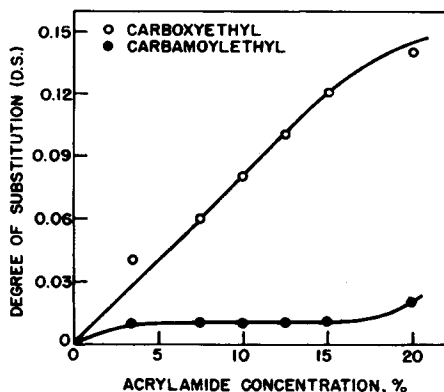


Fig. 1. Effect of acrylamide concentration in the wet treatment of cotton. Acrylamide-12% sodium hydroxide solution padded onto fabric and allowed to react at 70°C. for 1 hr.

plotted against the per cent acrylamide used. Carboxyethyl substitution increased almost linearly to about D.S. 0.14 at the solubility limit of acrylamide in the presence of this sodium hydroxide concentration. The degree of carbamoylethylation was constant at about D.S. 0.01, except for the treatment with 20% acrylamide which gave D.S. 0.02.

TABLE II
Effect of Reagent Concentrations upon the Wet Reaction of
Acrylamide-Sodium Hydroxide Solutions with Cotton

Pad bath concentrations ^a		Product analyses				
Acrylamide, %	Sodium hydroxide, %	COOH, %	D.S. carboxyethyl	N, %	D.S. carbamoylethyl	Ratio CE/CA ^b
7	5	0.98	0.04	0.10	0.01	4.00
7	7.5	0.95	0.04	0.07	0.01	4.00
7	10	0.84	0.03	0.00	—	—
7	12.5	1.41	0.05	0.00	—	—
7	15	1.98	0.07	0.00	—	—
7	20	1.96	0.07	0.00	—	—
14	5	1.09	0.04	0.26	0.03	1.33
14	7.5	1.53	0.06	0.10	0.01	6.00
14	10	2.05	0.08	0.07	0.01	8.00
14	12.5	2.64	0.10	0.01	—	—
14	15	2.94	0.11	0.00	—	—
14	20 ^c	—	—	—	—	—

^a Samples of cotton print cloth were immersed in solutions containing the indicated concentrations of acrylamide and sodium hydroxide, padded to remove excess (about 85% pickup), rolled on glass tubes, wrapped with plastic film, and allowed to react while wet for 1 hr. at 70°C., then neutralized, washed, and dried.

^b Approximate ratio of the number of carboxyethyl groups to carbamoylethyl groups.

^c Solution could not be used. Precipitation resulted upon mixing.

TABLE III
Effect of Time upon the Wet Reaction of Acrylamide-Sodium Hydroxide Solutions with Cotton

Time, min.	Product analyses				
	COOH, %	D.S. carboxyethyl	N, %	D.S. carbamoyl-ethyl	Ratio CE/CA ^a
14% Acrylamide-5% sodium hydroxide treatment ^b					
15	0.53	0.02	0.41	0.05	0.40
30	0.65	0.02	0.42	0.05	0.40
60	1.09	0.04	0.26	0.03	1.33
120	1.43	0.05	0.15	0.02	2.50
240	1.16	0.04	0.14	0.02	2.00
14% Acrylamide-10% sodium hydroxide treatment ^b					
15	1.66	0.06	0.45	0.05	1.20
30	1.68	0.06	0.34	0.04	1.50
60	2.05	0.08	0.07	0.01	8.00
120	1.99	0.07	0.03	—	—
240	1.94	0.07	0.00	—	—

^a Approximate ratio of the number of carboxyethyl groups to carbamoylethyl groups.

^b Samples of cotton print cloth were immersed in solutions containing these concentrations of acrylamide and sodium hydroxide, padded to remove excess (about 85% pickup), rolled on glass tubes, wrapped with plastic film, and allowed to react while wet at 70°C. for the indicated times, then neutralized, washed, and dried.

Sodium Hydroxide Concentration. In Table II are shown the results of wet treatment at 70°C. for 1 hr. of cotton impregnated with solutions of various sodium hydroxide concentrations together with 7% or 14% acrylamide. The degree of carboxyethylation increased with sodium hydroxide concentration while carbamoylethylation was practically nil with the higher concentrations.

The strength of the fabric was essentially unchanged by the treatment. For example, the sample of carboxyethyl D.S. 0.10, produced by treatment with 14% acrylamide-12.5% sodium hydroxide, had a breaking strength of 47.1 lb. as compared with 46.9 lb. for the untreated starting material.

Reaction Time. Treatment with 14% acrylamide-5% sodium hydroxide and with 14% acrylamide-10% sodium hydroxide at 70°C. was investigated for reaction periods of 15-240 min. The effect of time on the course of the reaction is demonstrated in Table III.

In the shorter treatment times lower ratios of carboxyethyl to carbamoylethyl substitution were obtained. Longer times yielded higher carboxyethyl substitutions and a higher ratio of acid to amide substituents. The total degree of substitution (the sum of carboxyethyl and carbamoylethyl groups) was not increased by longer reaction times. In fact, with the higher concentration of sodium hydroxide, the total degree of substitution decreased progressively with reaction time. Additional evidence of the in-

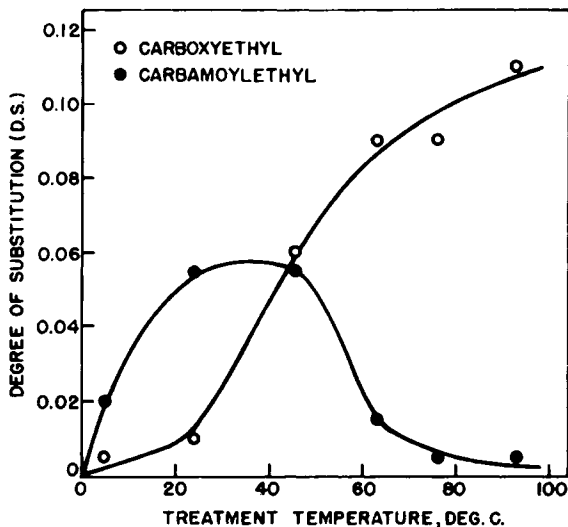


Fig. 2. Effect of reaction temperature in the wet treatment of cotton with 10% acrylamide-12% sodium hydroxide for 1 hr.

fluence of reagent concentrations on the composition of the products also can be seen from these data.

The carboxyethyl ether of cellulose may not be completely stable under alkaline conditions. In both sets of treatments reported in Table III, a peak substitution was reached at an intermediate reaction time. Loss of some of the carboxyethyl substitution as well as further amide hydrolysis may have resulted upon prolonged treatment.

As in the previous set, strength of the samples was little affected by the treatment. Strengths varied from 91% to slightly greater than that of the untreated.

Reaction Temperature. The results of wet treatment of cotton with 10% acrylamide-12% sodium hydroxide solution for 1 hr. at various temperatures ranging from 5 to 93°C. are shown in Figure 2. Below 46°C., carbamoylethyl groups predominate. At this temperature, however, the ratio of substituents reverses with a steady increase in the number of carboxyethyl groups as reaction temperature is raised and a rapid decrease in the number of carbamoylethyl substituents with temperature.

Treatment with Excess Solution

Another wet reaction method studied was the treatment of sodium hydroxide-impregnated cotton with excess solution of acrylamide. The liquor to cotton ratio in these treatments was more than 10:1, in contrast to the above described wet treatments in which this ratio was about 1:1.

Skins of mercerized yarn weighing about 8 g. were soaked in 25% sodium hydroxide solution, centrifuged to about 200% wet pickup, and treated with a solution of 5 g. of acrylamide in 80 ml. of water or isopropanol at 40-80°C. for 30 min. After treatment, samples were neutralized, washed, and dried. Results are given in Table IV.

TABLE IV
Treatment of Sodium Hydroxide-Impregnated
Cotton with Excess Acrylamide Solution

Reaction medium ^a	Temperature, °C.	Product analyses				
		COOH, %	D.S. carboxyethyl	N, %	D.S. carbamoyl-ethyl	Ratio CE/CA ^b
Water	40	0.06	<0.01	0.30	0.04	—
Isopropanol	40	0.89	0.03	0.20	0.02	1.50
Water	60	0.85	0.03	0.74	0.09	0.33
Isopropanol	60	3.82	0.15	0.16	0.02	7.50
Water	80	1.59	0.06	0.27	0.03	2.00
Isopropanol	80	4.06	0.16	0.06	0.01	16.00

^a Samples of mercerized cotton yarn (about 8 g. in weight) were immersed in 25% sodium hydroxide solution, centrifuged to about 200% pickup, treated with 5 g. of acrylamide in 80 ml. of water or of isopropanol at the temperature indicated for 30 min., neutralized, washed, and dried.

^b Approximate ratio of the number of carboxyethyl groups to carbamoylethyl groups.

As in the other type of wet reaction, higher temperatures favored higher carboxyethyl to carbamoylethyl ratios. However, the total degree of etherification was no higher at 80°C. than at 60°C. In fact, with water as the reaction medium higher total substitution was obtained at 60°C. indicating more extensive cleavage of carbamoylethyl groups at the higher temperature. Etherification was greater for reaction in isopropanol than in water at each temperature. The carboxyethyl D.S. of 0.16 was the highest achieved in this study.

Alkaline Hydrolysis of Carbamoylethylated Cotton

A sample of cotton was treated by the pad-bake method of Frick et al.² to produce a fabric with a carbamoylethyl D.S. of 0.22 (1.71% nitrogen) and a carboxyethyl D.S. of 0.01 (0.27% COOH). This fabric was divided into several portions which were subjected to hydrolysis by treatment with

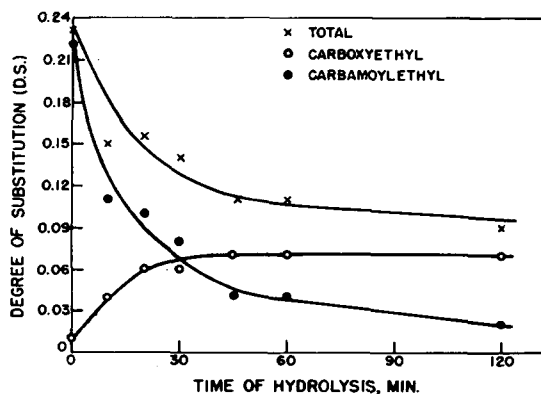


Fig. 3. Hydrolysis of carbamoylethylated cotton with 5% sodium hydroxide solution at 80°C.

5% aqueous sodium hydroxide solution at 80°C. for various times to 2 hr. Changes in the degree of substitution of carbamoylethyl and carboxyethyl groups as the hydrolysis proceeded are shown in Figure 3. The fall in the total degree of substitution is indicated in the figure also.

As in previously reported alkaline hydrolysis of carbamoylethylated cotton,² more groups were lost through cleavage of the ether linkages than were hydrolyzed to the carboxyethyl ether. Even short hydrolysis times result in drastic loss of substituents. The carboxyethyl groups formed, however, appear to be stable under these hydrolytic conditions.

DISCUSSION

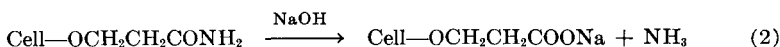
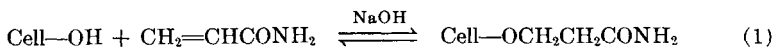
It has been established previously that a pad-bake process can be used to carbamoylethylate cotton by treatment of fabric with acrylamide and sodium hydroxide. Efficient reaction was obtained employing relatively low concentrations (2-5%) of sodium hydroxide to produce carbamoylethylation without any significant degree of carboxyethylation.

Modification of the pad-bake technique appears to offer little promise for carboxyethylating cotton although use of higher concentrations of sodium hydroxide did increase the amount of this ether substituent in the modified fabric. However, the degree of substitution achieved was relatively low.

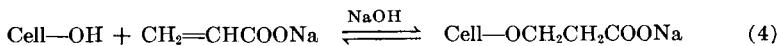
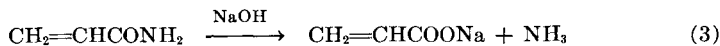
Wet treatments can be used to effect higher carboxyethyl substitutions. Two such treatments were developed. In one, cotton fabric is padded with a solution containing acrylamide and sodium hydroxide, and allowed to react while wet. By wrapping the impregnated fabric with impermeable plastic film, elevated temperatures and prolonged times could be employed in the treatment without drying the fabric. In the second type of wet treatment, sodium hydroxide-impregnated cotton is immersed in at least ten times its weight of acrylamide solution.

The effect of variables in these wet treatments has been studied, and generalizations of their influence on the course of the reactions can be made.

It appears that carbamoylethylation takes place initially through conjugate addition and that hydrolysis of the amide groups under the influence of alkaline catalysis proceeds with the liberation of ammonia to produce carboxyethyl groups in the form of the sodium salt. The two steps of the reaction may be represented by eqs. (1) and (2):

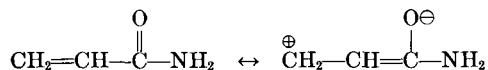


Carboxyethyl groups result almost exclusively from hydrolysis of the carbamoylethyl groups rather than from etherification of cotton with sodium acrylate, the hydrolysis product of acrylamide. This latter route would be represented as shown in eqs. (3) and (4).

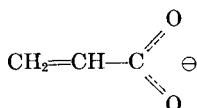


Sodium acrylate has been shown to be a poor reagent for the etherification of cellulose,^{1,10} and this was substantiated in a control experiment under the wet reaction conditions.

The reactivity of acrylamide in conjugate additions is attributed to resonance which places a partial positive charge on the β -carbon:

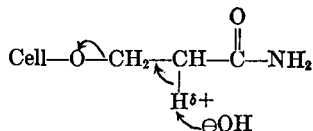


However, in the case of sodium acrylate there is a negative charge delocalized over the three-atom system of the carboxylate anion:



which inhibits this type of resonance. The magnitude of the partial positive charge at the β -carbon is greatly reduced and conjugate addition becomes unlikely.

The reversal of the addition reaction apparently occurs by abstraction of the acidic α -hydrogen and depends upon the acidity of this hydrogen:



The carbonyl carbon of the amide function is electron deficient and therefore withdraws electrons from the α -carbon making the hydrogens on this carbon partially positive, i.e., they become acidic. However, in the acrylate anion the carbonyl carbon is much less electron-deficient and hence, is much less efficient in activating the α -hydrogen.

Thus, in the reaction of acrylamide with cellulose under alkaline conditions, the amount of each of the two ether substituents depends upon the addition of acrylamide and the extent of hydrolysis or cleavage of the carbamoylethyl groups.

In wet treatment, the higher concentrations of acrylamide produce the greatest degree of reaction. For a given concentration of acrylamide, higher total degrees of reaction were obtained with higher sodium hydroxide concentrations. Likewise, the higher sodium hydroxide concentrations resulted in greater conversion of the substituent to the carboxyethyl group. Practical limitations are imposed by compatibility of the two reagents.

Both longer times and higher temperatures in wet treatment favor carboxyethylation. It appears that at the lower temperatures, although carbamoylethylation proceeds fairly readily, hydrolysis of the amide groups must be relatively slow. As the reaction temperature is raised, the rate of carbamoylethylation is increased, but the hydrolysis of the amide group is increased to a much greater extent resulting in an overall marked increase in carboxyethylation.

Long treatment times result in carboxyethylation because of the greater stability of this group to alkaline hydrolysis. Carbamoylethyl groups introduced in the treatment can be hydrolyzed to carboxyethyl groups or are cleaved in prolonged reaction.

The inefficient conversion of carbamoylethylated cotton to carboxyethylated cotton by hydrolysis with hot alkali solution also can be explained by the difference in stability of the two ether groups. Carbamoylethyl groups cleaved by reversal of the addition reaction are lost while those hydrolyzed to carboxyethyl are relatively stable. Under the hydrolytic conditions studied, about twice as many groups were cleaved as were hydrolyzed to carboxyethyl.

In treatment of sodium hydroxide-impregnated cotton with excess acrylamide solution, high degrees of reaction were achieved. Use of isopropanol as the reaction medium produced much higher degrees of carboxyethylation than a completely aqueous system.

Strength of modified cottons produced in this study were essentially the same as that of the untreated controls. Properties of carboxyethylated cotton are generally similar to those of carboxymethylated cotton which have been extensively reported.¹³

SUMMARY

Methods for introducing carboxyethyl groups into cotton textile materials by treatment with acrylamide have been developed. Wet treatments of cotton with this amide and sodium hydroxide can yield both carboxyethyl and carbamoylethyl groups, or under controlled conditions only carboxyethyl groups.

A pad-bake process previously has been reported for carbamoylethylation of cotton with acrylamide. Now, by proper selection of treatment conditions this reagent can be used to produce carbamoylethylated or carboxyethylated cotton, or products with combinations of these two ether substituents.

A mechanism for reaction in the wet treatment is proposed. Acrylamide reacts with the cellulose through conjugate addition to produce carbamoylethyl groups. Under the alkaline conditions, these groups can be cleaved by reversal of this reaction or hydrolyzed to carboxyethyl groups.

Because of difference in the stabilities of the two ether groups, the wet treatments favor production of carboxyethylated cotton with survival of few carbamoylethyl groups. Conditions yielding various distributions of the substituents have been determined. In general, use of higher sodium hydroxide concentrations, longer times, and higher temperatures result in greater proportions of carboxyethyl groups.

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Résumé

De nouvelles méthodes pour le traitement du coton par l'acrylamide ont été étudiées en vue de permettre la préparation de tissus modifiés avec des degrés relativement élevés de substitution carboxylée. Le traitement humide avec l'acrylamide et la soude caustique peut être utilisé pour obtenir des cotons portant des substituants à la fois carboxyéthylés et carbomyléthylés. Adjustant les conditions de réaction, on peut contrôler la quantité et le rapports de ces substituants. Les effets des variations de concentrations en réactifs, du temps et de la température de réaction, et des solvants utilisés comme milieu de réaction, ont été déterminés. Certaines explications du mécanisme chimique sont également fournies. Un travail antérieur avait montré que le traitement thermique à sec imprégné d'acrylamide et d'alcali pouvait être utilisé en vue de produire des substitutions carbamoyléthylées élevées avec peu ou pas de substitutions carboxyéthylées. Le présent travail permet une extension de ce traitement du coton par l'acrylamide grâce auquel un tissu peut être obtenu qui ne contient que des groupes carboxyéthylés ou carbomoyléthylés, ou le mélange des deux.

Zusammenfassung

Neue Methoden zur Behandlung von Baumwolle mit Acrylamid wurden untersucht, welche die Darstellung von modifizierten Geweben mit relativ hohem Carboxyäthylsubstitutionsgrad erlauben. Eine nasse Behandlung mit Acrylamid und Natriumhydroxyd kann zur Herstellung von Baumwolle mit Carboxyäthyl- und Carbamoyläthyläthersubstituenten verwendet werden. Durch Anpassung der Reaktionsbedingungen kann die Menge und das Verhältnis dieser Substituenten kontrolliert werden. Der Einfluss der Variation der Konzentration der Reaktionsteilnehmer, der Reaktionsdauer und -temperatur, sowie der verwendeten Lösungsmittel wurde bestimmt. Eine gewisse Arbeiten haben gezeigt, dass die trockene Erhitzung von acrylamid- und alkali-imprägnierter Baumwolle zur Herstellung einer hochgradigen Carbamoyläthylsubstitution mit wenig oder ohne Carboxyäthylsubstitution verwendet werden kann. In der vorliegenden Arbeit wird die Baumwollacrylamidbehandlung erweitert, so dass Gewebe mit blossen Carboxymethyl- oder Carbamoyläthylgruppen oder mit Mischungen dieser beiden hergestellt werden können.

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