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A Possibility for Shrinkage Decrease of Textile Fabrics Made from Cotton and Viscose Fibres

Abstract
This article presents experimental data concerning variation of the physico-mechanical properties of cotton and viscose fabrics cross-linked with a modified product on the basis of dimethyloldihydroxy ethylenecarbamide in the presence of different catalysts. The basic aim is to decrease the shrinkage of the fabric, which is why relatively low concentrations of the cross-linking agent are used. The results obtained are of definite interest, verifying the high catalytic activity of the mixed catalyst $MgCl_2$ - citric acid - tartaric acid.

Key words: *cross-linking agents, shrinkage finish, crease-resist finish, cross-linking catalysts, cotton, viscose.*

Introduction

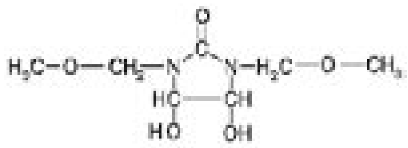
The textile materials are usually under significant lengthwise stress during the refinement processes. This results in considerable shrinkage during subsequent use because of the effect of the corresponding relaxation forces. Mechanical and chemical interventions provide possibilities for stabilising the dimensions of the cellulose textile materials. The chemical method leading to cross-linking of the cellulose macromolecules through the introduction of different products is one of the most widely used. In this way, the current state of the fabric is fixed and subsequent shrinkage is hindered. The cross-linking is usually achieved by the application of chemical compounds which are applied in the crease-resist finish of the fabric. The treatment proceeds in the presence of lower concentrations of the additive when shrinkage alone, and not creasing decrease, is required. It is known that a number of parallel reactions leading to an essential decrease in cloth stability [1,2] run simultaneously with the main reaction of cross-linking the cellulose macromolecules. When the cross-linking agent and the corresponding procedure are not correctly chosen, the degree of whiteness is significantly changed [3,4].

The aim of the present investigations is to find out whether it is possible to decrease the shrinkage of cotton and viscose fabrics by using a cross-linking agent of the dimethyloldihydroxy ethylenecarbamide (DMDHEC) type in the presence of different catalysts, and to follow the cross-linking effect on some of these materials' properties.

Experimental

The experiments were carried out with the following materials:

- 100% cotton fabric with a surface mass of 154 g m^{-2}, leno weaving, a linear density of the basic threads of 32 tex, a linear density of weft threads 23 tex, a warp density of 270 cm^{-1}, a weft density of 220 cm^{-1};
- 100% viscose crêpe fabric with a surface mass of 114 g m^{-2}, leno weaving, a linear density of the basic threads of 14 tex, a linear density of weft threads 14 tex, a warp density of 360 cm^{-1}, a weft density of 270 cm^{-1};
- dimethyloldihydroxy ethylenecarbamide (DMDHEC) used as a cross-linking agent:



- catalyst 1: melamin, an acidic donor used in carbamide cross-linking;
- catalyst 2: $MgCl_2$;
- catalyst 3: $MgCl_2$ - citric acid - tartaric acid (1:1:1);
- catalyst 4: citric acid - tartaric acid (1:1).

All the textile fabrics investigated were treated by the dry cross-linking method, which consists in:

- single soaking of foulard in an aqueous solution of the cross-linking agent at a temperature of 20°C, and a squeezing degree of 80%;
- drying at 130°C for 5 min;
- thermal treatment at 150°C for 4 min.

The concentrations of the cross-linking agent used are within the range of 5 to 30 g l^{-1}. It is important to note that in case of creasing decrease, the concentrations used usually range between 30 g l^{-1} and 50 g l^{-1}. The catalyst's quantity amounts to 30% of that of the cross-linking agent. In order to outline the effect of the cross-linking agent and that of the catalyst on the properties of the fabrics treated, this procedure was followed in all the experiments carried out.

Results and Discussion

The effectiveness of the cross-linking is evaluated by the total angle of relaxation which follows the creasing, according to BSS 9589-72 (the Somer method), by the variation of the dimension (the shrinkage) after machine washing according to BSS ISO 6330, by the tensile strength according to BSS ISO 5081, and by the degree of whiteness according to BSS EN ISO 105 J02. The values obtained are compared to those of the same indices but concerning non-treated samples.

Table 1 shows the values of the indices studied, depending on the concentration of the cross-linking agent and the catalyst type in case of a cotton fabric. It is seen from Table 1 that the total angle of relaxation is increased from 16 to 78 degrees when compared to that of the non-treated sample. It should be noted that crease resistance is only slightly increased when the concentration of DMDHEC is 5 g $l^{-1}$and 10 g l^{-1}. These results can be explained by the insignificant quantity of the cross-linking agent.

The shrinkage is considerably improved when the cross-linking procedure is carried out in the presence of DMDHEC at a concentration of 30 g l^{-1}, irrespectively of the catalyst used. The shrinkage decreases even with lower concentrations of DMDHEC (10 g l^{-1} and 20 g l^{-1}) if catalysts 3 and 4 are used. A higher

Table 1. *Effect of the catalyst type and the concentration of the cross-linking agent used on some properties of the cross-linked cotton fabric. Shrinkage was measured with an accuracy of ±5% for all types of fabrics.*

Catalyst, g l-1		Cross-linking agent DMDHEC, g l-1	Total angle of relaxation, degree	Shrinkage		Whiteness, %
				warp, %	weft, %	
-		Untreated cotton fabric	97	-3.5	-3.0	78.3
Catalyst 1	1.5	5.0	113	-3.0	-3.0	78.3
	3.0	10.0	129	-2.5	-2.0	78.3
	6.0	20.0	137	-2.0	-1.0	78.5
	9.0	30.0	149	-0.5	-0.5	81.7
Catalyst 2	1.5	5.0	132	-3.0	-3.0	78.3
	3.0	10.0	137	-2.0	-2.0	79.8
	6.0	20.0	153	-2.0	-1.5	80.2
	9.0	30.0	168	-0.5	-0.5	81.2
Catalyst 3	1.5	5.0	130	-2.0	-2.5	81.8
	3.0	10.0	132	-1.0	-1.5	81.0
	6.0	20.0	160	-1.0	-1.0	80.3
	9.0	30.0	175	-0.5	-0.5	78.5
Catalyst 4	1.5	5.0	100	-2.0	-2.0	78.8
	3.0	10.0	133	-1.5	-1.5	78.0
	6.0	20.0	148	-1.0	-1.0	77.0
	9.0	30.0	160	-0.5	-0.5	75.5

crease-resistance is reached with the application of catalyst 3 when compared to that with the rest of the catalysts studied, provided that one and the same concentration of DMDHEC is used. This is due to the higher catalytic activity of the catalyst mixture $MgCl_2$ - citric acid - tartaric acid, most probably determined by a synergistic effect. It is assumed that the latter is connected with the formation of a complex between the metal ion and the organic acid.

It is known that cellulose threads turn yellow on prolonged heating at temperatures within the range of 105°C-120°C, because of the accumulation of carbonyl groups at C2 and C3, or as a result of the presence of residues of wax-like substances. The degree of whiteness does not significantly change in most of the cases studied, which means that the short-term temperature treatment does not affect the threads' structure. The only exception is observed in the case of catalyst 4 application, where the solutions are acidic (the pH is ca. 2) and partial hydrolysis of the cross-linked agent is possible. The latter results in side products which affect the samples' whiteness.

Figure 1a illustrates the effect of the different catalysts used on the tensile strength in the process of cotton fabric cross-linking with DMDHEC of a concentration of 30 g l^{-1}. All the other samples investigated have a similar behaviour, although a tendency to tensile strength variation is outlined. It is found that it approaches that of the untreated material upon decrease of the cross-linking agent concentration. Figure 1 also shows the drastic decrease in the tensile strength when catalysts 3 and 4 are used. This results from the high catalytic activity and the lower pH values of the working solutions which in fact favour cellulose hydrolysis. This is the reason to recommend lower temperatures when carrying out the cross-linking procedure.

Figure 1b shows the elongation variation with the catalysts studied in a cotton fabric cross-linking in the presence of DMDHEC of a concentration of 30 g l^{-1}. It is seen that the elongation of the treated material is lower in all the experiments carried out when compared to that of the untreated one, and moreover it corresponds to decreased tensile strength. These effects are determined by the abrupt decrease in the macromolecules' mobility resulting from the cross-linking. In addition, the formation of each new covalent bond is connected with the disruption of a great number of hydrogen bonds. The energy of the latter is higher than that of the newly-formed one.

The investigations carried out show that the cross-linking with DMDHEC is effective at concentrations not lower than 20 g l^{-1} to 30 g l^{-1}. This determines the choice of the illustrative data for a viscose fabric in this work. Table 2 shows the values of the indices investigated for a viscose fabric in dependence on the cross-linking catalysts used. The data are not comparable to those for cotton fabric. The basic reason lies in the great difference between the structures of both fabrics.

For example, it is known that the amorphous regions accessible to physico-chemical interactions are only about 20% for cotton, while they are 50-60% for the viscose fabric. Besides, the viscose

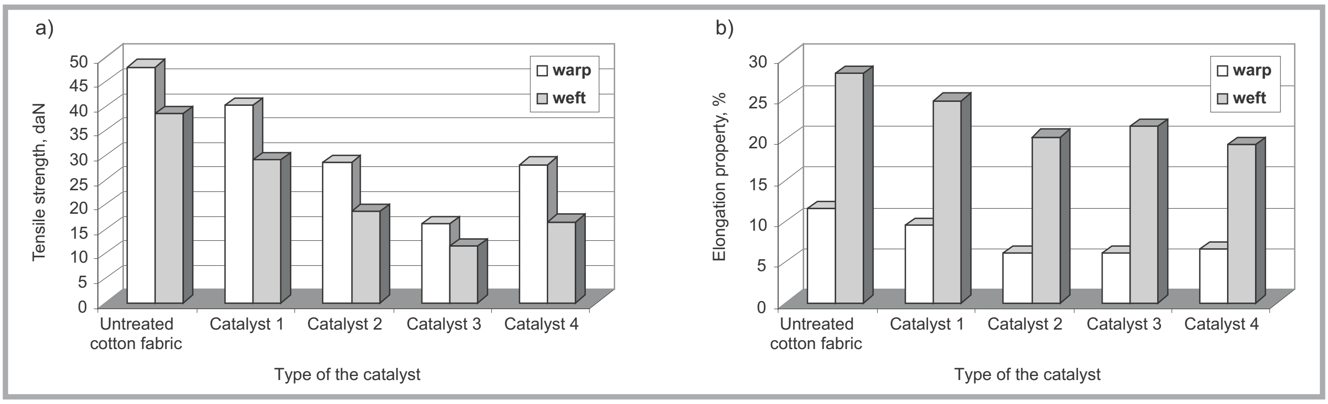


Figure 1. *Catalyst effect on the tensile strength (a) and elongation (b) of a cotton fabric; the cross-linking is carried out in the presence of DMDHEC with a concentration of 30 g l^{-1}.*

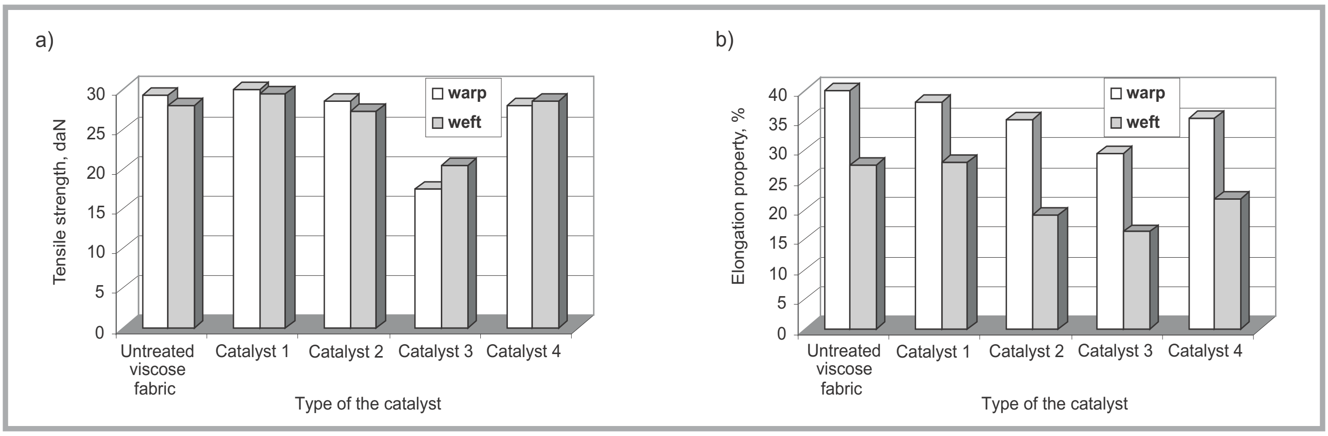


***Figure 2.** Catalyst effect on the tensile strength (a) and elongation (b) of a viscose fabric; the cross-linking is carried out in the presence of DMDHEC with a concentration of 30 g l^{-1}.*

threads have a lower average degree of polymerisation (300-400), a lower degree of orientation of the macromolecules towards the threads' axis, and a lower number of hydrogen bonds when compared to those referring to the natural cellulose threads [5]. All these facts in fact determine that the viscose structure is more accessible. But it is also worth mentioning that viscose fabrics are known for their high creasing and shrinkage, which usually reach to 12-16%.

The results in Table 2 also show that the cross-linking degree is high, which affects not only the relaxation angle but the fabric's shrinkage also. The decreased shrinkage effect obtained when catalysts 2 and 3 are used is long-lasting, and is most probably determined by the stabilisation of viscose fabric structure resulting from the formation of a great number of covalent bonds. The variation in the tensile strength (Figure 2a) is insignificant. It is likely that the total energy of the bonds formed compensates for that caused by the disruption of the hydrogen bonds. The degree of whiteness is not changed when compared to that of the untreated viscose fabric.

Figure 2 shows the effect of the catalysts studied on the tensile strength and the elongation of a viscose fabric in the case of cross-linking in the presence of DMDHEC of a concentration of 30 g l^{-1}. A change in the indices followed is observed only in the case of the application of catalyst 3. This is probably due to the presence of an acid in the working solutions, which catalyses not only the cross-linking reaction but that of the cellulose hydrolysis as well. The macromolecules' mobility does not decrease upon the introduction of a small number of chemical cross bonds to the hydrate cellulose threads. The changed values of elongation of cross-linked samples in the presence of catalyst 3 show that the reaction results in the formation of a greater number of covalent bonds. The latter limit the macromolecules' mobility, which in turn leads to decreased tensile strength and elongation.

***Table 2.** Effect of the catalyst type and the concentration of the cross-linking agent used on some properties of the cross-linked viscose fabric. Shrinkage was measured with an accuracy of ±5% for all types of fabrics.*

Catalyst, g l^{-1}		Cross-linking agent DMDHEC, g l^{-1}	Whiteness, %	Total angle of relaxation, degree	Shrinkage 1st washing		Shrinkage 5th washing	
					warp, %	weft, %	warp, %	weft, %
-		Untreated viscose fabric	66.7	145	-6.0	-3.0	-6.2	-3.4
Catalyst 1	6.0	20.0	64.3	190	-4.5	-2.0	-5.5	-3.0
	9.0	30.0	67.0	194	-4.0	-1.5	-5.0	-3.0
Catalyst 2	6.0	20.0	65.4	200	-2.0	-2.0	-2.5	-2.5
	9.0	30.0	67.3	207	-0.5	-1.5	-1.0	-2.0
Catalyst 3	6.0	20.0	66.0	205	-1.5	-1.0	-2.0	-1.0
	9.0	30.0	68.0	214	-0.5	0.0	-1.0	0.0
Catalyst 4	6.0	20.0	66.5	203	-2.5	-2.0	-4.5	-3.0
	9.0	30.0	67.3	212	-2.0	-1.5	-4.0	-2.0

The effect of HCHO quantity present in the treated fabrics is not covered in the present investigation because (i) the cross-linking agent is of a low HCHO content, and (ii) the concentrations used are lower than those required for creasing-resist finish.

Conclusion

The cotton and viscose fabrics cross-linked by a modified DMDHEC (of 20 g l^{-1} and 30 g l^{-1}) attain a long-lasting effect of decreased shrinkage. The application of the catalyst mixture of $MgCl_2$ - citric acid - tartaric acid intensifies the process of cross-linking, but this is also accompanied by a considerable loss in the cotton fabric strength. The use of the citric acid-tartaric acid catalyst mixture is linked with similar effects.

The structure of the viscose threads probably contains fewer hydrogen bonds, which determines the higher resistance towards the presence of acid when compared to cotton threads. This leads to a smaller strength loss when the cross-linking is carried out with DMDHEC in the presence of all the catalysts studied.

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Received 01.06.2004 Reviewed 26.10.2004