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# Crease-resistant Finish of Linen/Viscose Blend Fabrics

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Abstract

Linen/viscose blend fabrics are treated in different conditions with various concentrations of polycarboxylic acids (BTCA, CA, GA and MA) and N-methylol reagent (DMDHEU) with a relevant catalyst. The characterisation of such fibres is dependent on the degree of crosslinking, which is evaluated by the dry wrinkle recovery angle and tensile strength retention. In trials BTCA showed much better effects on the crease resistant finish of linen/viscose blend fabrics; when comparing the mixture of polycarboxylic acids, BTCA/GA treatment also gives moderate effects. High temperature curing is a handicap for linen/viscose blend fabric treatment when considering strength loss, whereas DMDHEU solutions give better results due to their lower acidity. In order to optimise the crease resistant finish of linen/viscose blend fabrics, it is acceptable to add a triethanolamine additive with the same ratio of the catalyst and cure at 160°C.

**Key words:** crease resistance, finish, fabrics, linen, viscose.

#### Introduction

One of the greatest problems with cellulosic fabric is its tendency to crease easily. Similarly this problem should be carefully considered for linen/viscose blend fabrics, which are widely used for outwear clothing as they exhibit a higher tendency when compared to cotton.

The approach mostly applied to improve crease recovery is introducing crosslinks between individual fibre chains. N-methylol reagents, such as dimethyloldihydroxylethyleneurea (DMDHEU), have long been used as durable press finishes for the production of wrinkle-resistant fabrics. However, formaldehyde-based finishing agents have caused worldwide concern over their impact on human health and the environment [1]. There have been efforts to achieve nonformaldehyde alternatives to replace traditional N-methylol reagents [2-6]. In 1988, Welch reported that 1,2,3,4-butanetetracarboxylic acid (BTCA) imparts a high level of wrinkle resistance to cotton fabric [7]. Moreover, BTCA has been confirmed as the most effective reagent with sodium hypophosphite (SHP) as the catalyst for cotton fabrics [8-11]; however, the high cost of BTCA has prevented its commercial application. The effectiveness of some other polycarboxylic acids as crease-resistant agents has also been studied [12-16]. It is now known that the crosslinking effect of polycarboxylic acids is maintained by the esterifaciton mechanism, and many studies have shown that cellulose esterification with a polycarboxylic acid is able to to form a cyclic anhydride and then to form an ester with a hydroxyl group in the cellulose macromolecule. Research has also established that improving the reactivity and number of hydroxyl groups in the macromolecules would also improve their accessibility to the acid and catalyst [17]. However, in the treatment of fabric with polycarboxylic acids, the fabric strength loss is considerable. Another downside of polycarboxylic acid finishing is the yellowing of the fabric treated [18]. Triethanolamine (TEA) has proved particularly effective as a curing additive in terms of yellowing and tensile strength loss [7, 19].

This study was undertaken to compare the crease resistance property of linen/ viscose blend fabrics treated with various polycarboxylic acids and a N-methylol reagent. During the literature search, we recognised that the optimum treatment conditions for these types of fabrics were not evident; hence we investigated the effect of the crosslinking agent concentration, the usage of the TEA additive, as well as curing conditions on the performance properties of the fabrics treated by us. The finishing recipes were grouped according to these purposes, and the performance measurements of the fabrics treated by these groups were evaluated.

### Experimental

In this study we used scoured and bleached linen (55%)/viscose (45%) blend fabric; Nm 30/1 viscose ends (28) x Ne 33/1 linen picks (26), weighting 156 g/m². The N-methylol crosslinking agent was dimethyloldihydroxylethyleneurea (DMDHEU), supplied by Rudolf-Duraner (*Rucon FAN*), and cata-

lyst MgCl<sub>2</sub> was used for the DMDHEU 1,2,3,4-butanetetracarboxylic system acid BTCA, citric acid CA, gallic acid GA, maleic acid MA, sodium hypophosphite SHP (Na<sub>2</sub>H<sub>2</sub>PO<sub>2</sub>) catalyst and additive trietanolamine TEA were used for polycarboxylic acid finishes, supplied by Fluka (BTCA and SHP), Sigma-Aldrich (CA, GA) and Merck-Schuchardt (MA), respectively. A polyethylene softener – PE, supplied by Rudolf Duraner (Ruco-LUB KSA), was used for the both polycarboxylic acids and DMDHEU. The fabric samples were padded twice to about an 80% wet pickup with aqueous solutions, details of which as well as drying and curing conditions are given in Table 1 (see page 92).

For fabric performance evaluation, we measured the conditioned dry wrinkle recovery angle (WRA) of the samples according to AATCC test method 66 using a James H. Heal Crease Recovery Angle Tester, as well as the tensile strength according to ASTM test method 1682 using an Instron 3360 Tensile Tester, respectively. The treated samples were also observed using a Jeol JSM 6060 scanning electron microscope at an accelerating voltage of 10 KV.

Recipes R3-R6 were based on 8% of the total crosslinking reagent concentration, which differ in the application of BTCA only and the mixture of BTCA with CA, GA and MA, respectively. All these solutions contained SHP as a catalyst with a 2:1 acid-to-catalyst ratio (w/w) and were cured at 180°C; meanwhile recipes R7-R10 were formed by reducing the total crosslinking agent concentration to 6%, the curing temperature to 160°C and applying the TEA additive with the same ratio of SHP catalyst. To make a com-

Table 1. Details of the crease-resistant finishes.

Recipe Code	Chemical Concentrations, % o.w.f.									Drying		Curing	
	DMDHEU	MgCl <sub>2</sub>	BTCA	CA	GA	MA	SHP	TEA	PE	°C	min	°C	min
N-methylol Systems													
R1	6	1,2	-	-	-	-	-	-	1	100	5	180	1,5
R2	6	1,2	-	_	_	_	_	_	0,5	100	5	160	3
Polycarboxylic acid Systems													
R3	-	-	8	-	-	-	4	-	1	85	5	180	1,5
R4	_	-	2	6	-	-	4	-	1	85	5	180	1,5
R5	-	-	2	-	6	-	4	-	1	85	5	180	1,5
R6	-	-	2	_	-	6	4	_	1	85	5	180	1,5
R7	_	_	6	_	_	_	3	3	0,5	85	5	160	3
R8	_	-	1	5	-	-	3	3	0,5	85	5	160	3
R9	-	-	1	-	5	-	3	3	0,5	85	5	160	3
R10		-	1	_	_	5	3	3	0,5	85	5	160	3
R11	_	_	2	6	_	_	3	3	0,5	85	5	160	3
R12	-	-	3	6	_	-	3	3	0,5	85	5	160	3
R13	_	-	3	6	_	_	3	3	0,5	85	5	135	3
R14		-	1	5	_	_	3	3	0,5	85	5	135	3
R15	_	_	1	5	_	_	3	3	0,5	85	5	135	5
R16	_	_	1	5	_	_	3	3	0,5	85	5	135	8
R17	_	_	2	4	_	_	3	3	0,5	85	5	135	3
R18	_	_	2	4	_	_	3	3	0,5	85	5	135	5
R19	_	_	2	4	_	_	3	3	0,5	85	5	135	8

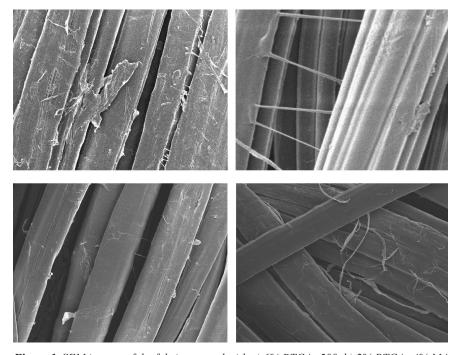


Figure 1. SEM images of the fabrics treated with a) 6% BTCA x500, b) 2% BTCA+4% MA x1.50k c), 2%BTCA+4% GA x500, d) %6 DMDHEU x500.

parison with the fabrics treated with DM-DHEU, recipes R1 and R2 were used.

To evaluate the effect of the crosslinking agent concentration only, we applied recipes R8, R11 and R12, respectively (where the concentrations were 6%, 8% 9%), which contained BTCA and CA.

The effect of the curing period was investigated using recipes R14-R16 and R17-R19 at different acid-to-acid ratios, where one can also find valuable information about the curing temperature by comparing R8 and R14 at a 6% crosslinking agent concentration or R12 and R13 at a 9% crosslinking agent concentration.

#### Results and discussion

Samples of SEM images of the treated linen/viscose blend fabrics are given in *Figure 1*. Crosslinking within the fibers can be observed.

Tensile strength retentions (%) and WRA values of the fabrics treated under different conditions (curing temperature, curing time and chemical concentration) are given in *Figures 2* and 3, the results of which are mean values of four replicates.

Figure 2 primarily shows that the tensile strength retention of the treated fabrics is generally higher in the warp direction, where the ends are viscose. Cellulosic chains are much shorter in viscose, leading to many more free hydroxyl side groups. In addition we expect that this would increase the degree of intermolecular crosslinking between viscose fibers. This can be also correlated with the higher WRA values in the warp directions of the treated fabrics, as seen in Figure 3, because it is believed that the increase in WRA is due to intermacromolecular crosslinking in fibers, whereas the strength loss is mainly due to the intramacromolecular crosslinking and acidity of the treatment solutions [8]; hence it is possible to assume that intramolecular crosslinking has a higher degree in linen ends, in which there is a greater extent of fiber polymerisation. This case should be experimentally investigated by treating cotton/viscose fabrics whose ends differ in the degree of polymerisation.

The effect of acidity on linen/viscose blend fabric treatment is evident when comparing R3-R6, the pH values of which are 2.3, 2.0, 4.0 and 2.1, respectively; since the tensile strength retentions in both directions are marginally higher when R5 with a lower acidity is applied. The finishing bath pH of DM-DHEU solutions is around 6.0, and the tensile strength retentions of fabrics treated by DMDHEU is higher than those of fabrics treated by polycarboxylic acid solutions. An improvement in strength retention and WRA is achieved by reducing the chemical concentration and curing temperature, as well as by adding the TEA additive to the solutions (R7-R10). The results obtained after treatments with R1 and R2 also highlight that reducing the curing temperature brings about improvements in the crease finish with DMDHEU. Since

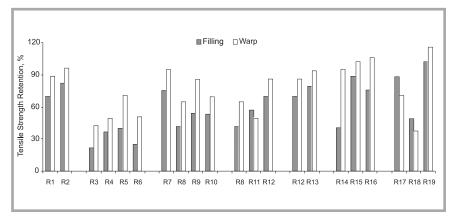


Figure 2. Tensile strength retentions in the filling and warp directions of the fabrics treated.

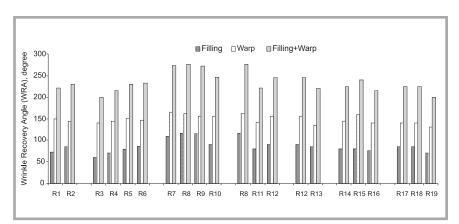


Figure 3. Wrinkle recovery angle (WRA) values of the fabrics treated.

exposure to high temperatures deteriorates, in particular, viscose fibers, curing at 160°C for 3 minutes gives moderate treatment effects. 6% BTCA treatment of linen/viscose blend fabric with SHP and TEA and curing at 160°C give the best result when considering strength retention and WRA. BTCA/CA, and BTCA/MA treatments give relatively lower retention or WRA values for this type of fabric; BTCA/GA treatment gives a better effect when comparing the mixtures of polycarboxylic acids.

Increasing the crosslinking chemical concentration (R8, R11 and R12) causes an increase in tensile strength retention but a decrease in WRA values, which may be explained by the importance of catalyst-to-acid and additive-to-acid ratios (w/w): when the ratios were lowered in R11 and R12, the degree of crosslinking was reduced. Another conclusion that can be made is that the critical (w/w) ratio of the catalyst-to-acid in the crease resistant finish of linen/viscose blend fabric is 2:1. The effect of the curing temperature can be observed when comparing the results of R8 and R14 or R12 and R13. A decrease in WRA and an increase in strength retention are obtained by reducing the curing temperature to 135°C. When the curing period is increased to 135°C, slight changes are observed; however, curing at 135°C for a certain amount of minutes gives lower WRA values, indicating an inadequate degree of crosslinking.

#### Conclusion

In this study, we investigated the crease resistance finish of linen/viscose blend fabrics treated with various polycarboxylic acids and a N-methylol reagent. We evaluated the efficiency of crease resistance systems in terms of the tensile strength retention and dry wrinkle recovery angle. We found that the crease resistant effect can be varied in a fabric whose ends have different fiber characteristics. For linen/viscose blend fabrics, the optimum treatment conditions for a crease resistant finish was as follows: 6% BTCA, 2:1 acid-to-catalyst (SHP) and acid-to-additive (TEA) ratio, and curing at 160°C. BTCA/GA treatment gave a better effect when comparing the mixture of polycarboxylic acids. The magnitude of the fabric tensile strength

loss was great for linen/viscose blend fabrics when curing at 180°C, whereas it was lower for DMDHEU systems, which could be attributed to the higher acidity of polycarboxylic acid systems.

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