Scanning Electron Microscopy, X-ray Microanalysis, and Thermogravimetric Assessment of Linen Fabrics Treated with Crease-Resisting Compound

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ABSTRACT: The physical, chemical, and thermal changes in fabrics treated with a commercial crease-resisting compound (CPNS) were investigated. The samples were analyzed by a scanning electron microscope equipped with an energy dispersive X-ray microanalyzer, elemental analysis, and differential thermogravimetry. X-ray microanalysis of the treated fabrics revealed that changes in gross integral counts of O, N, Mg, Al, Si, S, Cl, and Ca as a result of the treatments were related to the application rate of the compound. This result was broadly supported by mineral analysis data determined by standard wet chemistry methods. The effects of over- and undercuring were also investigated. In addition, the increase in nitrogen content as a result of the higher application rate can also be detected by EA. Differential thermogravimetric analyses of the samples showed distinct changes in the profile of the thermally stable fraction. This could be due to the formation of a crosslinked cellulose complex, which pyrolyzed at the significantly higher decomposition temperature of 481°C in comparison with 473°C for untreated material. The advantages of using instrumental techniques for assessing the efficiency of treatments are briefly discussed. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 1209-1219, 1999

Key words: linen; crease recovery; thermogravimetry; scanning electron microscopy; X-ray detector

INTRODUCTION

The bast fibers as a group possess a distinctive morphology; their complex fine structure requires specialized processes and chemical technologies and yields characteristic differences in yarn structure and physical performance relative to other cellulosic fibers.^{1–5} However, only flax is of commercial importance in fine textile manufacture, and it now constitutes a mere 1–2% of an-

nual consumption. Of the principal textile fiber classes, cellulosics collectively exhibit the poorest resistance to creasing, and attempts to investigate and improve the underlying mechanisms have attracted much research effort since the 1920s, when the first practical treatments were developed.⁶⁻⁸ Despite many advances during the subsequent period, in general, the achievement of full-easy-care properties on pure cellulosic fibers is difficult as the degree of crosslinking required can yield serious losses in tear/tensile strength and resistance to flexure and abrasion, with linen being particularly severely affected.^{1,6,9-12} Since the early 1980s, linen apparel fabrics have enjoyed an exclusive niche market with little apparent consumer pressure for crease-resist (CR) per-

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formance, ensuring a decade of relative prosperity for the industry but negligible reinvestment in research.

To date, a fully satisfactory CR finish for linen has proved elusive, and the available techniques are essentially modifications or fine-tuning of existing processes for cotton and rayon. An enormous variety of potential reagents and catalytic systems have been developed over the years,^{13,14} but all are based on reaction with hydroxyl groups on the cellulose molecule. Commercially, the Nalkylol compounds formed by reaction of a carbonyl species with an ---NH-containing nucleo-philic compound have consistently dominated the market, although, increasingly stringent legislation governing formaldehyde levels threatens to reduce their cost-effectiveness. Conformance requires chemical modification and has led to reinvestigation of alternative compounds, which are genuinely zero-formaldehyde and cannot undergo rearrangement or hydrolyze to generate formaldehvde.15

Although flax fiber featured prominently in early published work,^{16,17} rapid contraction of the linen industry provided little stimulus for further investigation until the mid-1980s. Elsewhere, scanning electron microscopy has long been successfully applied to assess changes in surface characteristics and topography of cotton fibres^{18,19}; but due to various technical difficulties, investigations of flax fiber or fabric using scanning electron microscopy (SEM) equipment equipped with X-ray analyzers have not been reported. The changes in the thermogravimetric (TG) weight loss and peak temperature of bast fibers^{20,21} and yarn²² have been investigated. Previous works have also suggested that crosslinking agents applied to fabric will change thermal characteristics.^{23,24} In addition, the effects of under or overcuring of the fabric on the pyrolysis patterns have not been investigated.

The aims of this investigation were to identify relationships between the changes in the thermograms and the application of the crosslinking agents on the fabric samples, and also to evaluate the potential use of SEM equipped with X-ray microanalysis for assessing surface characteristics.

MATERIALS AND METHODS

Treatment of Fabric

The test fabric used in this study was a fully bleached 260 g m^{-2} caustic-shrunk dress linen (Bal-

wide 2, Moygashel Ltd.) utilized as a reference in previous investigations.^{11,25} A proprietary reactant, Fixapret commercial crease-resisting compound (CPNS) (BASF) was selected on the basis of its widespread use in linen finishing and known performance. The catalyst employed was magnesium chloride hexahydrate, Mg Cl₂ 6H₂O (GPR), added at 15% on the weight of fiber (owf). Application of the catalyzed solution was by industrial pad-mangle, in a conventional batch pad-dry-cure process, with the cure cycle using a temperature of 150°C for 4 min. Where applied, afterwashing consisted of 5 min of agitation in a 10 g L soda ash and 5 g L warcodet K (Warwick Chemicals) solution at 60°C, and rinsing. All samples were dried to original dimensions under tension to eliminate shrinkage. Each treatment was prepared in triplicate.

The reactant applied was assumed to be pure monomeric 1,3-dimethylol 4,5-dihydroxy-2-imidazolidinone (trivially known as DMDHEU), a long established compound yielding an excellent balance of properties.^{7,9,13,14}

The CPNS structure (mol wt \sim 176.08) is as follows:



Estimation of Crease Recovery and Abrasion Resistance

Fabric samples were assessed for crease resistance by the Shirley method²⁶; and abrasion resistance was assessed by a modified Martindale abrasion tester²⁷ in a NATLAS-approved labora-

Table I	Assessment of Changes in Dry Crease
Recovery	Angle (CRA) and Modified Martindale
Abrasion	Rubs/Revs (MMA) of Fabric
Samples '	Freated with CPNS

Resin Applied (%)	MMA	CRA (°)
0	770	67
2 SC	1030	72
4 SC	430	100
$5 \ SC$	390	110
3 OC	60	136

A mean of four replicates per sample; all samples afterwashed. SC is the abbreviation for standard cure; OC, for overcured.



Figure 1 Micrographs of fibers from undercured fabric showing (a) minimal changes in surface characteristics (bar = 10 μ m) and (b) higher magnification showing fine surface striations corresponding to fibril alignment (bar = 5 μ m).

tory. Each sample was analyzed in triplicate. The levels of free and release formaldehyde in finished fabrics were determined by Shirley and AATCC methods, respectively, to verify the continuity of reagents and the finishing process.

In order to assess the changes in the surface composition of treated fabrics, the samples were abraded to breakdown point on a standard Martindale abrasion tester. The same materials were finely cut with the aid of a pair of serrated scissors. The cut samples were used for chemical and instrumental analyses.

Scanning Electron Microscopy and X-ray Microanalysis

Dry samples of the prepared fabrics were mounted on SEM stubs and sputter-coated with platinum for visual SEM, and carbon evaporation coated for X-ray microanalysis, before examination in a JEOL 35CF SEM. Photographs of the samples were taken randomly. A minimum of three X-ray scans per treatment for 1200 s per analysis were carried out. The data derived from the assessments were calculated qualitatively by X-ray microanalysis software (Link, eXL System, Oxford Instrument).

Thermogravimetric Analysis

All samples for differential thermogravimetry were prepared by cutting the samples to less than 1 mm length with a pair of serrated scissors. Differential thermogravimetric (DTG) analysis of the fibers was determined at heating rate of 20°C min with an air flush rate of 20 mm min. A rep-



Figure 2 Micrographs of fibers from overcured fabric (a) heavily etched surface layers (bar = 5 μ m) and (b) damaged areas and associated abraded debris (bar = 5 μ m).



Figure 3 Micrographs of untreated fabric (a) untreated fabric showing weft ends (bar = $300 \ \mu$ m), (b) fiber surface showing no fibril structure (bar = $5 \ \mu$ m), and (c) fibers showing residual nonfibrous materials (bar = $20 \ \mu$ m).

resentative subsample in the range of 3.1–3.2 mg of flax was used for each run. Each sample was analyzed 3–4 times. The DTG curve and the thermogravimetric (TG) weight loss data were calculated by Graphware (Mettler, Toledo). Details on the pyrolysis pattern of fiber fractions have been

described previously.²⁸ The primary decomposition band extended from 200 to 400°C, this being followed by a minor peak from 400 to 600°C. The changes in weight losses and decomposition temperatures within the two ranges were analyzed.



Figure 4 Micrographs of samples treated with 2% CPNS (a) fabric (bar = 300 μ m) showing fiber ends from both warp and weft yarns, (b) fiber surface showing fibril alignment (bar = 5 μ m), and (c) fibers showing residual nonfibrous materials (bar = 20 μ m).



Figure 5 Micrographs of samples treated with 4% CPNS (a) fabric showing reduced fiber ends (bar = 300 μ m), (b) fiber surface showing deep fissures between the fibrils (bar = 5 μ m), and (c) fibers showing residual nonfibrous materials and damage (bar = 20 μ m).

Chemical Analysis

The samples were analyzed for ash content and the presence of magnesium, aluminium, silicon, sulfur, calcium, and chloride by methods recommended by the society of analytical chemists.²⁹

Elemental Analysis

The samples were analyzed by combusting (6-7 mg) in pure oxygen with an inert carrier gas (argon). The resultant gases, carbon dioxide, water, and nitrogen dioxide were measured as carbon



Figure 6 Micrographs of samples treated with 5% CPNS (a) fabric (bar = 300 μ m) showing fiber ends, (b) fiber surface showing fine striations and cracks (bar = 5 μ m), and (c) fibers showing residual nonfibrous materials (bar = 20 μ m).



Figure 7 Micrographs of fabric samples after abrasion testing (a) 2% CPNS, normal curing conditions (bar = 300 μ m) showing deformation of the weave structure and free fiber ends, (b) 2% CPNS undercured, showing embrittled fiber ends (bar = 300 μ m), (c) abraded-control fabric showing cleanly broken off fiber ends (bar = 300 μ m), and (d) as (a) but nonafterwashed showing clean fracture of individual fibers (bar = 300 μ m).

(C), hydrogen (H), and nitrogen (N), respectively, using gas chromatography (PE 2400 CHN). Each sample was analyzed in triplicate.

RESULTS

Crease Recovery Angles and Abrasion Resistance

Improvements in the crease recovery angles of the treated fabrics were related to the proportion of reactant applied; and at a nominal 2% CPNS application rate, the abrasion resistance improved significantly compared to untreated control samples, a phenomenon peculiar to linen and in accordance with previously reported data.^{11,25} However, at higher application rates, abrasion resistance declined progressively (Table I) as crease recovery angles increased. Excellent crease recovery performance was associated with very poor abrasion resistance

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	Ν	0	Mg	Al	Si	S	Cl	Ca
3% OC	670	20,005	3539	4986	4164	3473	3816	6387
5% UC	397	15,117	2512	3415	3623	3441	4562	5635
Control	506	21,207	2749	4223	3409	2579	3489	6767
2C SC	384	12,378	2175	3434	2829	2281	3045	3913
4C SC	395	12,627	2330	3434	2889	2163	2899	3701
5C SC	588	15,428	2995	4247	3684	2784	3588	4749

Table II Comparison of the Gross Integral Counts $(\times 10^2)$ of Eight Elements Present on the Fabric Samples Treated with CPNS at Different Concentrations

A mean of a minimum of four counts per sample; OC is the abbreviation for overcured; UC, for undercured; and SC for standard cure.

tance in overcured fabric samples treated at the 3% rate.

Scanning Electron Microscopy

The micrographs reveal that the effect of crosslinking is visible as a gradual increase in the definition of fine structural detail in the surface regions of individual fibers. Undercured samples exhibited minimal changes in surface characteristics [Fig. 1(a)]. At higher magnification, fine surface striations corresponding to fibril alignment are apparent [Fig. 1(b)]. In contrast, overcuring at a 3% CPNS application rate was easily detectable as fibers showed heavily etched surface layers with extreme sensitivity to mild flexure or abrasion incurred during the afterwashing and drying processes [Fig. 2(a)]. At higher magnification, the damaged areas and associated abraded debris are clearly visible [Fig. 2(b)].

At low magnification, the micrographs show the effects of shrinkage tension on the surface

Table III Carbon (C), Hydrogen (H), and Nitrogen (N) Contents of the Fabric Samples Treated with CPNS at Various Concentrations and Curing Degrees

Treatment	% Carbon	% Hydrogen	% Nitrogen
3% OC	41.67	6.47	0.41
5% UC	41.76	6.58	0.06
Control	41.51	6.45	0.05
2% SC	41.70	6.30	0.12
4% SC	41.43	6.31	0.37
5% SC	41.37	6.32	0.46
SE	$0.195 \mathrm{NS}$	0.144^{a}	0.032^{a}

OC is the abbreviation for overcured; UC, for undercured; SC, for standard cure; SE, for standard error of means. ^a Significant at P < 0.001; NS, not significant.

topology of untreated control fabric [Fig. 3(a)] in contrast to dimensionally stabilized crosslinked samples [Figs. 4(a), 5(a), and 6(a)]. The main changes in the surface characteristics of the fibers treated at 0, 2, 4, and 5% CPNS application rates can be seen as a cleaner surface appearance at higher application rates [Figs. 3(b) and (c), 4(b) and (c), 5(b) and (c), and 6(b) and 6(c)]. In addition, deep fissures appear to be better defined as the crosslink density increases to 4% and, thereafter, apparently declined at a 5% level. The micrographs showed that the warp varns remained largely intact in all samples. The fabric samples after abrasion testing revealed significant reduction of free fiber ends on standard, [Fig. 7(a)] compared to undercured [Fig. 7(b)], samples due to fiber embrittlement. In addition, weave structure of the standard-cured fabric has undergone permanent deformation and spreading as a result of the mechanical shear and stress. On the abraded control samples, individual fibers of the weft yarns were cleanly broken off, illustrating the characteristic response of linen to abrasive actions [Fig. 7(c)] compared with the standard or overcured samples [Fig. 7(a) and 7(b)]. Extensive but localized fibrillation is apparent at the cut ends (Figs. 7(a)-(c)]. In contrast, the nonafterwashed sample treated at the 2% level showed a clean fracture of individual fibers with little fibrillation [Fig. 7(d)].

X-ray Analysis

Detailed X-ray analyses of the major ions present in the fabric revealed that gross integral counts of N, O, Mg, Al, Si, S, Cl, and Ca present on the surface of the fabrics increased at higher application rate (Table II). The effects of undercuring can be detected as reduction in the counts of Mg and N compared with the controls; and, in contrast, high levels of all elements were obtained in overcured samples (Table II).

Elemental Analysis

The changes in levels of carbon, hydrogen, and nitrogen as a result of treating the fabrics with CPNS at different rates was determined, and results suggested that significant changes were mainly associated with nitrogen content (Table III). The levels of carbon and hydrogen in all the treated fabric samples remained unchanged.

Differential Thermogravimetry

Thermal analyses of the test materials revealed that the fiber components pyrolyzed into two distinct decomposition peaks. The thermograms of the treated fabrics were affected by changes in the application rate (Table IV). The weight losses in temperature range of 200–400°C representing the primary peak were significantly lower (P< 0.001) in the treated samples compared with the control. The effect at the higher pyrolysis band of 400–600°C was the reverse as the lowest weight loss was detected in control samples and the highest in treated fabrics (Fig. 8). These differences were also significant (P < 0.001). The changes in the peak decomposition temperatures of the primary peak (PP) and secondary peak (SP) were also related to increases in the application rate of the CR compounds. The quantity of residual ash left in the crucible at the end of the DTG run correlated to the presence of crosslinking.

The effects of undercuring fabrics treated at 4 and 5% at 150°C for 2 min can be detected as a higher weight loss in the primary peak and lower

weight loss in the secondary peak compared with treated fabric cured at the standard 4 min (Table IV). Similarly, overcuring of treated fabrics (3%) can also be detected from the high decomposition temperature of 485°C compared with 473°C with standard curing time of 4 min. In contrast, decomposition temperatures in both the PP and SP were reduced in nonafterwashed samples treated at a 3% application rate compared to the control (Table IV). Moreover, the presence of an additional decomposition peak at near 278°C in the thermogram of nonsoaped samples due to the presence of catalyst and reactant on the fabric can also be detected (Fig. 8). These changes were associated with a reduction in weight loss in the main peak and an increase in the latter decomposition peak.

Chemical Analysis

Analysis of the samples for the test minerals has shown that fixation of CPNS during under- and overcuring of the test samples can be detected as changes in the concentrations of Mg, Si, Ca, and S and chlorides. At a 2% application rate, significant (P < 0.001) increases in the concentration of Al, Mg, Si, S, and chloride were detected; and, thereafter, at the higher levels, the elements and chloride content declined. In contrast, calcium concentration increased at 4% and then reduced at 5% application rates. Undercuring at 5% significantly increased the concentration of all the test minerals except Si compared to the control sample. However, on overcured samples, significant reductions in the levels of Mg, Si, and Ca, and significant increases in Al and chloride concentrations were detected (Table V). Ash contents of all samples were within normally acceptable

Sample	PP WTL	PP T	SPWTL	SPT	DTG-ASH
3% NA	66.9	334.5	24.4	467.4	6.2
3% OC	72.3	345.3	19.7	484.6	5.8
5% UC	76.6	345.3	16.0	475.2	4.1
Control	75.5	347.7	17.8	479.1	3.0
2% SC	73.6	346.9	17.9	472.9	6.1
4% SC	71.6	345.3	19.9	478.3	6.4
5% SC	70.2	346.1	21.3	481.5	6.3
SE	0.863^{a}	0.801^{b}	0.227^{b}	2.272^{b}	0.376^{a}

Table IVThermogravimetric Analysis of Control and Treated Fabric Samples Showing WeightLosses and Decomposition Temperature in the Two Pyrolysis Peaks

PP is the abbreviation for primary peak; SP, secondary peak; NA, not afterwashed; OC, overcured; UC, undercured (min); SC, standard cure (min); SE, standard error of means.

^a Significant at -P < 0.001.

^b Significant at -P < 0.01.



Figure 8 Comparison of thermograms of fabrics: (a) 3% CPNS, nonafterwashed; (b) 3% CPNS, afterwashed; (c) untreated showing changes in the weight loss curve (- - -) and differential decomposition peaks (----).

limits, but an increase in the application rate correlated with reduction in ash in the treated fabric samples. However, on overcuring, a slight increase in ash content was detected compared to the control (Table V).

Degree of Fixation

The ratio of excess nitrogen in finished fabric to reactant uptake was calculated on the basis of a complete crosslinking reaction with cellulose and agreed well with the obtained abrasion–CR performance. The standard curing conditions yielded satisfactory results; the moderately overcured sample exhibited excellent fixation but incurred severe tendering, while the undercured sample underwent little crosslinking.

DISCUSSION

Despite the current emphasis on more subtle aspects of textile performance via fabric objective measurement (FOM) assessment³⁰ and surface morphology,³¹ the current literature on flax fiber is far from satisfactory. No modern reviews of published work on morphology and instrumental analysis, for example, have been published apart from specific references collated in relevant sections of Mukherjee and Radhakrishnan,⁴ and Sharma and van Sumere,³² and this is exacerbated by much material appearing in limited circulation publications. Recent comprehensive reviews of work on physical, chemical, and instrumental analysis of textile fibers^{33,34} contain scattered references to flax, but significant omissions occur.

As CR treatment of linen is primarily intended to improve dry crease recovery performance, the reaction is carried out in the dry, dehydrated state.¹⁰ In contrast with cotton fibers, dramatic changes in appearance do not occur with flax fiber. However, a gradual increase in the definition of fine structural elements within the fiber surface can be observed, as these are partially constrained in the dehydrated conformation by crosslink formation. This may offer a means of examining the substructure of ultimate fibers.

The fabric construction (caustic-shrunk plain weave) used in such dress linen leads to exposed weft crowns, and, consequently, these undergo most abrasive damage. Elastic recovery and resilience, initially low in untreated fiber, deteriorate further on crosslinking, leading to poor resistance to abrasive or flexural forces. Individual fibers exhibit clean fractures/breakage with little or no fibrillation or fraying visibly evident. The cumulative evidence from DTG, SEM, and physical tests underline previous reviewers comments^{10,12,35} on the difficulties inherent in achieving durable CR finishes on bast fibers.

The use of SEM equipped with an X-ray analyzer to identify the effects of crosslinking has shown that the changes in the proportions of nitrogen, oxygen, Mg, Si, Ca, and Cl with increasing concentrations of reactant can be detected by this technique. In addition, the X-ray data also agrees with the changes in mineral concentrations, as

Treatment	Al (g/kg)	Mg (g/kg)	Si (g/kg)	S (%)	Ca (g/kg)	Chloride (%)	Ash (%)
3% OC	34.05	29.58	6.33	0.16	13.41	0.12	0.56
5% UC	12.69	61.78	7.26	0.15	58.91	0.18	0.82
0% Control	8.23	33.84	17.41	0.13	45.91	0.06	0.50
2% SC	5.16	28.52	5.77	0.16	18.05	0.22	0.66
4% SC	2.17	18.46	3.97	0.12	45.69	0.19	0.41
5% SC SE	2.59 0.374 ^a	09.33 0.395ª	1.29 0.551ª	0.11 0.017 ^a	$38.54 \\ 0.555^{a}$	$0.16 \\ 0.012^{\rm b}$	$0.36 \\ 0.052^{a}$

Table V Comparison of Al, Mg, Si, S, Ca, Chloride (g kg) and Ash (%) Present in the Treated and Control Fabrics

OC is the abbreviation for overcured; UC, undercured; SC, standard cure; SE, standard error of means.

^a Significant at -P < 0.001.

^b Significant at -P < 0.01.

determined by the wet chemistry method. The micrographs also revealed that the reactants were distributed well throughout the fabric, and this can be observed from the distribution of certain minerals determined by X-ray analysis. Ash content decreased with increases in the density of crosslinking, and this trend could be explained by reduction in fiber absorbency and regain. Alternatively, this could be due to catalyst interaction with insoluble calcium salts. The water supply contained ~ 280 ppm of total hardness, which may have contributed to the ash values obtained.

The thermograms of fabric samples treated at the higher application rate correlated with a reduction in weight loss in the primary peak and an increase in weight loss in the secondary peak. Furthermore, the decomposition temperatures were reduced in the primary peak and increased in the secondary peak. This indicated that crosslinking agents on the fabric increased thermal stability,^{23,24} enhancing the formation of residual carbonaceous char. The observed changes in DTG-ash content of the treated samples did not correlate quantitatively with application rate or nitrogen content but proved very sensitive to the presence of crosslinks. On this basis, thermal analysis could be used to detect the presence or absence of crosslinking in fabric samples.

Further, analyses of fabric samples treated with a similar application rate but differing in curing time have shown that even this can be detected as changes in the pyrolysis pattern, mainly as an increase or reduction in weight losses or higher decomposition temperatures. This technique can also readily identify afterwashed samples, through changes in weight loss and pyrolysis peak (278°C) are associated with dehydration of MgCl₂ nH₂O.

One of the key factors in the market decline of linen apparel fabrics was that true smooth-drying wash and wear performance was not attainable with 100% linen due to difficulties in imparting a good crease recovery angle while maintaining acceptable abrasion resistance.^{6,10} The relationship between the two characteristics for a range of reactants on linen has been examined. The re-

OWF Nominal %	OWF Conc of Reactant	N (%) ^a	Nitrogen (%) ^b	Nitrogen (%) ^c	Fixation (%)
3% (OC)	2.81	0.18	0.41	0.36	81.9
5% (UC)	4.38	0.72	0.06	0.01	1.40
0% (Control)	0.00	0.00	0.05	0.00	00.0
2% (SC)	1.80	0.29	0.12	0.07	24.1
4% (SC)	3.51	0.56	0.37	0.32	56.7
5% (SC)	4.38	0.71	0.46	0.41	58.1

Table VI Degree of Fixation Based on Initial Concentration of Reactant

OWF is the abbreviation for "on the weight of fiber"; SC, standard cure; OC, overcured; UC, undercured.

^a Total nitrogen determined from the OWF concentration of the reactants.

^b Total nitrogen as determined by elemental analysis.

^c After subtraction of nitrogen in the untreated fabric.

sults from the current study support the relationship reported by Kernaghan.¹¹

Previously, the assessment of linen fabrics treated with crosslinking agents was limited to methods such as uncreasing angle, abrasion resistance, acid hydrolysis, and viscometry.^{33,34,36,37} This investigation has shown that instrumental methods, such as thermal analysis and SEM equipped with an X-ray analyzer could be used for assessing the degree, effect of crosslinking, and changes in the composition of treated fabrics to aid development of new reactant compounds and curing techniques for a range of linen products.

To investigate whether DTG is generally applicable to crosslinked fabrics, irrespective of the precise chemical nature of the reactant/resin crosslinks, preliminary trials with other commercially available CR agents and reactants offering a range of structures are being carried out, forming the basis of a further report.

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