

FEATURE ARTICLE

Novel delayed-cure, durable press, shrink-resist treatment of wool fabrics and garments

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Commonwealth Scholarship Commission (UK); University of Mauritius

Abstract

The relatively high heat stability of commercial easy-care chemicals for cotton allows for greater flexibility in garment manufacturing, in particular the delayed curing of durable press, wrinkle-resistant, shrink-resist (SR) finishes on garments. Typically, the technology involves application of the wet chemical finish to the open width fabric, drying, storage, garment manufacture, heat pressing of the creases/pleats, and lastly garment baking to fully cure the durable press/crease-resistant/SR finish. By contrast, for the wool industry, there is no comparable technology, and this presents an obvious commercial weakness. The reason for this technical deficiency is that the prepolymers used for imparting machine washability and potentially durable press to wool apparel polymerise on the fabric at room temperature in storage, thereby losing their reactivity. Subsequent manufacturing into the final garment still allows the fabric to offer stability to laundering, but the loss of prepolymer reactivity precludes any potential for introducing durable creases/pleats into the garment as an integrated late-stage garment process. In this study, we present a simple solution to this technical deficiency through the use of cyclodextrin-based technology and deliver a delayed-cure, durable press, machine-washable wool technology ready for market. In any commercial textile process, the effect of chemical finishing on the final fabric colour is important. It is demonstrated in this study that the addition of HP- β -cyclodextrin into the Synthappret BAP and EC 1354 formulations has an instrumentally detectable effect on the colour of the dyed wool fabric, but that this difference is less than one colour difference (ΔE_{CMC}) unit.

Editor-in-Chief's recommendation: The appeal of a garment for some depends not just on the cut and colour of its fabric, but also on how easy it is to care for. Processing technologies that enable woollen textiles to withstand the rigours of machine washing are therefore prized by industry. They usually form an important part of the complex chain of steps needed to manufacture an



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item of clothing from raw wool. One means of bestowing resilience on wool fabric entails treatment with reactive macromolecules to stabilise its shape, dimensions and surface towards repeated laundering. This Feature article outlines a commercially attractive approach to extend the capability of such polymer-only techniques. The authors found that inexpensive and straightforward modification of industrial pre-polymer formulations slowed their reactivity sufficiently to allow them to be used in another capacity. In addition to fulfilling their original purpose, the applied reactive materials could also be utilised to confer long-lasting shaping on the fabric, such as definition of creases, by means of a final heat-setting step. This possibility has not previously been an option owing to interaction of wool with the applied reactive materials. Another welcome aspect of the novel methodology is the lack of any significant impact on fabric coloration: changes in colour of the wool caused by the modifications were on the fringes of being perceptible. This Feature article will thus be of great interest to researchers and technologists engaged in producing more durable, higher-quality garments from this sustainable substrate.

Technology and subsequently became Head of School. He is Director of the 3D Weaving Innovation Centre at the University of Leeds. His main interests are in the modification of fibrous materials to improve performance and the complementary associated bulk analysis and surface chemistry. Research has focused on easy care finishing of cotton and wool, novel coloration, fibre degradation and protective mechanisms, cultural heritage science, hair modification and performance, general wet chemical and biotechnological modification of textiles, recycling, technical textiles, 3D weaving, paper technology and health-care textiles/materials. He has published widely with over 100 research publications and is a member of several Editorial Boards for international research journals. He is a Liveryman in the Dyers Company in London and was awarded the Weavers Company Silver Medal for outstanding contribution to Textile Education and Research in 2008. In 2015 he was awarded the Society of Dyers and Colourists Gold Medal for outstanding contribution to Textile & Colour Education and is currently a Trustee on the Society of Dyers and Colourists Board.



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ORIGINAL ARTICLE

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1 | INTRODUCTION

The increasing demand from consumers for easy-care garments has prompted the development of processes that impart dimensional stability to laundering, maintenance of appearance and durable press. These performance features can be readily engineered into cotton and polyester fabrics by either applying crosslinking crease-resistant finishes to the cotton or by heat setting the fabric at different stages along

the garment production route.¹⁻⁴ By contrast, for wool-based materials/garments, there is much less process flexibility and no comparable technology is currently available.^{5,6}

Most commonly, wool can be treated as top in the chlorine/Hercosett process to modify the fibre surface and eliminate the directional frictional effect and associated felting.^{6,7} Alternatively, wool can be treated with soft prepolymers as a wet pad application to the open width fabric and is then subsequently heat-cured to introduce inter-fibre bonding that

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“locks-in” the fabric dimensions.⁶ While these are effective as individual processes, there is no wider opportunity for processing at different stages in manufacturing or inter-process integration. Therefore, a major challenge in the production of easy-care wool products is the introduction of durable creases and machine washability/shrink resistance into wool garments at the garment-processing stage.^{5,7} Currently, this can be achieved by:

- utilising a crease-setting technique, which is labour-intensive and includes the spray application of a reducing agent onto the pre-shrink-resist-treated garment and then steam pressing to impart permanent creases;
- applying a shrink-resist (SR) prepolymer to the fabric, drying, making up the garment, and then introducing durable press and machine washability/shrink resistance by curing the polymer by garment baking at an elevated temperature of *ca.* 100–160°C.⁸ However, a major drawback of this polymer treatment is the undesirable premature polymerisation of the prepolymer during clothing manufacture or during fabric storage at relatively high ambient temperatures in areas such as the Far East;
- dipping the final garment into the SR prepolymer formulation, drying, heat pressing to introduce the creases, and then finally garment baking. However, increasingly, the application of wet chemicals at the garment stage has become less attractive in the textile clothing industry and this is not a preferred option.

Therefore, the need for a flexible, delayed-cure process based on a pad application of the prepolymer formulation onto the fabric, drying, potentially storing for an extended period, garment manufacturing, and then finally introducing permanent press and machine washability at the garment stage by a heat-curing process, similar to existing cotton technology, is highly attractive and needed in the wool industry.^{5,7,9–11}

In 1988, Szejtli reported that α -, β - and γ -cyclodextrins, and their derivatives, are cyclic, three-dimensional oligosaccharides molecules with a central hydrophobic cavity and hydrophilic outer surface, which ensure compatibility with aqueous solutions.¹² The central cyclodextrin cavity can reversibly complex with “guest” molecules and form inclusion complexes. A key factor in cyclodextrin inclusion complex formation is the cavity size and whether the molecular species can “fit” into the inclusion cavity. α -cyclodextrins have relatively small cavities and accordingly are limited in the range of species that can be accommodated to form the inclusion complex. Comparison of the dimensions of the β -cyclodextrins and the γ -cyclodextrins indicates that the β -cyclodextrin molecule is intermediate in size but offers a broad range of complexation potential. This, coupled to the relatively higher costs of the γ -cyclodextrins, results in the much greater research into and commercial focus upon the β -cyclodextrins.

Another factor in inclusion complex formation is the nature of the polarity of the central cavity. Typically, the cyclodextrin cavities are hydrophobic but can be altered by incorporating non-polar or polar substituents into the glucose rings. In this study, we examine both the (M)- β -cyclodextrin and hydroxypropylated β -cyclodextrin derivatives as well as the unmodified cyclodextrin.

There is extensive research reported in the literature related to the structure and applications of cyclodextrins with key areas involving odour absorption, fragrance release, drug delivery, wider medical applications, and also the ability to protect molecules from heat and ultraviolet (UV) light.^{12–16}

In this study, the potential for extending the lifetime stability of shrinkproofing prepolymers on the wool fabric was investigated. A simple process was developed based on current commercial technology that establishes the opportunity for late-stage, garment-based heat curing, similar to that of cotton garment processing, and imparting machine washability and durable press. A range of cyclodextrin derivatives was evaluated, and their relative effectiveness in imparting improved thermal stability and associated delayed curing of the finish on the wool fabric was established.

2 | EXPERIMENTAL

2.1 | Materials

A 100% wool worsted fabric (180 g/m²) was used throughout this study, a yarn-dyed, 2/1 twill construction (1/32 × 1/32 Nm). For the coloration study a 100% wool worsted fabric (170 g/m²) was used, and the fabric was dyed with 4% owf Neutrilan Black M-R (1:2 metal complex dye, disulphonated).

β -cyclodextrin (Cavamax W7), (M)- β -cyclodextrin (Cavamax W7 M) and HP β -cyclodextrin (Cavamax W7 HP) were kindly supplied by Wacker-Chemie. Synthappret BAP (tri-functional carbamoyl sulphonate-terminated poly(ether) urethane prepolymer) was kindly supplied by Bayer AG. The shrinkproofing polymer, EC 1364 (Bunte salt terminated prepolymer), was kindly supplied by eChem. Protolan 94612 (modified polyisocyanate terminated prepolymer) and Securlana K (tri-functional polyether-based tris(sulphothio-glycolate) prepolymer, were kindly supplied by Rotta GmbH and Cognis, respectively.

The sodium bicarbonate and sodium carbonate, laboratory grade, were purchased from Aldrich Chemicals.

2.2 | Application of SR prepolymer formulations to wool fabric

The application of Synthappret BAP followed the manufacturer's recommended methodology, in which the wool fabrics

were padded with 40 g/L Synthappret BAP (50% w/v) solution (adjusted to pH 7.3-7.5 using sodium bicarbonate) at 90%-100% wet pick-up, dried at 70°C for 5 minutes, then immediately cured at 150°C for 3 minutes.

The recommended EC 1364 treatment involved padding the wool fabrics with 80 g/L EC 1364 (40% w/v) solution (adjusted to pH 8.2 using sodium carbonate) at 90%-100% wet pick-up, drying at 70°C for 5 minutes then immediately curing at 150°C for 3 minutes.

To assess the effect of β -cyclodextrin and its derivatives on the delay-curing of the SR prepolymers, the Synthappret BAP or EC 1364 liquids were mixed thoroughly with varying amounts of solid β -cyclodextrin and its derivatives to produce a homogeneous paste. The paste was then dissolved gradually in distilled water and the solution was adjusted to pH 7.4-7.5 using sodium bicarbonate with the Synthappret BAP formulation, or was adjusted to pH 8.2 using sodium carbonate with the EC 1364 formulation. The wool fabric samples were then padded with the liquor to 90%-100% wet pick-up of the SR prepolymer formulations, dried at 70°C for 5 minutes then incubated for specified time periods (1-20 days) and temperature conditions (40-80°C) prior to heat curing. Following incubation, one set of fabrics was washed without being cured and was identified as IW (pad-dry-incubate-wash). The other set of fabric samples was heat-cured after incubation and was identified as ICW (pad-dry-incubate-heat cure-wash). The incubated and incubated-cured samples were then subjected to the fabric shrinkage tests.

2.3 | Felting area shrinkage

The felting shrinkage of the treated wool fabrics was assessed using a Wascator FOM 71P machine, where the fabrics were subjected to five wash cycles based on the ISO 6330:1984 5A wash cycle programme (5 \times 5A wash cycle), with 2 g/L of ECE phosphate-based reference detergent and 2 kg wash load (100% polyester knitted fabric). After washing, the fabrics were flat-bed-pressed to even out any crease marks, dried, conditioned, and then the fabric area shrinkage was determined following the standard methodology. To ensure reproducibility, triplicate samples were prepared and the average results are presented.

2.4 | Cuff edge differential shrinkage

The cuff edge differential shrinkage gives the difference in shrinkage between the felting shrinkage at the edge/cuff and the felting shrinkage in either the length or width direction of the fabric. The felted fabric edge differential shrinkage test was performed based on the Woolmark test method (TM 31) for the washing of wool textile products.¹⁷ A cuff was introduced into the SR-treated polymer fabrics (300 \times 300 mm) in

both the warp and weft directions of the sample 50 mm from the edge. The fabric was then steam pressed at 102-108°C for 20 seconds followed by 20 seconds of lock-pressed (steam off) and vacuum application for 10 seconds using an industrial press. A chain stitch was then sewn 30-35 mm from the cuff edge and the fabric was 5 \times 5A washed and dried. The cuff edge differential shrinkage was calculated using Equation 1:

$$\text{The Cuff Edge Differential Shrinkage (\%)} = \text{CS} - \text{WS} \text{ or } \text{CS} - \text{LS}, \quad (1)$$

where CS is the cuff shrinkage (%) = $[(\text{FCM} - \text{RCM}) / \text{RCM}] \times 100$, FCM = felted cuff measurement, RCM = relaxed cuff measurement, WS = mean width dimensional change (%), and LS = mean length dimensional change (%).

2.5 | Preparation of durable press-treated wool fabrics

The prepolymer and hydroxypropyl (HP)- β -cyclodextrin-treated and dried fabrics were incubated for 12 and 2 days at 40 and 60°C, respectively, prior to converting into a trouser leg following TM 31.¹⁷ The trouser leg was steam pressed at 102-108°C for 20 seconds under mechanically applied pressure, followed by 20 seconds of lock-pressed (steam off) and vacuum application for 10 seconds using an industrial press (Fairhaven Machinery, Model K1-COS), with the seams central. The samples were then washed (3 \times 5A wash cycles) using the Wascator FOM 71P machine, and after washing the creases in the fabric were smoothed out by hand prior to tumble-drying. The samples were then conditioned at 20°C before visual assessment of the leg crease and measurement of the crease angle.

2.6 | Visual assessment of leg crease

The conditioned trouser leg was cut open along one of the seams and the open leg was gently laid flat in a standard light cabinet. Two assessors visually assessed each side of the leg pleat, under D65 lighting, against the crease/pleat durability standard IWS photographs (Ichinomiya Technical Centre, 1986).¹⁸ A rating (1-5) was given for each leg pleat of the trouser leg and the result, for each trouser leg, was the average over four readings (Figure 1).

2.7 | Measurement of the crease retention angle

The crease retention angles of the treated fabrics were measured using the equipment developed at University of

Manchester Institute of Science and Technology.¹⁹ Crease angle measurements were performed on fabric strips 4 cm long \times 1.5 cm wide, with the crease positioned centrally across the 4 cm length. One of the 1.5 cm edges was secured onto a polished plate along a 0.5 cm strip parallel to the crease, leaving the remaining 3.5 cm free to move. The free edge of the creased fabric was gently stretched, released and allowed to recover for 10 seconds. The crease angle was then measured by illuminating the sample on one side and measuring the projected crease angle on a screen using a protractor. The measurement was repeated twice for each fabric strip. In addition, for each sample two fabric strips were cut out from each pressed edge of the trouser leg. Thus, the crease set retention angle result was the average over eight measurements.

The set retention (%) is defined as (Equation 2):

$$\text{Set retention (\%)} = [(180 - A) \times 100] / 180, \quad (2)$$

where A is the average crease retention angle (Figure 2).²⁰

2.8 | Colour measurement

The reflectance values of the fabrics were measured using a Datacolor Spectraflash (SF600⁺) spectrophotometer under illuminant D65 with the specular component excluded, UV component included, using a 10° standard observer and LAV aperture. The fabric sample was folded into four layers to ensure opacity; the K/S , CIEL* a^* b^* (Lightness value, L^* ; redness-greenness, a^* ; yellowness-blueness, b^*), $Chroma$, C^* , lightness difference, DL^* and colour difference (ΔE_{cmc}) values presented are each the average of four measurements.

3 | RESULTS AND DISCUSSION

The felting area shrinkages of the untreated fabric, Synthappret BAP- and EC 1364-treated wool fabrics are shown in Table 1, and these were used as “benchmark”

standards for subsequent delay-curing studies. The area shrinkage values of the treated and cured wool fabric were consistent with previous research work and clearly indicate the improved reduction in area shrinkage of the polymer-treated fabrics relative to the untreated wool fabric.^{21,22} It was also apparent that if the SR finishes were washed off prior to heat curing of the prepolymers then no anti-felting effect was imparted to the wool fabrics.

During washing, the folded edge of a woven garment suffers more abrasion and mechanical action and thus felts more rapidly than the body of the fabric. This may lead to tightening of the edge and eventually puckering.¹⁷ It was therefore important to determine the performance of the SR polymers with respect to the felted cuff differential (FCD) shrinkage. Table 1 also shows the FCD shrinkage of Synthappret BAP- and EC 1364-treated wool fabrics and it is apparent that both SR polymer-treated wool fabric samples satisfied the standard requirement of $\leq 1\%$ shrinkage for machine washability.²³

As identified earlier, a technical processing deficiency of the typical SR polymers applied to wool fabric is their relative thermal sensitivity and associated rapid curing on the wool fabric at an ambient temperature. Table 2 highlights the effect of elevated temperatures in initiating polymerisation of the SR-treated, dried and incubated wool fabrics, and this is evidenced by the reduction in area shrinkage of the 5 \times 5A washed wool fabrics. At 40°C there is a progressive reduction in shrinkage over the 12 days' incubation but at 60 and 80°C the SR-treated (pad-dry-incubate) wool fabrics became fully shrink-resistant after 2 days' thermal exposure. This rapid and extensive polymerisation of the SR polymer on the fabric highlights the technical challenge for any late-stage garment processing of the pretreated fabrics based on the reactivity of the prepolymers.

Although the majority of the cyclodextrin literature focuses on smell/fragrance and healthcare applications, it has also been reported that cyclodextrins can offer protection against heat and light,¹²⁻¹⁶ although this effect has been little explored, particularly in the textiles sector. In this study, a range of cyclodextrins was incorporated into the typical Synthappret BAP formulation at varying levels with a view

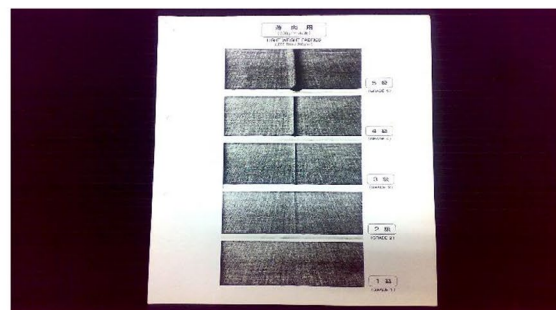
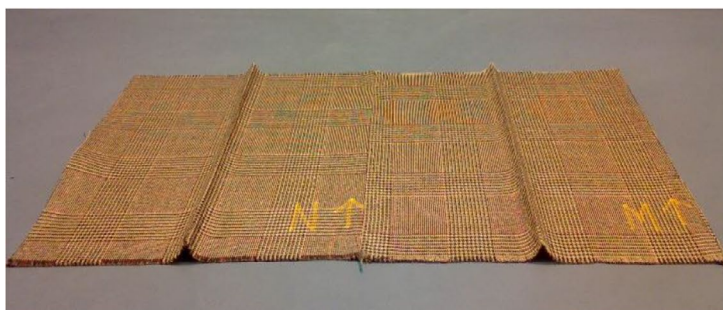


FIGURE 1 Trousler leg opened along seam in order to visually assess crease retention and graded against IWS crease/pleat durability standard photographs (Ichinomiya Technical Centre, 1986)

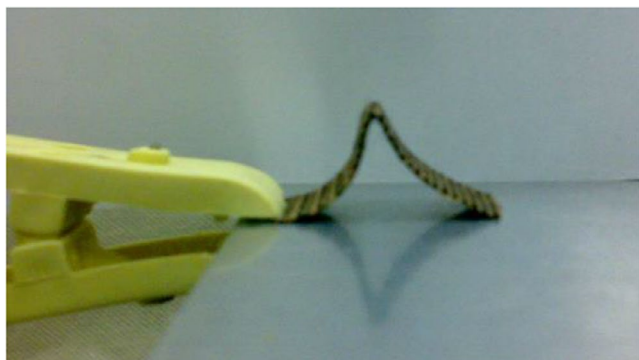


FIGURE 2 Measurement of crease angle on fabric strips

TABLE 1 Felting area shrinkage and felted cuff edge differential (FCD) shrinkage of untreated wool fabric, Synthappret BAP-treated (40 g/L) wool fabric and EC 1364-treated (80 g/L) wool fabric after ISO 6330:1984 laundering, 5 × 5A wash cycles

Sample treatment	Polymer application level			
	40 g/L Synthappret BAP		80 g/L EC 1364	
	Area shrinkage (%)		Area shrinkage (%)	
Untreated	28.5		28.5	
Pad/dry	31.0		33.5	
Pad/dry/heat cure	-1.5 ^a		-1.0	
FCD shrinkage (%)	Weft	Warp	Weft	Warp
	0.5	0.0	-0.5	0.5

^aNegative value means dimensional expansion.

to establishing any beneficial thermal protective effects. From the data, it was apparent that the type of cyclodextrin incorporated into the SR formulation had a significant effect on the extent of any reduction in thermal curing (Tables 3 and 4). This effect is probably related to the cavity size, cyclodextrin polarity and the ability to “host” and “stabilise” the reactive carbamoyl sulphonate functionality. For the unmodified β -cyclodextrin molecule some beneficial effect was observed over 2 days at 40°C but the effect was not durable to 6 days at elevated temperature, as evidenced by the reduced shrinkage for the pad/dry/incubate/washed-off fabrics (Table 3).

By contrast, the HP- β -cyclodextrin/Synthappret BAP and methylated (M)- β -cyclodextrin/Synthappret BAP formulations were effective at 15–20 g/L in providing remarkable delayed-curing protection to the prepolymer for up to 20 days at 40°C (Table 3). Further examination of the thermal stability of the cyclodextrin/Synthappret BAP formulations over 6 days at 60°C indicated the relative reduction in thermal

TABLE 2 Felting area shrinkage of Synthappret BAP-treated (40 g/L) incubated wool fabrics, with and without thermal curing, after ISO 6330:1984 laundering, 5 × 5A wash cycles

Incubation conditions	Area shrinkage (%) of SR polymer-treated, incubated wool fabric after 5 × 5A wash cycles	
	IW ^a	ICW ^b
2 d, 40°C	23.0	-2.0
6 d, 40°C	21.0	-3.5
12 d, 40°C	17.0	-1.5
2 d, 60°C	-2.0	-2.5
6 d, 60°C	-1.5	-3.0
2 d, 80°C	-2.5	-2.0
6 d, 80°C	-2.0	-2.5

Note: Untreated fabric felting shrinkage *ca.* 28.5%.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

protection for the (M)- β -cyclodextrin/Synthappret BAP formulation in comparison with the 20 g/L HP- β -cyclodextrin/Synthappret BAP formulation, which still provided a viable delay-cured effect (Table 4). Subsequent incubating for 12 days at 60°C resulted in heat curing of the Synthappret BAP polymer and reduced felting shrinkage. However, examination of the 40 g/L HP- β -cyclodextrin/Synthappret BAP formulation under 40, 60 and 80°C incubation conditions indicated that relatively high levels of HP- β -cyclodextrin in the Synthappret BAP formulation could inhibit a full curing of the polymer. Therefore, it is apparent that the working ratio of HP- β -cyclodextrin to Synthappret BAP on the fabric needs to be optimised to deliver effective delay-curing without inhibiting the final full cure during the subsequent 150°C baking process.

Examination of the behaviour of the (M)- β -cyclodextrin/Synthappret BAP formulations indicated that the M- β -cyclodextrin derivative offers a similar delay-curing performance to HP- β -cyclodextrin over 20 days incubating at 40°C with 15–20 g/L of M- β -cyclodextrin incorporated into the Synthappret BAP formulation (Table 3). However, at both 60 and 80°C, M- β -cyclodextrin was not as effective as the 20 g/L HP- β -cyclodextrin/Synthappret BAP formulation, probably due to the relatively lower stability of the host complex (Table 4). To assess the delay-curing potential of the cyclodextrin interaction with commercially available shrinkproofing polymers, a range of cyclodextrins was incorporated into the EC 1364 (Bunte salt terminated prepolymer), Protolan 94612 (modified polyisocyanate terminated prepolymer) and Securlana K (tri-functional polyether-based tris(sulphothiolglycolate)) formulations at similar levels evaluated in the Synthappret BAP studies.

TABLE 3 Felting area shrinkage of Synthappret BAP- (40 g/L) and β -cyclodextrin derivative-treated wool fabrics after incubation at 40°C, with and without thermal curing, after ISO 6330:1984 laundering, 5 \times 5A wash cycles

Synthappret BAP + β -cyclodextrin derivative addition treatment		Area shrinkage (%) of treated wool fabric incubated at 40°C after 5 \times 5A wash cycles							
		IW ^a		ICW ^b		IW		ICW	
		2 d	2 d	6 d	6 d	12 d	12 d	20 d	20 d
β -cyclodextrin	20 g/L	28.0	-1.5	6.5	-1.5	1.0	-0.5	—	—
	40 g/L	26.5	-1.5	1.5	-2.5	0.5	0.5	—	—
M- β -cyclodextrin	10 g/L	—	—	25.5	-2.0	29.5	0.0	23.0	-2.0
	15 g/L	—	—	27.0	-2.0	28.0	-2.0	28.5	-1.5
	20 g/L	29.0	-2.5	30.0	-1.5	27.5	-1.0	28.0	-1.0
	40 g/L	29.5	2.0	32.0	0.0	29.0	2.5	—	—
HP- β -cyclodextrin	10 g/L	—	—	29.0	-2.0	28.5	0.0	27.5	-3.5
	15 g/L	—	—	29.0	-1.0	31.0	0.0	31.5	-2.0
	20 g/L	31.0	-0.5	31.0	0.0	29.0	-1.0	29.5	-1.0
	40 g/L	31.0	7.5	30.5	7.5	29.5	4.5	—	—

Note: Untreated fabric felting shrinkage *ca.* 28.5%.

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

TABLE 4 Felting area shrinkage of Synthappret BAP- (40 g/L) and β -cyclodextrin derivatives-treated wool fabrics after incubation at 60 and 80°C, respectively, with and without thermal curing, after ISO 6330:1984 laundering, 5 \times 5A wash cycles

Synthappret BAP + β -cyclodextrin derivatives addition treatment		Area shrinkage (%) of treated wool fabric after 5 \times 5A wash cycles									
		Incubated at 60°C					Incubated at 80°C				
		IW ^a	ICW ^b	IW	ICW	IW	ICW	IW	ICW	IW	ICW
		2 d		6 d		12 d		2 d		6 d	
β -cyclodextrin	20 g/L	-3.0	—	-3.0	—	—	—	-2.5	—	-0.5	—
	40 g/L	1.5	—	-2.0	—	—	—	-1.0	—	0.5	—
M- β -cyclodextrin	20 g/L	22.0	-2.5	19.0	-4.5	—	—	-1.5	—	0.0	—
	40 g/L	28.0	5.5	24.0	4.0	—	—	0.0	—	3.0	—
HP- β -cyclodextrin	10 g/L	0.0	-1.5	0.0	-2.0	1.0	1.0	—	—	—	—
	15 g/L	14.0	-0.5	1.5	-1.0	1.0	5.5	—	—	—	—
	20 g/L	28.0	-1.5	29.0	-2.0	1.5	4.5	5.5	-1.5	7.0	-3.0
	40 g/L	29.0	10.5	31.5	10.0	—	—	17.0	11.0	14.0	10.5

Note: Untreated fabric felting shrinkage *ca.* 28.5%.

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

For brevity, only the EC 1364 data are presented, but similar results were obtained with the Protolan 94612 and Securlana K finishes.

Table 5 highlights the effect of elevated temperatures in initiating polymerisation of the SR-treated, dried and incubated wool fabrics, and this is evidenced by the reduction in area shrinkage of the 5 \times 5A washed wool fabrics. At 40°C there is a progressive reduction in shrinkage over 12 days'

incubation, but at 60 and 80°C the SR-treated (pad-dry-incubate) wool fabrics became fully shrink-resistant after 2 days of thermal exposure.

The effect of incorporating β -cyclodextrin, M- β -cyclodextrin and HP- β -cyclodextrin into the EC 1364 formulation was to introduce an obvious beneficial increase in thermal stability during the thermal incubation at 40°C at all application levels (Table 6). Even with the unmodified

β -cyclodextrin, there was significant prepolymer reaction inhibition up to 20 days, which perhaps reflects the better host/complex formation with the EC 1364 Bunte salt terminated finish relative to Synthappret BAP. This enhanced stabilising effect observed with the unmodified β -cyclodextrin is also evident at 60°C, where significant thermal protection is offered (Table 7). However, at 80°C, as expected no benefit was provided by β -cyclodextrin. Similarly, in evaluating the M- β -cyclodextrin and HP- β -cyclodextrin derivatives in the EC 1364 formulation at 40 and 60°C, it is clear that both were effective in offering thermal stability at 10–20 g/L at 40°C,

TABLE 5 Felting area shrinkage of EC 1364-treated (80 g/L) incubated wool fabric, with and without thermal curing, after ISO 6330:1984 laundering, 5 × 5A wash cycles

Incubation treatment	Area shrinkage (%) of SR polymer-treated and incubated wool fabric	
	IW ^a	ICW ^b
2 d, 40°C	22.0	−1.5
6 d, 40°C	9.0	−4.5
12 d, 40°C	10.0	−3.5
2 d, 60°C	−2.5	−2.0
6 d, 60°C	−3.5	−3.5
2 d, 80°C	−3.5	−4.5
6 d, 80°C	−4.5	−5.5

Note: Untreated fabric felting shrinkage is ca. 28.5%.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

but only HP- β -cyclodextrin delivered thermal inhibition at 60°C. While this protection at 60°C is remarkable it does not extend to 80°C. In addition, the effect of “overloading” the EC 1364 formulation was evident with the addition of 40 g/L HP- β -cyclodextrin, where subsequent full curing of the SR finish was not possible. Therefore, for the subsequent felting and crease-durability studies the level of HP- β -cyclodextrin in the Synthappret BAP and EC 1364 formulations was optimised at 15–20 g/L.

The durable press properties of SR-treated wool fabrics were rated subjectively and were also evaluated by the objective measurement of the crease retention angle (Table 8).

A high level of set retention was observed for all three Synthappret BAP control fabrics that had been steam pressed, cured, washed (3 × 5A) and tumble-dried prior to evaluation, and the durable press performance of the Synthappret BAP-treated creased fabrics were assigned a maximum rating of 5. The level of set retention did not increase significantly with increasing levels of Synthappret BAP prepolymer (40–70 g/L), which reflects the inherent “crispness” of the treated wool fabrics.

The EC 1364 polymer is known to offer a softer handle to SR-treated wool fabrics than the Synthappret BAP. This lower rigidity effect is reflected in the higher levels of the EC 1364 formulation that needed to be applied to the wool fabric to achieve machine washability and to impart acceptable durable press performance (Table 8). According to Woolmark test methods TM31/281,²³ the pass level for after-wash appearance for crease retention of worsted garments is 3 for minimum iron and 4 for non-iron finish.

TABLE 6 Felting area shrinkage of EC 1364- (80 g/L) and β -cyclodextrin derivatives-treated wool fabrics after incubation at 40°C, with and without thermal curing, after ISO 6330:1984 laundering, 5 × 5A wash cycles

EC 1364 + β -cyclodextrin derivative addition treatment		Area shrinkage (%) of treated wool fabric incubated at 40°C after 5 × 5A wash cycles							
		IW ^a		ICW ^b		IW		ICW	
		2 d		6 d		12 d		20 d	
β -cyclodextrin	20 g/L	32.0	−3.5	29.0	−2.0	30.0	1.0	19.0	−1.0
	40 g/L	32.5	−1.0	27.0	−1.5	31.0	0.0	21.0	1.0
M- β -cyclodextrin	10 g/L	—	—	26.5	−3.5	30.0	−1.5	34.0	−2.0
	15 g/L	—	—	26.0	−1.5	32.5	0.0	32.5	−3.0
	20 g/L	31.0	−3.0	29.0	−2.5	29.0	−1.0	34.5	−1.5
	40 g/L	31.5	−1.5	30.5	−3.5	34.0	6.0	—	—
HP- β -cyclodextrin	10 g/L	—	—	26.0	−3.5	27.0	−1.0	35.5	−2.0
	15 g/L	—	—	27.5	−2.5	31.5	−1.5	33.0	−2.0
	20 g/L	31.5	−1.5	32.0	−3.5	34.0	0.5	35.0	−1.5
	40 g/L	32.0	4.5	29.5	7.5	31.5	5.5	—	—

Note: Untreated fabric felting shrinkage ca. 28.5%.

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

TABLE 7 Felting area shrinkage of EC 1364 (80 g/L) and β -cyclodextrin derivatives-treated wool fabrics after incubation at 60 and 80°C, respectively, with and without thermal curing, after ISO 6330:1984 laundering, 5 \times 5A wash cycles

EC 1364 + β -cyclodextrin derivatives addition treatment		Area shrinkage (%) of treated wool fabric after 5 \times 5A wash cycles									
		Incubated at 60°C					Incubated at 80°C				
		IW ^a	ICW ^b	IW	ICW	IW	ICW	IW	ICW	IW	ICW
		2 d		6 d		12 d		2 d		6 d	
β -cyclodextrin	20 g/L	29.0	-1.5	27.5	-3.0	19.0	-1.0	0.0	-2.0	5.0	4.5
	40 g/L	25.0	0.0	25.5	1.5	—	—	1.0	1.0	7.5	7.0
M- β -cyclodextrin	20 g/L	28.0	-1.0	28.5	-2.0	23.0	-1.0	7.0	-1.5	-1.5	-1.0
	40 g/L	28.5	-1.0	30.5	2.0	—	—	23.5	15.0	18.5	23.0
HP- β -cyclodextrin	10 g/L	28.0	-1.0	29.5	-3.0	31.5	-1.0	—	—	—	—
	15 g/L	27.0	-1.5	29.5	-4.0	33.0	-1.5	—	—	—	—
	20 g/L	30.0	-0.5	30.0	-2.0	32.5	-3.0	15.0	-0.5	14.5	2.0
	40 g/L	26.5	13.5	30.5	12.0	—	—	29.5	6.0	26.5	19.5

Note: Untreated fabric felting shrinkage *ca.* 28.5%.

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

^aIW – Pad/dry/incubate/wash-off.

^bICW – Pad/dry/incubate/heat cure/wash-off.

TABLE 8 Crease retention properties of Synthappret BAP (BAP)- and EC 1364-treated, pressed, cured, 3 \times 5A washed and tumble-dried wool fabric

Sample treatment (control)	Mean value of crease retention angle (°)	Set retention (%)	Subjective rating ^a (1-5)
40 g/L BAP	34	81	5
55 g/L BAP	32	82	5
70 g/L BAP	33	82	5
80 g/L EC 1364	52	71	4-5
100 g/L EC 1364	63	65	4
120 g/L EC 1364	65	64	4

^aBased on assessment using IWS crease/pleat durability standard photographs, 1986.

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

Table 9 shows the average crease retention angle and the percentage set retention of industrially pressed and cured wool fabric samples that had been SR-treated, with and without HP- β -cyclodextrin derivative, incubated at 60°C for 2 days, washed (3 \times 5A) and tumble-dried. For the set of samples treated without the HP- β -cyclodextrin derivative, the set retention decreased drastically with respect to the control samples, from a rating of 5 to 2/2-3. However, it is apparent that incorporating the HP- β -cyclodextrin derivative into the Synthappret BAP formulation improved the level of set retention, and the improvement was most significant with 55 g/L of SR polymer and 28 g/L of HP- β -cyclodextrin derivative.

Examination of the EC 1364 prepolymer data indicated that, in the absence of HP- β -cyclodextrin derivative, the set retention following washing and tumble-drying was low, 25% at the recommended level of 80 g/L, but improved

up to 50% with the 120 g/L of EC 1364 prepolymer application level (Table 9). Nevertheless, the treatment performances were still below the requisite “pass” level and suggested that EC 1364 polymerised extensively (100% shrink resistance) on the flat wool fabric during thermal incubation at 60°C over 2 days prior to hot pressing the creases. By contrast, for the combined HP- β -cyclodextrin/EC 1364 formulations, the treated wool fabrics showed a significant improvement in set retention from 25%-27% to 71%-72% with 80-100 g/L of the polymer and the corresponding 20-25 g/L of HP- β -cyclodextrin derivative. Therefore, the observed visual ratings of 4-5 demonstrated a commercially acceptable level of crease retention linked to machine washability.

An important effect of any chemical finish on a substrate is its influence on the final colour. The colour difference, ΔE_{CMC} , between the SR prepolymer wool fabrics

Sample treatment	Mean value of crease retention angle (°)	Set retention (%)	Subjective rating (1-5)
40 g/L BAP	94	48	2
55 g/L BAP	92	49	2
70 g/L BAP	83	54	2-3
40 g/L BAP + 20 g/L HP-CD	67	63	3-4
55 g/L BAP + 28 g/L HP-CD	50	72	4-5
70 g/L BAP + 35 g/L HP-CD	57	68	4
80 g/L EC 1364	134	25	1-2
100 g/L EC 1364	131	27	1-2
120 g/L EC 1364	90	50	2-3
80 g/L EC 1364 + 20 g/L HP-CD	52	71	4-5
100 g/L EC 1364 + 25 g/L HP-CD	50	72	4-5
120 g/L EC 1364 + 30 g/L HP-CD	52	71	4-5

Bold values have been used to draw the reader's attention to the duration over which the delayed-cure process is effective under the respective application conditions.

Fabric treatment		Colour parameters					
		L^*	a^*	b^*	C^*	DL^*	ΔE_{CMC}
Unwashed fabrics	Synthappret BAP, 40 g/L	13.2	-0.1	-0.3	0.3	0.5	0.5
	Synthappret BAP, 40 g/L + 20 g/L HP-CD	13.7	-0.1	-0.3	0.3		
Fabrics washed x1	Synthappret BAP, 40 g/L	13.5	-0.1	-0.3	0.3	0.4	0.4
	Synthappret BAP, 40 g/L + 20 g/L HP-CD	13.9	-0.1	-0.4	0.4		
Unwashed fabrics	EC 1364, 80 g/L	13.4	-0.1	-0.3	0.3	0.6	0.6
	EC 1364, 80 g/L + HP-CD, 20 g/L	14.0	-0.1	-0.4	0.4		
Fabrics washed x1	EC 1364, 80 g/L	13.7	-0.1	-0.4	0.4	0.5	0.5
	EC 1364, 80 g/L + HP-CD, 20 g/L	14.3	-0.1	-0.4	0.4		

L^* : lightness, a^* : redness-greenness, b^* : yellowness-blueness, C^* : chroma, DL^* : difference in lightness.

treated both with and without HP- β -cyclodextrin derivative, was well within the commercially acceptable visual limit of $\Delta E_{CMC} \leq 1.0$ (Table 10). This was true for both sets of unwashed and washed samples (1 \times CO6 C2S), with ΔE_{CMC} being marginally less for the washed fabrics.

4 | CONCLUSIONS

The incorporation of β -cyclodextrin or the HP- β -cyclodextrin and the M- β -cyclodextrin derivatives into commercial shrinkproofing prepolymer formulations for wool fabric has potential as a commercial, delayed-cure, durable press, machine-washable wool technology. The HP- β -cyclodextrin derivative was found to be the most effective of the

TABLE 9 Crease retention properties of Synthappret BAP (BAP)- and EC 1364-treated with and without HP- β -cyclodextrin derivative (HP-CD), incubated (2 days at 60°C), pressed, cured, 3 \times 5A washed and tumble-dried wool fabric

TABLE 10 Colour analysis of black woven wool fabric treated with Synthappret BAP and EC 1364 formulations, with and without HP- β -cyclodextrin

cyclodextrins evaluated in inhibiting the thermal curing of SR polymers on wool fabrics at 40 and 60°C. Following incubation at 40 and 60°C, the SR prepolymer formulation could be fully heat-cured and impart commercial machine washability, provided an excess of cyclodextrin was not present in the SR formulation.

The incorporation of HP- β -cyclodextrin into the Synthappret BAP and EC 1354 formulations inhibited the thermal curing of the “flat” treated fabrics at 40 and 60°C and allowed the introduction of creases into the fabrics, post-incubation, which were durable to 3 \times 5A washing and tumble-drying.

Colour is an important performance criterion for textile products and it is vital that chemical finishes have no deleterious effect on the product appearance. In this study, it was

demonstrated that the addition of HP- β -cyclodextrin into the Synthappret BAP and EC 1354 formulations had an instrumentally detectable effect on the colour of the final dyed SR-treated fabric, but that this difference was less than one ΔE_{CMC} unit.

In conclusion, this study presents for the first time a viable route to delayed-cure, durable press, machine-washable, wool fabric processing. However, in any commercial process, the benefits and additional process flexibility need to be justified against any extra costs, and in this case the addition of cyclodextrin into the shrinkproofing polymer formulation will typically add an extra 20%-25% to the overall cost.

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