International Journal of Research
Available at https://edupediapublications.org/journals
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

Acetone-Formaldehyde Resins Used For Improvements In Crease Resistance of Cellulose

Ph.D. Khasanova Saodat Khaitovna
Tashkent Institute of textile and light industry, Republic of Uzbekistan
Doctor of science Nabiyeva Iroda Abdusamatovna
Tashkent Institute of textile and light industry, Republic of Uzbekistan

Abstract. *In this paper it is shown that a new and innovative composition of AF-resins improves drastically on the mechanical properties of cellulose. By using this new type of resin a much smaller strength decrease was observed in combination with a large improvement in crease resistance. In addition, it is shown that the fabrics treated with this resin show a much higher resistance against dissolution in Cadoxen and do not swell so intensively as is the case for the same fabric treated with established crease resistance resins (such as Carbamol SEM). Finally, also a solution is proposed for the problem that fabrics treated with AF-resins show a yellowish colour.*

Keywords: Acetone-formaldehyde resin, crease resist finishing, carboxylic acids, carbamol SEM, ester functions, cellulose.

Introduction

Object research In the past few years' improvement in the ecological and hygienic properties of crease resist finishing processes, without an important loss of shrinkage behavior, was observed. These improvements were obtained by modification of the cellulose structure with di- and poly functional chemical compounds with limited or no formation and use of formaldehyde [1-3]. Also in the search for more permanent press finish procedures of cotton fabrics traditionally used formaldehyde based reagents are replaced by other, more environment friendly compounds, such as polycarboxylic acids [1-3]. However, these carboxylic acids affect largely the mechanical properties of cotton, with sometimes a decrease of 50% in strength. Therefore, one of the aims of the investigation reported in this paper is to find other substituent's that also are not based on formaldehyde and result into a modified cotton structure without loosing its beneficial mechanical properties. Preliminary reports were found with use of citric acid [4], Taconic acid [5], 1, 2, 3-propanetriearboxylic acid and but an tetra carboxylic acid [6-7], which gave promising but not yet satisfactory results.

American scientists made use of so called acetone-formaldehyde resin (AF-Resin), but it was not a breakthrough solution because the resin itself colored the fabric into a yellowish product, which is unacceptable [8-10].

In this paper an alternative approach will be shown with other compounds to obtain a better crease resist cotton fabric treatment without or with limited loss of its mechanical properties, such as strength and shrinkage and a solution for the colour problem will be proposed.

Analysis. Polyvinylalcohol (PVA), epichlorhydrine, acrylamide, glycerin, polyacrylonitrile, polyethylene glycol, glucose, maleic acid, caustic soda, potassium hydrogen fosphate and sodium hypophosphite were obtained from Sigma-Aldrich, while Carbamol SEM from Russia, AF-resin and Cadoxen were obtained from Fergana Furan Factory in Uzbekistan. Cotton textile structures were home prepared; the cotton raw material was obtained from local Uzbekistan factories. Impregnation of the cotton structure (142 g m"^{2}) was done according to the following procedure: impregnation of the cotton sample in the solution at pH= 9-10, followed by squeezing in a padder to wet pick up of 80+1%, drying of the sample at 100-110 °C and finally a curing treatment at 120-140 °C K about 5-10 minutes.

Samples were cut and mixed with KBr. IR-spectrometry was performed with a system 1000 of Perkin Elmer in a frequency range of 3800-400 cm'^{1}.

III. 1 Composition of the impregnating solution

In Uzbekistan textile industry Carbamol SEM is a commonly used impregnating resin against crease and shrinkage of cellulose. A drawback of this resin is the conditions of usability, namely a pH of 5-6. At such pH values the cellulose strength decreases significantly and in combination with the treatment of Carbamol SEM this decrease is about 50%. AF- resins show a much weaker decrease, therefore this type of resin was used as an alternative. However, it was found that this type of resin has a large amount of free formaldehyde (about 20%), which is unacceptable from environmental and human exposure point of view. Alternatively, formaldehyde free AF-resins [8-10] were used but it was also found that the efficiency of these resins is rather small and they are yellow colored, which means that these resins have an unacceptable influence on the colour of the impregnated cotton fabric.

In this paper a new type of impregnating solution, based on an AF-resin, is described. The composition of this solution is given in Table I.

Table I : Composition of the impregnating solution

Compound	Concentration
AF-resin	80 g L'^{1}
K_2HPO_4	12.5 gL'^{1}
Poly (Vinyl Alcohol)	10 gU^{1}
Maleic Acid	25 g L'^{1}
NaOH	Until pH = 9-10 is obtained

First the optimal concentration of AF-resin (structure is given in Fig. 1) was determined as a function of wrinkle recovery angle (WRA) (Fig. 2).

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

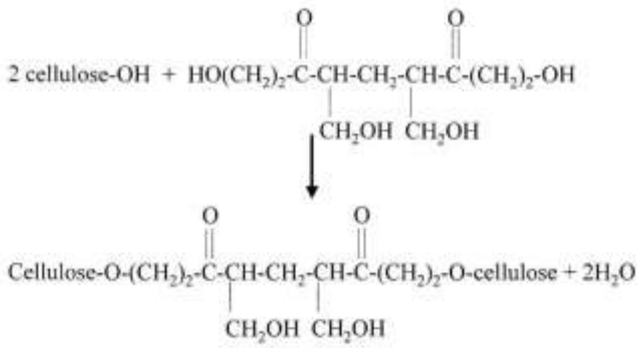


Fig. 1 : Shematic representation of the reaction between cellulose and resin to obtain a modified cellulose structure.

It can be seen that a maximum value of the WRA is obtained using a concentration of 80 g $L^{'1}$ and this value is much higher (135°) compared to a similar experiment using an equivalent concentration of Carbamol SEM (95°). It should be noted that all experiments using AF-resin are performed at a pH of 9-10, established by adding caustic soda (NaOH). In this pH range AF-resins have the most optimal values towards crease-resistance and shrinkage properties (investigated by a preliminary experiment) while cellulose strength retention is maximal in this pH range. However, the AF-resin efficiency seems limited and its use results in yellowish coloured structures.

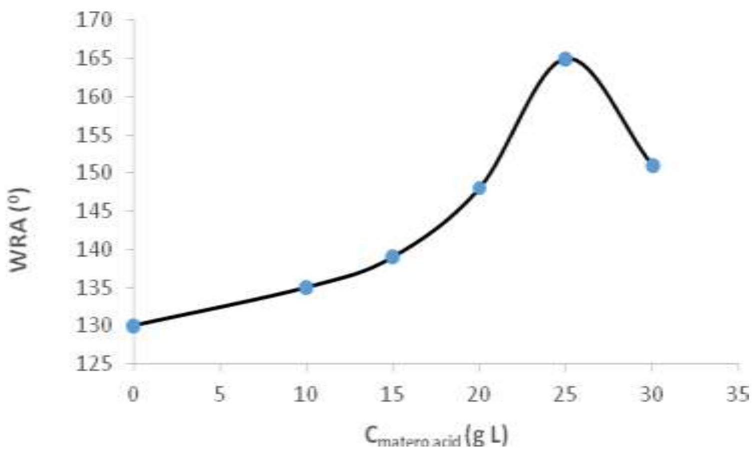


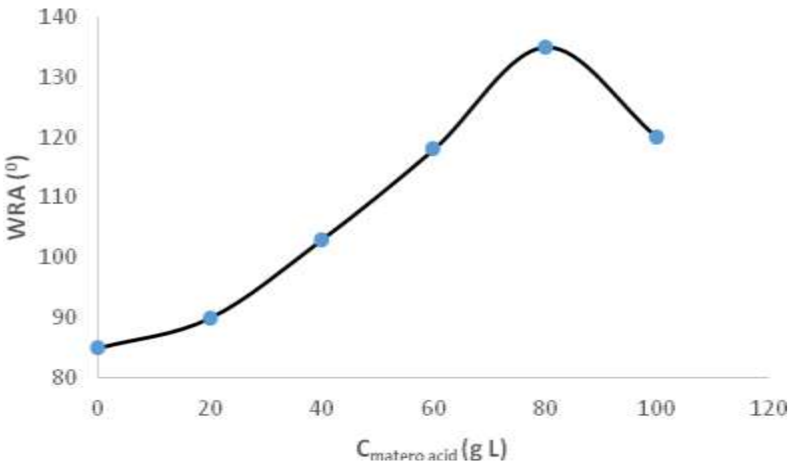
Fig. 2 : Variation of the WRA as a function of AF-resin concentration in the solution For treatment of cotton fabric. Other compound concentrations are as given in Table I except for maleic acid, which is absent.

The yellow colour could be eliminated by adding about 10 g $L^{'1}$ of polyvinyl alcohol (PVA). Since it was found that this compound did not chemically react with the cellulose and/or AF-resin, its presence was not further investigated. Probably PVA forms a complex or an aggregate with the functional group that is responsible for the yellow colour. This interaction results, for the used AF-resin, in a shift of the wavelength of absorption outside the range of visible light (400-700 nm). It is also found in literature [1-7] that compounds having a relatively small molecular weight and a polarizable group such as hydroxyl or carboxylic acid contribute

International Journal of Research
Available at https://edupediapublications.org/journals
p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

positively to the crease-resist properties of the impregnating solution. Therefore, compounds such as epichlorohydrin, acrylamide, glycerin, polyacrylonitrile, poly(ethyleneglycol), glucose and maleic acid (MA) were added to the solution and the crease-resistance properties of the impregnated cellulose fabrics investigated. It was found that the carboxylic acid functi onal group is deprotonated in a pH range of 9-10. In deprotonated form it is able to attack cellulose and to form an ester bond a hydroxyl group of cellulose. Fig. 3 shows clearly that an optimal concentration of maleic acid of 25 g L$^{'1}$ should be used to obtain a maximum of WRA of 165°, a value remarkably higher than 95° for Carbamol SEM and 135° for AF-resin without maleic acid.

Fig. 3 : Variation of the W^{r}RA as a function of maleic acid concentration and at optimal concentrations of the other compounds (see Table I) for treatment of cotton fabric



Finally a catalyst is added in order to increase the reaction rate and to make the technology economically feasible and interesting. Initially, Na_2HPO_2 was used as described in literature but no satisfactory results were obtained. In contrast, K_2HPO_4 resulted in the predicted acceleration of the reaction rate between cellulose, maleic acid and AF-resin. Additionally, it was also observed that the presence of K_2HPO_4 improved the value of WRA to a maximum of 220°, obtained at a catalyst concentration of 12.5 g L^{-1} in the presence of optimal concentrations of the other compounds (see Table II).

Table II : Mechanical properties of the fibers after treatment with crease-resist solutions

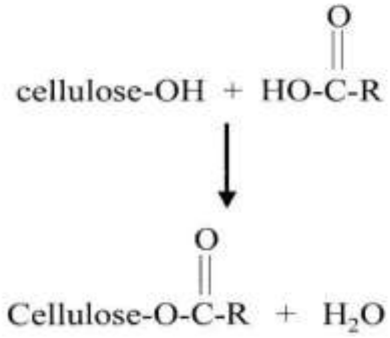
Type of solution	WRA (°)	Reduction of tear strength (%)		Reduction of elongation (%)		Fraction of free formaldehyde (%)
		warp	weft	warp	weft	
Carbamol SEM	95±5	29.6±1.2	34.0±1.4	4.9±0.5	30.0±0.9	0.40±0.03
AF-resin + maleic acid	220±5	15.0±0.8	21.9±1.1	3.8±0.4	23.1±0.9	0.06±0.02

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

III. 2 Chemical characterisation of impregnated cellulose

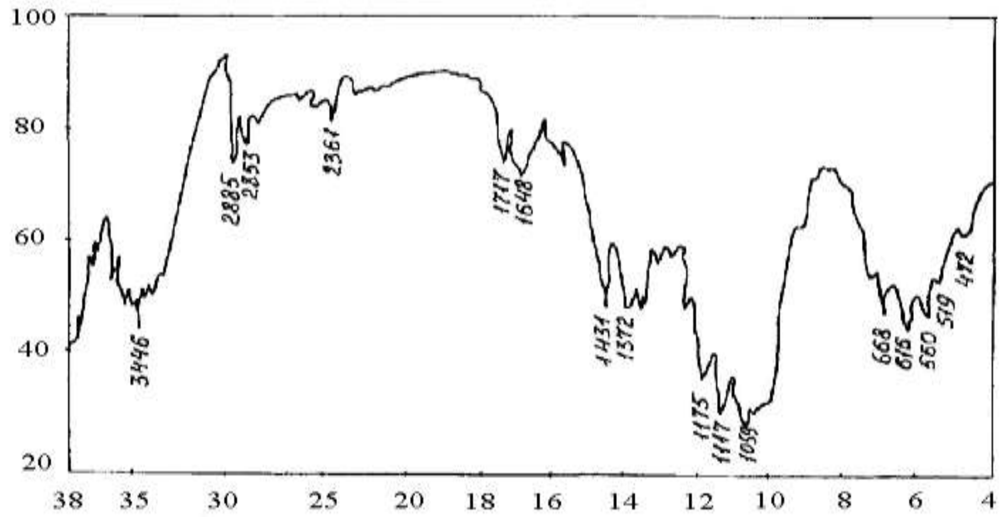
As shown in Fig. 1 it is expected that AF-resin reacts with cellulose through their hydroxyl functions resulting into an ether type bonding and formation of water. For the reaction between cellulose and maleic acid it is expected that an ester-bond is formed as shown in Fig. 4.

Fig. 4 : Reaction scheme of ester formation between cellulose and maleic acid



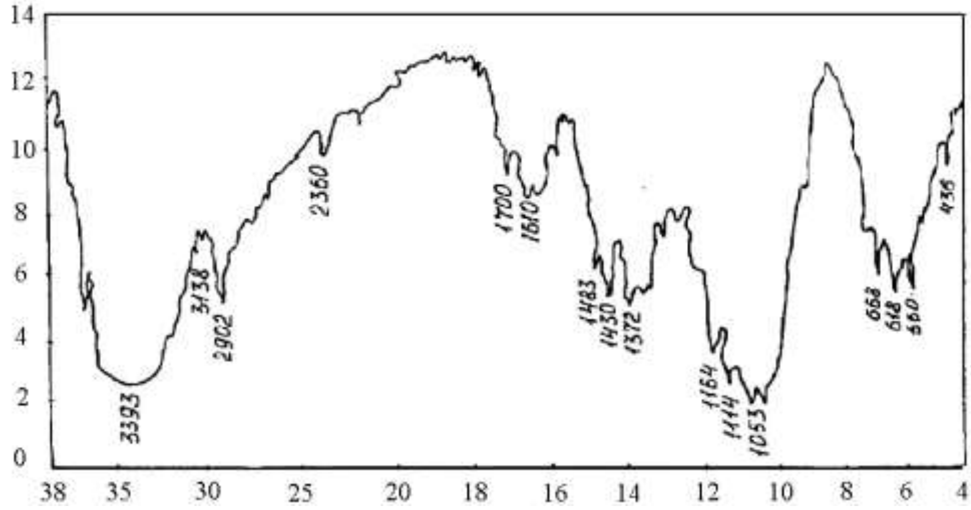
These hypotheses were confirmed by IR-analysis. In Fig. 5 IR-curves are shown of a cellulose sample before (Fig. 5a) and after treatment (Fig. 5b) with a solution of AF-resin containing maleic acid.

Fig. 5 : IR-spectra of the untreated (a) and the treated (b) cotton fabric with the developed AF- resin.



a)

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017



b)

The absorption band around 2902 cm$^{-'}$ shows clearly the symmetrical stretching of ether functions, while the shift of the absorption band at 1483 cm'^{1} (Fig. 5a) to 1430 cm'^{1} (Fig. 5b) shows the appearance of the deprotonated carboxyl groups of maleic acid and formation of ester functions. Also the appearance of the absorption band at 1610 cm"^{1} indicates that maleic acid reacts to form ester functions.

III. ***3. Properties of the treated cotton fabrics***

Table II summarizes a large range of experiments and shows the comparison of cotton fabrics treated with the commonly used solution, containing Carbamol SEM and the newly developed crease-resist treatment solution (Table I). A main improvement is the huge increase in WRA from about 90 to 220°. But also mechanical properties such as tear strength and elongation have improved in warp as well as weft direction. Finally, the quantity of free formaldehyde found after treatment (and formed as a side-product of this treatment) has been reduced remarkably, which is an important improvement in contributing to health issues.

Table III shows the resistivity of the fibers against treatment in solvent media, such as a Cadoxen solution. It can be seen from Table III that fibers treated with Carbamol SEM and AF-resin do not really differ from each other in dissolving properties, the only main difference is that the AF-resin treated fibers do swell much less than those treated with Carbamol SEM.

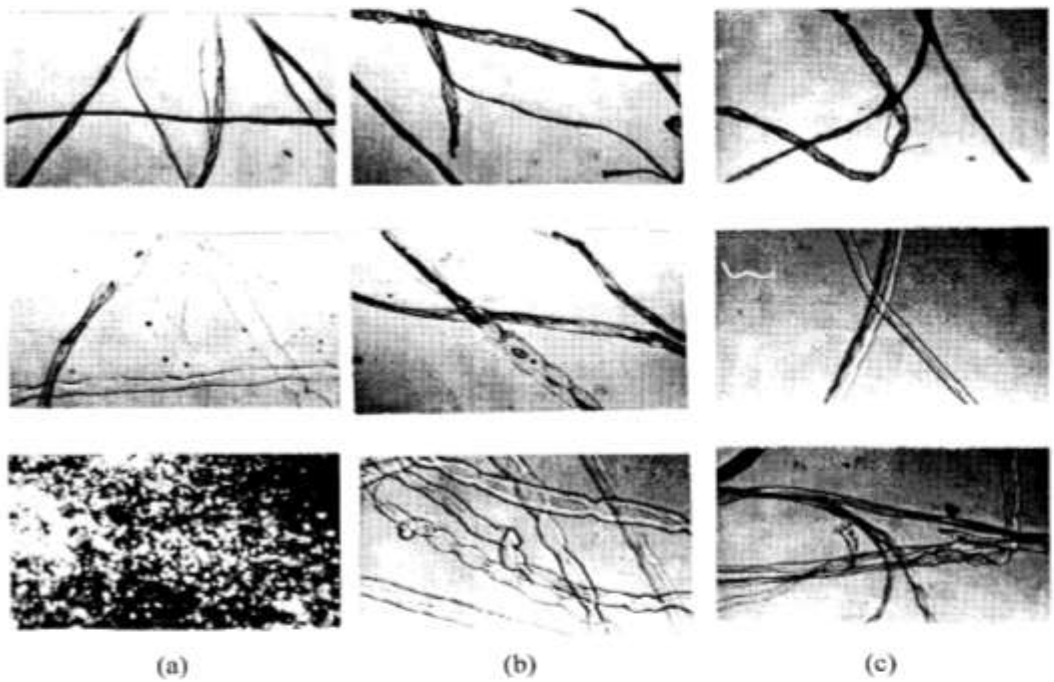
Table III : Properties of cotton fibers treated with crease-resist solutions and exposed to aggressive media.

	Model	Fiber size (|im)				Swelling (%)	Solubility after 20 hours in Cadoxen
		Before treatment		10 minutes treatment in Cadoxen			
		min	max	min	Max		
1	Untreated sample	7.0	30.5	23.1	69.3	166.5	high
2	Treated with Carbamol SEM	3.8	38.5	30.8	77.0	158	None
3	Treated with AF-Resin + maleic acid	3.8	30.5	15.4	77.1	89.2	None

Finally, Fig. 6 shows photographs of cotton fibres immersed in Cadoxen without treatment (a) and with treatment in Carbamol SEM (b) and with the new developed AF-resin (c).

Fig. 6: Pictures of cotton fibers taken after treatment in Cadoxen.

The cotton fibres were untreated (a), treated with Carbamol SEM (b) and treated with the developed AF-resin (c). Duration of Cadoxen treatment : top pictures is initial situation, middle pictures is after 10 minutes; below pictures is after 20 hours.



It can b e seen clearly that without treatment the cotton dissolves completely, which is not the case for the treated fibers. However the structure of the fibers treated with AF-resin is much better (even after 20 hours) than those treated with

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

Carbamol SEM. It is very clear that in (b) the fibers swell much more than those shown in Fig. 6c.

Conclusion

It can be concluded that a resin is developed for treatment of cellulose and that provides improved properties of the fabric such as strength retention, crease resitance, resistance against swelling and being dissolved.

REFERENCES

[1] C.M. Welch, Text. Res. J., 58 (1988) 480

[2] C. M. Welch, B. A. K. Andrews, US Patent 4820307 (1989)

[3] W. L. Xu, Y. Li, Text. Res. J,, 70 (2000) 588

[4] B. A. Andrews, D. P. Kottes, Text. Chem. Color. 29 (1997) 1

[5] H. M. Cloi, Text. Res. J., 62 (1992) 614

[6] C. Q. Yang, X. Wang, I. S. Kang, Text. Res. J. 67 (1997) 334

[7] C. Q. Yang, L. S. Xulan, Y. Jiang, Text. Res. J. 68 (1998) 457

[8] K. Zviskaya, US Patent 2159875 (1939)

[9] D. M. Gagarin, US Patent 2641591 (1953)

[10] V. S. Miller, US Patent 2711971 (1955)

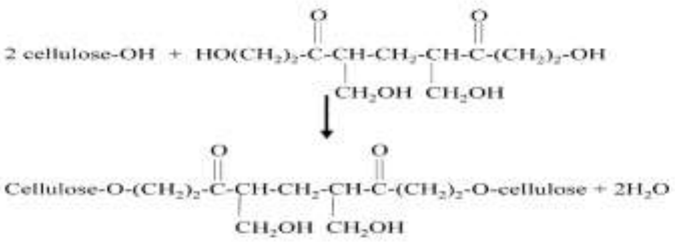


Fig. 1

p-ISSN: 2348-6848
e-ISSN: 2348-795X
Volume 04 Issue 02
February 2017

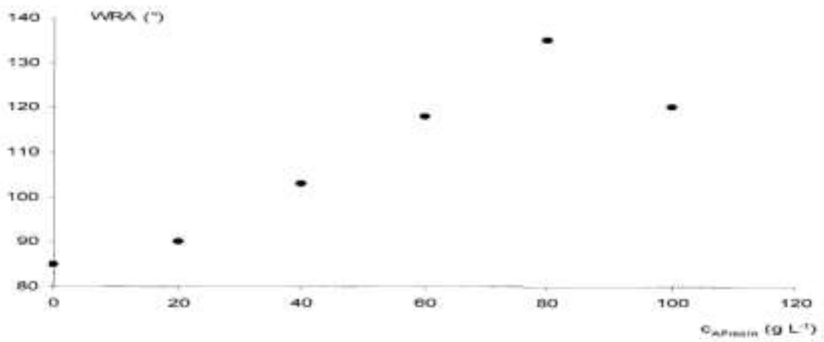


Fig. 2

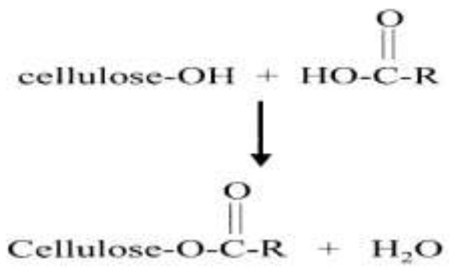


Fig. 4

	Model	Fiber size (|im)				Swelling (%)	Solubility after 20 hours in Cadoxen
		Before treatment		10 minutes treatment in Cadoxen			
		min	max	min	Max		
1	Untreated sample	7.0	30.5	23.1	69.3	166.5	high
2	Treated with Carbamol SEM	3.8	38.5	30.8	77.0	158	None
3	Treated with AF-Resin + maleic acid	3.8	30.5	15.4	77.1	89.2	None

Table III