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Union disperse printing and UV-protecting of wool/polyester blend using a reactive β-cyclodextrin

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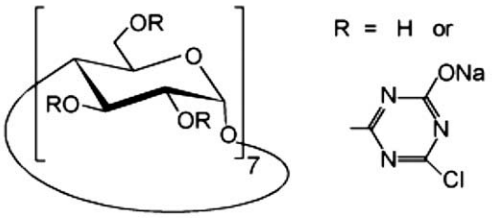
ABSTRACT

Pretreatment of wool/polyester blend fabric with monochlorotriazinyl β-CD was carried out for modifying the wool component to be able to form "host–guest" inclusion complexes with disperse dyes during the subsequent disperse printing step thereby leading to union disperse printing. The optimum sequence/conditions of treatment based on the data obtained were: padding of the blend fabric with an aqueous formulation composed of monochlorotriazinyl β-CD (60 g/L), Fixapret® ECO (20 g/L), citric acid (5 g/L), PEG-600 (10 g/L), wet-pickup (70%), thermofixing at 120 °C/5 min, thoroughly washing, drying, followed by post-printing with disperse dyes and finally steaming at 140 °C for 30 min. Our experimental results reveal that fixing of the used monochlorotriazinyl β-CD onto and/or within the wool component has modified its structure thereby increasing its ability to pick-up, adsorb as well as to retain the guest disperse dye vapors into its grafted hydrophobic cavities, which in turn resulted in attaining union-disperse printing with deeper shades and remarkable fastness properties. As a result of inclusions, the obtained prints exhibit excellent UV-protecting functions. The surface morphology has been studied using SEM micrographs.

1. Introduction

Blending of man-made fibres, e.g. polyester, polyamide… etc., with natural fibres, e.g. wool, cotton, etc., offers the most valuable options for economical reasons as well as for combining desirable physico–chemical properties contributed by both the fibre components. Wool/polyester blends have gained a considerable share in woven suiting. Because of the vastly different physico-chemical nature of the two components, two classes of dyes are used for coloration. (Chao & Lin, 1998; 2000; Choudhury, 2006).

Cyclodextrins are cyclic oligosaccharides of three types namely α-cyclodextrin, β-cyclodextrin and γ-cyclodextrin, which contain six, seven, and eight monosaccharide moieties, respectively. The toroidal shape of cyclodextrin molecules, with hydrophilic outside along with the presence of internal hydrophobic hollow cavities enable these hosting species to form stable inclusion complexes with a wide variety of hydrophobic guest molecules. (Buschmann, Denter, Knittel, & Schollmeyer, 1998; Szejtli, 2003; Valle, 2004).



Chemical structure of reactive β-cyclodextrin

On the other hand, considerable R&D efforts have been carried out to obtain colored textile materials with: better economics, higher quality, improved performance and fastness properties, reasonable and competitive prices along with more rapid delivery and quick response, taking in consideration both the end-user and the ecological demands. (Dawson & Hawkyard, 2000; Holme, 2003).

Accordingly, the present work is directed towards pre-modification of the wool component of wool/polyester blend fabric using monochlorotriazinyl β-cyclodextrin for attaining high performance union disperse prints with outstanding UV-protection functions.

2. Experimental

2.1. Materials

Mill-scoured and bleached wool/polyester blended woven fabric (50/50, 235 g/m^2) was used.

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Cavasol® W7MCT (monochlorotriazinyl β-cyclodextrin (MCT-β-CD) average molecular weight ~1560, degree of substitution 0.3–0.6 per anhydroglucose unit, Wacker, Germany), Tylose® C30 (carboxy methyl cellulose ether, low viscosity Clariant), and Fixapret® ECO (modified dimethylol drihydroxy ethylene urea-DMDHEU-BASF) were of technical grade.

Commercial disperse dyes namely: Disperse Red 54 (100%-Sinochem, China) Disperse Red 167 (150%-Sinochem, China), Disperse blue 183 (100%-Sinochem, China) as well as Samaron® Red HBL 150 (Dystar) were used.

Polyethylene glycol (PEG-600), citric acid, ammonium chloride and acetic acid were of laboratory reagent grade.

2.2. Methods

2.2.1. Pretreatment with MC-BCD

Fabric samples were impregnated by the aqueous solution containing the MCT-β-CD (0–80 g/L), DMDHEU (20 g/L), PEG-600 (0–30 g/L), and catalyst (0–7 g/L), roll squeezed, and thermofixed at 100–130 °C for 5 min, thoroughly washed to remove unreacted and any soluble byproducts and finally dried before being printed.

2.2.2. Disperse printing

Portions of untreated and MCT-β-CD loaded fabric samples were disperse printed using the flat screen technique and the following print paste formulation:

Constituent	g/kg paste
Disperse dye	30
Stock thickening (5%)	700
Acetic acid (30%)	20
Water	250
Total	1000 g

Printed fabric samples were then dried at 85 °C for 5 min and steam fixed at 140 °C for 30 min using Ariolt® CSL-steamer-Italy, rinsed thoroughly, soaped 15 min at 50 °C in the presence of 2 g/L Leomin® W (nonionic wetting agent and detergent-BASF), then rinsed well and finally dried at 85 °C for 5 min.

2.2.3. Testing

Nitrogen content was determined according to the Kjeldahl method. Depth of the obtained prints, expressed as K/S, was measured at the wave length of the maximum absorbance using an automatic filter spectrophotometer, and calculated by the Kubelka Munk equation. (Duff & Sinclair, 1989). Fastness properties to washing perspiration and crocking of printed samples were evaluated according to AATCC test methods: (61-1972), (15-1973) and (8-1972), respectively. UV-protection factor (UPF) was calculated according to the Australian/Newzerland standard (AS/NZS 4399-1996). (Ibrahim, Allam, El-Hossany, & El-Zairy, 2007). A scanning electron microscope (SEM) examination was carried out by mounting the untreated and modified fabric samples on sub with double stick adhesive tape and coated with gold in a S150A sputler coater unit (Edwards, UK), the gold film thickness was 150 Å, then viewed in a JEAOL JXA-840A electron probe microanalyses.

3. Results and discussion

In order to attain solid shades on wool/polyester blend fabric using one class of dyestuffs, i.e. disperse dyes, along with high fastness properties, loading of a reactive β-cyclodextrin, i.e. MCT-βCD, as a host molecule with a polar cavity and its ability to form inclusion complexes with several hydrophobic guest molecules such as disperse dyes, onto and/or within the wool component was tried. Results obtained along with their appropriate discussion follow:

3.1. MCT-βCD concentration

For a given set of pretreatment and post-disperse printing conditions, Fig. 1 discloses that increasing MCT-β-CD concentration from 0 up to 60 g/L in the pretreatment bath results in a dramatic improvement in the depth of the obtained prints, expressed as K/S values- from 7.8 up to 19.8. This dramatic increase in K/S values is a direct consequence of modifying the wool component via grafting of MCT-β-CD molecules onto and within its structure thereby enhancing its ability to pick up and include the hydrophobic disperse dye molecules, nonpolar guest molecules, within the internal hydrophobic hollow cavities, hosting species, in loaded MCT-β-CD during the subsequent printing step according to the following scheme of reactions: (Ibrahim et al., 2007; Szejtli, 2003; Abo-El-ola & Ibrahim, 2005).

i- Permanent fixation of MCT-β – CD on and/or into the wool component

$$\text{W-XH} + \text{DMDHEU} + \text{NaOMCT} - \beta - \text{CD} \sim (\text{OH})_n \xrightarrow[\triangle]{\text{H}+} \text{Modified wool structure}(\text{MW} - \beta - \text{CD}) \quad (1)$$

$$\text{W} - \text{NH}_2 + \text{H}^+ \rightleftharpoons \text{Wool} - \text{NH}_3^+ \quad (2)$$

$$\text{W} - \text{NH}_3^+ + \text{O}^- - \text{MCT} - \beta - \text{CD} \sim (\text{OH})_n \rightleftharpoons \text{W} - \overset{+}{\text{N}}\text{H}_3\text{O}^- - \text{R} - \beta\text{CD} \quad \text{MW} - \beta\text{CD} \quad (3)$$

ii- Inclusion of the disperse dye molecules in the inner cavity of the loaded β-CD (Buschmann et al., 1998).

$$\text{MW} - \beta - \text{CD} + \text{disperse dye} \rightleftharpoons \text{Inclusion complex} \quad (4)$$

iii- Side interactions

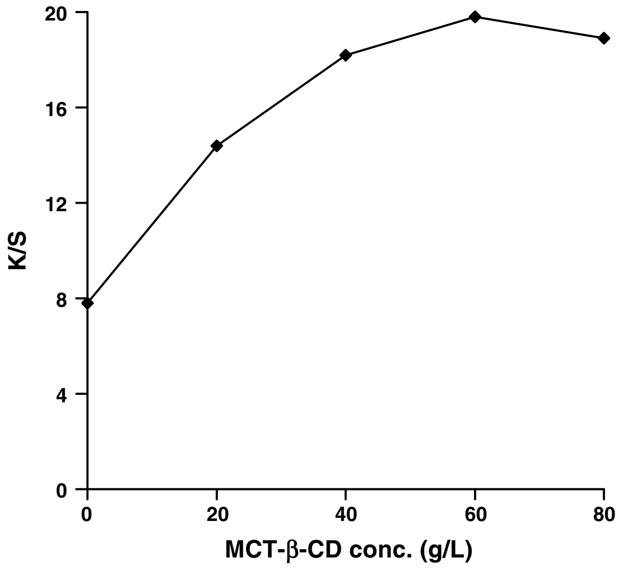


Fig. 1. Effect of pretreatment with different MCT-β-CD concentration on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (0–80 g/L), citric acid (5 g/L), DMDHEU (20 g/L), PEG-600 (10 g/L), padding to a wet pick (70%), followed by direct fixation at 120 °C/5 min. (ii) Post-printing conditions: Disperse red 54 (30 g/kg); stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C/5 min, steaming at 140 °C/30 min.

$$\mathrm{NaO-MCT-\beta-CD \sim (OH)_{\mathit{n}} + DMDHEU \xrightarrow[\triangle]{H+} inter\ and/or\ intra\ crosslinked-\beta-CD} \quad (5)$$

$$\mathrm{NaO-MC-\beta-CD \sim (OH)_{\mathit{n}} + DMDHEU + HO-CA \xrightarrow[\triangle]{H+} MCT-\beta-CD \sim\sim (OH)_{\mathit{n}-1} \sim\sim CA\ adduct} \quad (6)$$

$$\mathrm{W-XH + DMDHEU \xrightarrow[\triangle]{H+} partially\ crosslinked\ wool} \quad (7)$$

$$\mathrm{W-XH + DMDHEU + HO-CA \xrightarrow[\triangle]{H+} partially\ cross\ linked\ wool\ containing\text{-}CA\ moieties} \quad (8)$$

where W-XH = wool component, XH = NH_2, –SH, –OH,
NaO-MCT-β-CD_n $(OH)_n$ = monochlorotriazinyl-β-CD; HO-CA = citric acid.

On one hand, the higher K/S value of the printed sample in the absence of loaded MCT-β-CD reflects the positive impact of the hydrophobic character of polyester component along with its high affinity for disperse dyes. On the other hand, the positive role of PEG on fibre structure as well as the hydrophobic portions of the wool (Chao & Lin, 2000) on enhancing the extent of adsorption and subsequent trapping or fixation can not be ruled out. It is also observed that further increase in MC-β-CD, i.e. beyond 60 g/L, has practically a slight negative effect on the K/S of the obtained prints.

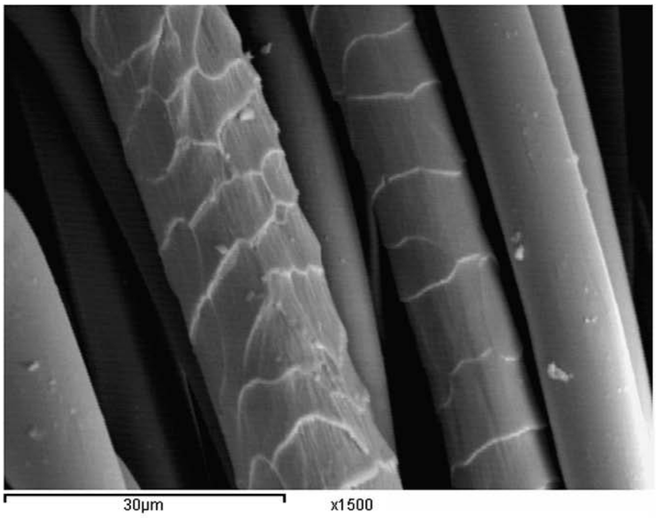
Additionally, the SEM images of the treated and untreated wool/polyester blend samples (Fig. 2) shows the deposition of the MCT-β-CD onto the wool component along with coating of the wool scales compared with the untreated sample.

3.2. Catalyst type and concentration

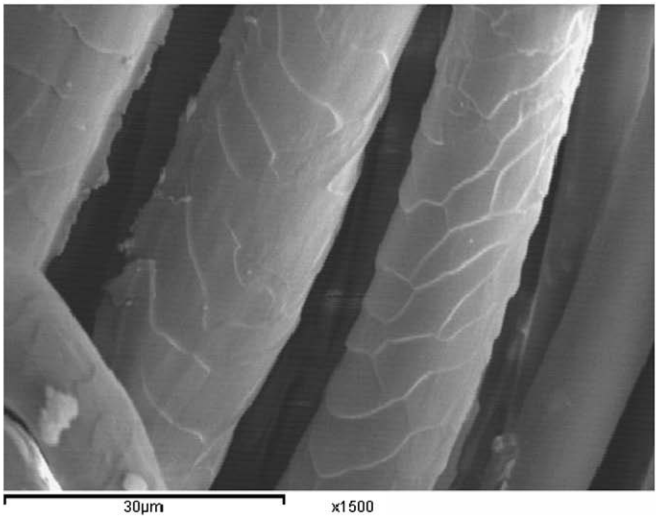
As far as the change in K/S values of the pretreated post-printed fabric samples as a function of type and concentration of the used catalyst, Fig. 3 discloses that: (i) increasing the catalyst concentration from zero up to 5 g/L results in a significant enhancement in the extent of post-disperse printing, regardless of the used catalyst, (ii) this improvement in K/S values is a direct consequence of enhancing the extent of chemical fixation of the MC-β-CD with its hydrophobic cavities, in and/or onto the wool component thereby upgrading its capability to capture and entrap the disperse dye molecules via the formation of inclusion complex, (Buschmann et al., 1998; Szejtli, 2003) (iii) the extent of modification as well as the subsequent disperse printing is governed by the type of the acid-catalyst, i.e. citric acid > ammonium chloride > none, reflects the differences between them: in efficiency of catalysation, rate of dissociation and/or decomposition to yield the hydrogen proton, degree of fixation of the used MC-β-CD, via DMDHEU bridge, on and/or within the wool structure, location and accessibility of the grafted β-CD moieties, with their lipophilic cavities, during the subsequent disperse printing step, along with extent and mode of interaction in addition to stability to after washing and/or steaming conditions, (iv) further increase in catalyst concentration, i.e. beyond 5 g/L, has practically no effect, and (v) the high K/S of pretreated samples, without using catalyst, most probably due to the hydrophobic nature of the polyester component along with the physical entrapment and/or the MCT-β-CD-wool ionic interaction (Eq. (3)).

3.3. Effect of PEG-600 concentration

The variation in the extent of post-disperse printing as a function of PEG-600 concentration used in the pretreatment step of wool/polyester blend fabric is shown in Fig. 4. For a given set of pretreatment and subsequent printing, it is clear that: (i) increasing the PEG-600 concentration up to 10 g/L enhances the extent of post-printing most probably due to the enhancement in wool swellability thereby enabling better penetration and modification of the wool structure by the used MCT-β-CD, i.e. more grafted β-CD moieties capable to capture more disperse dye molecules during the printing step, (ii) further increase in the PEG-600, up to 30 g/L results in a slight decrease in the depth of the obtained disperse prints, may be attributed to the decrease in the extent of fixation of MCT-β-CD onto and/or within the DMDHEU/wool matrix resulting from the terminal-OH groups competition for interaction with the *N*-methylol groups of the DMDHEU according to the following interactions: (Ibrahim, Abo-Shosha, Elnagdy, & Gaffar, 2002)



a. Untreated



b. Treated

Fig. 2. SEM of untreated and treated wool/polyester blend fabric samples with MCT-β-CD.

3.4. Thermo-fixation time

$$\mathrm{PEG \sim\sim OH + DMDHEU + W-XH \xrightarrow[\triangle]{H+} Wool\ structure\ containing\text{-}PEG\ moieties} \quad (9)$$

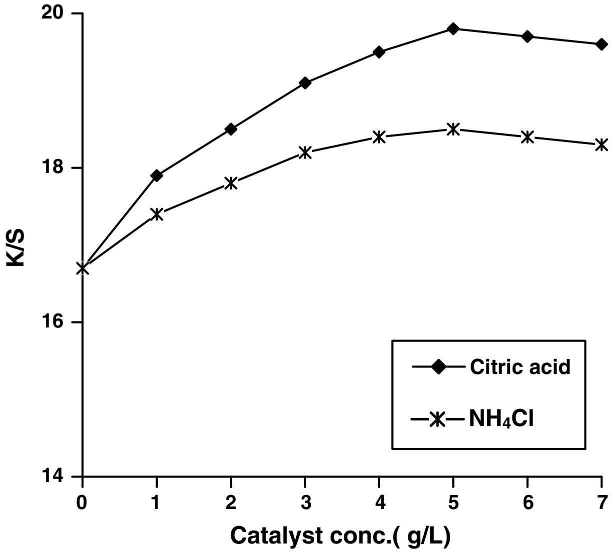


Fig. 3. Effect of type and concentration of the catalyst used in pretreatment step on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (60 g/L), citric acid (0–7 g/L), DMDHEU (20 g/L), PEG-600 (10 g/L), padding to a wet pick (70%), followed by direct fixation at 120 °C/5 min. (ii) Post-printing conditions: Disperse red 54 (30 g/kg), stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C/5 min, steaming at 140 °C/30 min.

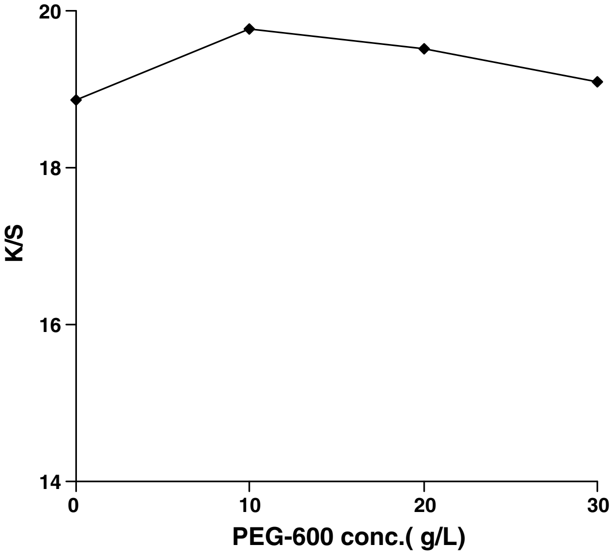


Fig. 4. Effect of PEG-600 concentration used in pretreatment step on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (60 g/L), citric acid (5 g/L), DMDHEU (20 g/L), PEG-600 (0–30 g/L), padding to a wet pick (70%), followed by direct fixation at 120 °C/5 min. (ii) Post-printing conditions: Disperse red 54 (30 g/kg), stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C/5 min, steaming at 140 °C/30 min.

$$\mathrm{PEG}{\sim\!\sim}\mathrm{OH} + \mathrm{DMDHEU} + \mathrm{NaO} - \mathrm{MCT} - \beta\mathrm{C}{\sim\!\sim}(\mathrm{OH})_n \xrightarrow[\Delta]{\mathrm{H}+} \mathrm{MCT} - \beta\mathrm{CD}(\mathrm{OH})_{n-1}{\sim\!\sim}\mathrm{PEG\ adduct} \qquad (10)$$

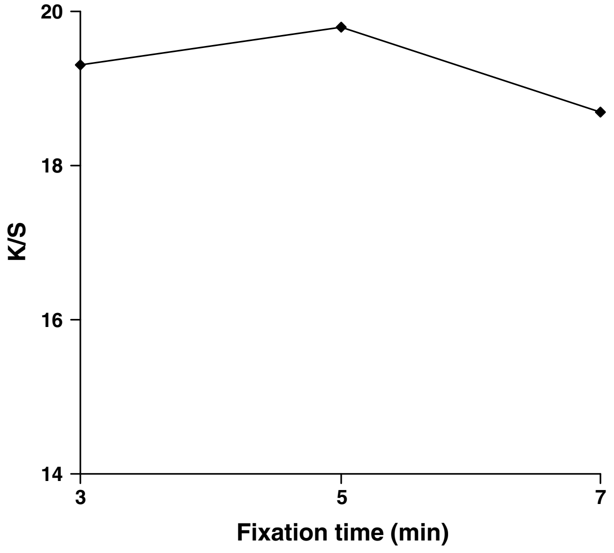


Fig. 5. Effect of thermo-fixation time after pretreatment step on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (60 g/L), citric acid (5 g/L), DMDHEU (20 g/L), PEG-600 (10 g/L), padding to a wet pick (70%), followed by direct fixation at (120 °C)for (3–7 min). (ii) Post-printing conditions: Disperse red 54 (30 g/kg), stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C/5 min, steaming at 140 °C/30 min.

As far as the change in the depth of the obtained disperse prints, expressed as K/S values, as a function of the fixation time of pretreated fabric samples, and for a given set of pretreatment conditions, Fig. 5 discloses that prolonging the fixation time up to 5 min enhances the K/S values which could be discussed in terms of better fixation of the used-MCT-β-CD onto and within the DMDHEU/Wool matrix thereby giving rise up to better dye uptake and fixation, i.e. higher K/S values. On the other hand further increase in fixation time i.e. beyond 5 min, results in a decrease in the extent of post-printing which may be attributed to the higher extent of crosslinking and/or side interactions at longer time thereby giving rise to lower extent of binding and fixing of the used MCT-β-CD on/into the wool structure, i.e. lower the extent of modification which adversely affect the extent of post-disperse printing.

3.5. Steam-fixation temperature

The variation of the K/S values of pretreated → post-printed fabric samples with respect to steam fixation temperature, Fig. 6 shows that: (i) increasing the steaming temperature from 100 °C up to 140 °C for 30 min is accompanied by a gradual sharp increase in the depth of the obtained disperse prints, and (ii) the outstanding improve in K/S value is a direct consequence of: improving the swellability of both the thickener film and the modified blend structure, facilitating the disperse dye release from the thickener film, sublime and diffuse into the accessible and available blend components, i.e. the hydrophobic polyester along with the modified wool component with its immobilized hydrophobic cavities, as well as enhancing the extent of dye retention and fixation thereby giving rise to darker depth of shade (from 0.9 up to 19.76 K/S values). (Choudhury, 2006; Ibrahim et al., 2002).

3.6. Steam-fixation time

Fig. 7 shows the effect of steaming duration on post-disperse printing of wool/polyester blend fabric anchored MCT-β-CD. For a given set of modification and subsequent printing step condition, it is clear that: (i) prolonging the steaming time up to 30 min at 140 °C results in a remarkable increase in the depth of the obtained disperse prints (from K/S = 14.8 up to 19.76), and (ii) this outstanding increase in the K/S value reflects the positive role of proper steaming time on: swelling the thickening film, enhancing the extent of release of dye molecules from the thickener film as well as dye sublimation, adsorption onto and diffusion within the

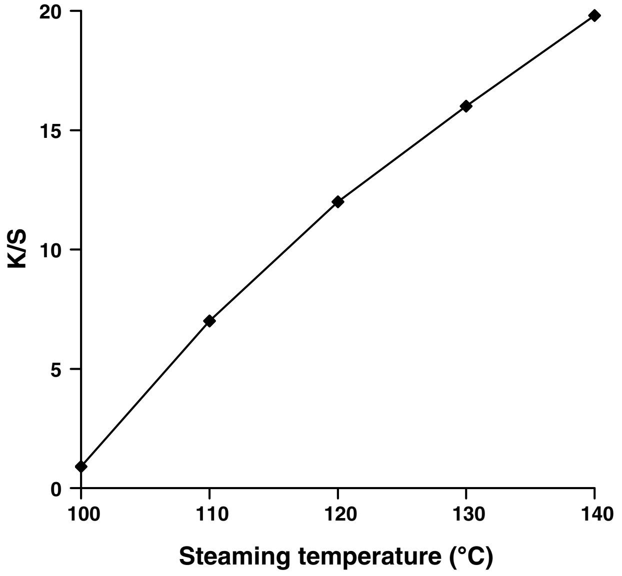


Fig. 6. Effect of steaming temperature on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (60 g/L); citric acid (5 g/L), DMDHEU (20 g/L), PEG-600 (10 g/L), padding to a wet pick (70%), followed by direct fixation at (120 °C)for 5 min. (ii) Post-printing conditions: Disperse red 54 (30 g/kg), stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C/5 min, steaming at (100–140 °C) for 30 min.

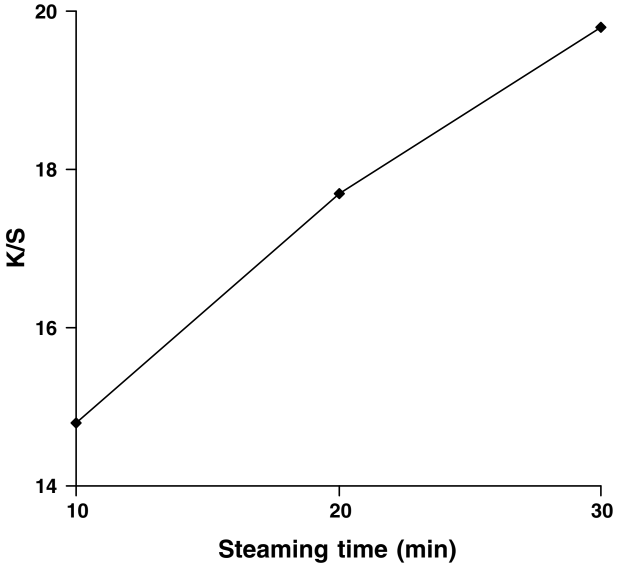


Fig. 7. Effect of steaming time on the K/S values of the obtained disperse prints. (i) Pretreatment conditions: MCT-β-CD (60 g/L), citric acid (5 g/L), DMDHEU (20 g/L), PEG-600 (10 g/L), padding to a wet pick (70%), followed by direct fixation at (120 °C)for 5 min. (ii) Post-printing conditions: Disperse red 54 (30 g/kg), stock-thickening agent—5% (700 g/kg), acetic acid—30% (20 g/kg), drying at 85 °C /5 min, steaming at140 °C for (10–30 min).

modified substrate thereby enabling the volatile and dye-vapor to be strongly adsorbed and retained by the hydrophobic polyester component along with to get into hydrophobic cavities of fixed MCT-β-CD and form inclusions. (Aspland, 1997; Choudhury, 2006; Ibrahim, Abo-Shosha, Allam, & El-Zairy, 2008).

3.7. Printing and UV-protecting properties

As far the variation in the nitrogen content (%N), the depth of the obtained prints (K/S), fastness properties namely washing (WF) rubbing (RF), perspiration (PF), handle as well as UV-protecting properties of the non-modified (NM-only printed) and the modified (M-pretreated with MCT-β-CD followed by disperse printing), as a function of the disperse dye, the data in Table 1 signify that: (i) the increase in the %N follows the decreasing order: M > NM > untreated, (ii) pre-modification, with MCT-β-CD, results in a remarkable increase in the K/S values as well as a significant improvement in the evaluated fastness properties of the obtained disperse prints along with union solid shades, irrespective of the used disperse dyes, (iii) the extent of improvement in the aforementioned properties is governed by the type of disperse dye, i.e. molecular size, chemical structure, functionalities, extent of release from the thickener film and sublimation, thermal stability, degree of penetration or diffusion coefficient along with mode of interaction, fixation and retention within the modified blend components, via hydrogen-bonding, Van der Waal's forces, dipole interactions and/or formation of inclusion compounds with the grafted MCT-β-CD on modified fabric samples (Aspland, 1997; Buschmann et al., 1998; Choudhury, 2006; Szejtli, 2003), (iv) pre-modification step has practically no harsh effect on the obtained prints-handle, (v) the outstanding UV-protection properties of the obtained prints, expressed as, UPF value, are governed by the treatment sequence, i.e. pretreatment with MCT-β-CD followed by disperse printing > disperse printing only without pre-modification > untreated, as well as the structure of the disperse dye molecules its ability to absorb the hazardous UV-radiation, especially UV-B along with the bonds between dye-molecules with blend components, (Gorensek & Slnga, 2004; Ibrahim, Refai, Youssef, & Ahmed, 2005) (vi) the high UPF value of untreated blend fabric reflects the positive role of polyester component with its aromatic units on exhibiting a high absorption of UV-B radiation along with the good UV-absorption properties of the wool component, (Gorensek and Slnga, 2004; Ibrahim et al., 2005) (vii) the modified fabric samples, functionalized with MCT-β-CD, exhibits higher UV-protecting capacities compared to the unmodified or untreated ones, suggesting the formation of the host-guest inclusion complex between the MCT-β-CD fixed at and/or within the finish/wool matrix and the hydrophobic disperse dye molecules, and the ability of the formed complex to act as a highly efficient UV-filter, (Ibrahim et al., 2008; Gorensek and Slnga, 2004; Ibrahim et al., 2005) and (viii) darker depth of shades, i.e. higher K/S values, significantly improve the UPF values, regardless of the used disperse dye.

4. Conclusion

Inclusion of the MCT-β-CD, with its hydrophobic cavities, onto and/or within the wool/polyester blend fabric was found to exhibit an outstanding increase in the printability with disperse dyes along a remarkable improvement in the fastness properties of the obtained solid-shade prints. This enhancement in the printing properties is direct consequence of modifying the ability of wool component to form solid inclusion complexes (host-guest complexes) with the vapors of the sublimable disperse dyes during the steam fixation step. The investigation reveals that-union printing of wool/polyester blend, deeper depth of shades along with better fastness properties are achieved if MCT-β-CD (60 g/L), DMDHEU (20 g/L), citric acid (5 g/L), and PEG-600 (10 g/L) are used in the pre-modification step followed by thermofixation at 120 °C 15 min and subsequent disperse printing according to the selected printing conditions. As a result of the inclusions, along with the subsequent improvement in the depth of shades, regardless of the used disperse dye, the UV-blocking functions of the obtained disperse prints were significantly increased.

Table 1

Printing and UV-protecting properties of the obtained disperse prints.

Disperse dye (30 g/kg)	Substrate	%N	K/S	Increase in K/S %	WF		RF		PF			
					Alt.	C	Dry	Wet	Acidic		Alkaline	
									Alt.	C	Alt.	C
None	Untreated (blank)	8.65	–	–	–	–	–	–	–	–	–	–
Disperse® Red 54	NM	8.79	7.93	149.2	2–3	2	2–3	2	2–3	2–3	2–3	2–3
	M	9.28	19.76		4–5	4–5	4–5	4	5	4–5	5	5
Disperse® Red 167	NM	8.89	8.98	125.3	2–3	2–3	2–3	2	2–3	2–3	2–3	2–3
	M	9.03	20.23		5	4–5	5	4–5	5	4–5	4–5	4–5
Disperse® Blue 183	NM	8.95	0.98	330.6	2–3	2–3	3	2–3	2	2	2–3	2–3
	M	9.25	4.22		4–5	4–5	4–5	4–5	4–5	4	4–5	5
Disperse® Red HBL	NM	8.73	7.52	173.8	2–3	2–3	2	2	2	2–3	2–3	2–3
	M	8.93	20.59		4–5	4–5	5	4–5	4	4–5	4–5	4––5

– Pre-modification step: -MCT-β-CD (60 g/L), DMDHEU (20 g/L), citric acid (5 g/L), PEG-600 (10 g/L), wet pick-up 70%, fixation at 120 °C/5 min.

– Post-printing: disperse dye (30 g/kg), thickening agent (700 g/L-5%), acetic acid (20 g/kg-30%), drying at 85 °C/5 min, steaming at 40 °C for 30 min, WF, Wash fastness; RF, rubbing fastness; PR, perspiration fastness; UPF, ultraviolet protection factor.

NM, non-modified (Only printed substrate).

M, modified (Printed substrate).

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