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# Facile Approach to Enhance Disperse Printability and Antibacterial Functionality of Wool/Polyester fabric



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#### Abstract

In this study, wool/polyester (50/50) blended fabric was chemically modified using monochlorotriazinyl  $\beta$ -cyclodextrin (MCT- $\beta$ CD) to create hydrophobic cavities onto/within the fabric structure. The introduced hydrophobic cavities had the ability to form inclusion complexes with both the disperse dye molecules and the selected antibacterial agents namely triclosan derivative, Invasan®, and hyperbranched poly (amid amine) loaded with nano silver, HBPAA/AgNPs composite, in subsequent disperse dyeing and functional finishing respectively. The obtained results revealed that chemical modification of wool/polyester structure using MCT-  $\beta$ CD (20g/L), followed by post-printing, and functional finishing with the nominated active ingredients, triclosan (10g/L) and AgNPs/HBPAA hybrid solution (10g/L) result in a significant improvement in the depth of the obtained disperse prints along with a noticeable increase in the imparted antibacterial functionality against the tested harmful pathogens namely Gram-positive (G+ve: *S. aureus*) and Gram-negative (G-ve: *E. coli*) bacteria.

On the other hand, the SEM and EDX analysis has confirmed both the deposition of MCT- $\beta$ CD onto the fabric surface as well as immobilization and fixation of the used bioactive agents.

Keywords: Wool/polyester fabric, MCT-βCD, Pre-modification, Post-disperse printing, Subsequent antibacterial finishing.

#### 1. Introduction

Textile fibers can be generally classified into natural fibers such as cellulose, wool fiber and man-made fibers which can be categorized into two main groups namely generated fiber such as viscose, lyocell and synthetic fibers such as polyester and polyamide, etc. Blending synthetic fibers with other natural/manmade fiber is carried out to minimize the total production cost, and to enhance the performance properties of the obtained textile qualities [1-5]. On the other hand, natural fibers can provide a proper environmental conditions for accommodation and growth of harmful microorganisms such as bacteria, fungi, etc. which in turn increase the medical and hygienic risk, generate unpleased odors along with degradation and discoloration of the textile products. Hence, the antimicrobial functionalization of natural textile materials has gained great attention by researchers for textile material protection from the attack of harmful microorganisms via utilization of the proper antimicrobial agents that can inhibit the growth or even killing the harmful bacteria.[6-13],

Additionally, chemical modification of textiles using  $\beta$ -Cyclodextrin is carried out to create hydrophobic cavities into/onto fabric structure. These hydrophobic cavities have the ability to host-gust certain molecules in their internal hydrophobic cavities in form of host-gust inclusion complexes. Certain dyestuff such as disperse dye, reactive dye, oils such as neem oil, tulsi, lavender, clove, etc. and other active ingredients like natural dyes, nanoparticles, silver nitrate, etc. This unique feature of  $\beta$ -Cyclodextrin offers an eco-friendly tool to develop high quality colored and functionalized textile products [14-21].

Therefore, this research work was carried out to modify wool/polyester (50/50) structure to develop disperse prints with remarkable antibacterial activity using MCT- $\beta$ CD as modifying agent and triclosan or HBPAA/ AgNPs hybrid as antibacterial agents.

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#### 2. Experimental

#### 2.1. Materials

Plain woven mill-scoured and bleached wool/polyester (50/50,  $230g/m^2$ ) blended fabric was used.

Cavasol® W7MCT (monochlorotriazinyl  $\beta$ -cyclodextrin, MCT-  $\beta$ CD, average molecular weight  $\sim 1560$ , degree of substitution (0.3-0.6 per anhydro glucose unit- Wacker, Germany), and Invasan® (based on triclosan- Huntsman, USA),Dialgin®LV-100 [Na-alginate of low viscosity, BF-Goodrich Diamalt, GmbH, Germany), and Leomin® W (nonionic wetting agent and detergent, BASF, Germany) were of commercial grade.

Disperse Red 74, and Disperse Blue 183 (Sinochem Ningbo, China) were used for disperse printing of the nominated substrate.

Hyper branched poly (amide-amine-HBPAA)/AgNPs composite was synthesized as described before [22]. The produced AgNPs were spherical in shape with particle size ranged between 32 and 35 nm Fig. 1

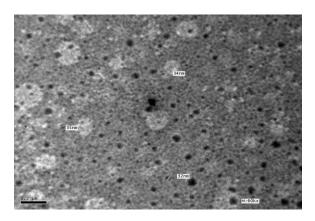


Fig. 1 TEM images of AgNPs/HBPAA hybrid

#### 2.2.1. Pre-loading of MCT-βCD

Aqueous solution of MCT-βCD (0-20g/L) along with sodium carbonate, Na<sub>2</sub>CO<sub>3</sub>,(0-20g/L) and nonionic wetting agent (2g/L) were prepared with constant stirring. Fabric samples were padded twice in the prepared solutions to wet-pick up 80% owf, followed by direct fixation at 120°C for 10 min. The treated fabric samples were then rinsed for 10 min to remove

the unfixed and/or partially hydrolyzed MCT- $\beta$ CD and finally dried at 100°C for 5min.

#### 2.2.2. Post-disperse printing

The pre-modified fabric samples and un-modified ones were post-printed using the nominated disperse dyes using the flat screen technique according the following conditions:

Printing paste components	g/kg paste
Disperse dye	20
Na-alginate (10%)	500
Acetic acid (30%)	15
Water	465
Total	1000

Printed fabric samples were then dried at 85°C for 5 min and steam fixed at 120°C for 15 min using Ariolt® CSL-steamer, Italy. Post-printed fabric samples were rinsed thoroughly, soaped for 15 min at 60°C in the presence of 2g/L nonionic wetting agent, then thoroughly rinsed and finally dried at 85°C for 5 min

## 2.2.3. Post-finished with antibacterial agent

## 2.2.3.1. Invasan®(triclosan derivative)

Portion of printed fabric samples were post-finished with triclosan derivative aqueous solution (10g/L) along with a nonionic wetting agent (2g/L) at pH 5 using acetic acid, LR (1/20), using sample dyeing machine with agitation rate (40 rpm), at 50°C for 30 min followed by squeezing to give a wet pick-up of 80%, thermofixed at 150°C for 3min, thoroughly rinsed to remove excess and unfixed active ingredients and finally dried at 100°C for 5 min.

#### 2.2.3.2. AgNPs/HBPAA hybrid

Another portion of active prints were padded twice in a finishing formulation containing AgNPs/HBPAA (10g/L), along with a nonionic wetting agent (2g/L) at pH5, using acetic acid, to give a wet pick-up of 80%, followed by direct thermofixation at 150°C for 3min, rinsed thoroughly, washed at 50°C for 10 min

in the presence of a nonionic wetting agent to remove unreactant and unfixed hybrid, rinsed and finally

dried at 100°C/5 min.

#### 2.3. Test methods

Nitrogen content %N was determined according to the micro-Kjeldahl method [23].

The depth of the obtained reactive prints before and after post-finishing, was determined at the wavelength of the maximum absorbance using an automatic filter spectrophotometer, and calculated using the Kubelka Munk equation: K/S = (1-R)2/2R, where K, S and R are the absorption coefficient, the scattering coefficient and the reflectance respectively [24].

Fastness properties to washing, rubbing, perspiration and light of the obtained prints were evaluated according to AATCC test methods: (61-1972), (8-1972), (15-1973) and (16A-1972) respectively[25].

Antibacterial activity of the treated and untreated fabric samples against Gram-positive (S. aureus, G+ve) and Gram-negative (E. coli, G-ve) pathogenic bacteria was evaluated qualitatively according to AATCC Test Method (147-1988), and expressed as zone of growth inhibition (ZI, mm)[10].

Scanning electron microscope (SEM) images of selected samples were evaluated using a JEOL, JXA-840A electron probe microanalyzer equipped with disperse X-ray spectroscopy (EDX) for the surface composition analysis.

## 3. Results and Discussions

## 3.1. Effect of sodium carbonate concentration as a catalyst

The variation in N% of the modified substrate using  $Na_2CO_3$  as a catalyst and MCT- $\beta$ CD as a modifying agent is given in Fig. 2a. Fig. 2a shows that, increasing  $Na_2CO_3$  concentration from zero to 10g/L results in a significant increase in the nitrogen content, %N, of the modified fabric samples as a direct consequence of chemical fixation of MCT- $\beta$ CD onto/into the fabric structure [26, 27].

Further increase in the used catalyst concentration, beyond 10g/L, has a slight or no effect on the %N.

Moreover, Fig. 2b illustrates the effect of modifying the wool/polyester structure and creating of hydrophobic active sites on subsequent disperse prints. For a given set of treatment conditions, It is clear that increasing of Na<sub>2</sub>CO<sub>3</sub> (up to 10 g/L) brings about a significant improvement in the depth of the developed prints expressed as K/S values, which could be discussed in terms of the ability of the loaded and fixed MCT-βCD into/onto textile prints with its hydrophobic cavities to form inclusion complex with the used disperse dye molecules [28]. Further increase in Na<sub>2</sub>CO<sub>3</sub> showed lower K/S values most probably due to the partial hydrolysis of MCTβCD at high Na<sub>2</sub>CO<sub>3</sub> concentration (> 10g/L), thereby minimizing its extent of loading and immobilization as well as the number and availabilty of grafted hydrophobic active sites to form inclusion complex with the disperse dye molecules, i.e. less K/S values [Eq. 1] [16].

Therefore, 10g/L Na<sub>2</sub>CO<sub>3</sub> was considered to be the optimal alkali concentration for the pre-modification step.

CI-MCT-
$$\beta$$
CD + H<sub>2</sub>O  $\xrightarrow{\text{OH}^-}$  Steam
HO- MCT- $\beta$ CD + HCl

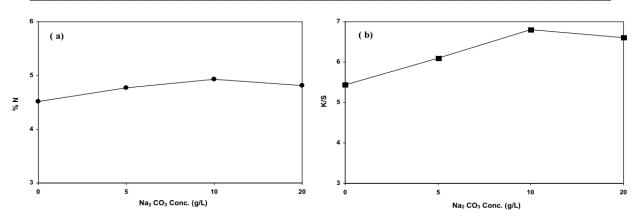


Fig.2. Effect of  $Na_2CO_3$  on extent of fixation of MCT- $\beta$ CD onto the wool/polyester structure (%N, a) and its impact on post-printing with the disperse dyestuff (K/S, b)

## 3.2. MCT-βCD concentration

Fig. 3a demonstrates that increasing concentrations of MCT-βCD up to 20g/L is accompanied by an increase in the %N of the treated samples as a direct consequence of increasing the extent of fixation of MCT-βCD into/onto the modified substrate [Eq. 2].

PET 
$$\sim$$
W-XH + Cl-MCT- $\beta$ CD  $\xrightarrow{Na_2CO_3}$  PET  $\sim$ W-X-MCT- $\beta$ CD + HCl (2)

Wool/polyester Modified Wool/polyester

where PET= Polyester, W-XH = wool , -XH = -NH<sub>2</sub>, -SH, -OH

Further increase in MCT- $\beta$ CD concentration, >20g/L, has practically marginal effect on the N% content most probably due to the shortage and non-acceptability and availability of the substrate active sites [14].

On the other hand increasing MCT- $\beta$ CD concentration (up to 20g/L) in pre-modification step positively affects the extent of disperse dye fixation onto the grafted hydrophobic cavities (Fig.3b) of the fixed and grafted  $\beta$ CD moieties [15].

### 3.3. Post-finishing with antibacterial agents

#### 3.3.1. Post-finishing with Triclosan

The impact of post-finishing with Invasan®, triclosan derivative, (10g/L) on the extent of coloration and antibacterial functionalization of the MCT-βCDloaded → post-printed fabric samples is demonstrated Table 1. Inclusion of Triclosan in the post-finishing bath brings about a remarkable increase in %N, K/S values and enhancement in the fastness properties of the post-finished prints. This improvement in the abovementioned coloration properties is most likely attributed to the improvement in the fixation degree of the disperse dye molecules into/onto the premodified-post loaded substrate. Moreover, the improvement in the imparted antibacterial activity of the modified-printed wool/polyester is due to the ability of the loaded BCD-moieties to form host-guest inclusion complex with Invasan®. Additionally, the imparted antibacterial activity against both harmful bacteria (S. aureus), and (E. coli) attributed to the capability of loaded Triclosan to disrupt the bacteria cell and/or via its ability to inhibit bacterial fatty acid synthesis [29, 30]. On the other hand, the antibacterial efficacy of the finished wool/polyester fabric against tested pathogenic bacteria follows the descending order G+ve> G-ve [31, 32].

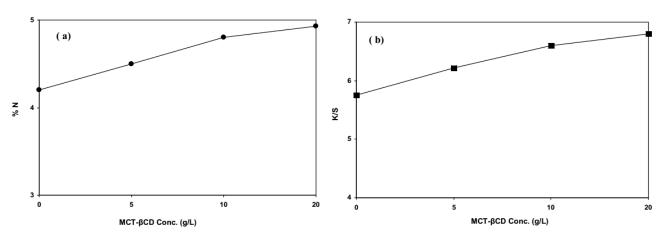


Fig.3. Effect of MCT- $\beta$ CD concentration on the modification of wool/polyester (%N, a) and its impact on post-printing with the disperse dyestuff (K/S, b)

Table 1

Effect of post-treatment using Invasan® on disperse printing and antibacterial finishing of polyester/wool blend

fuff	Substrate	%N	K/S	Incr. in K/S (%)	WF		RF		PF					al Ac	acteri tivity mm)
Dyestuff					Alt	С	Dry	Wet	Acidic		Alkaline		LF	(2-2, 11111)	
									Alt	C	Alt	C		G +ve	G -ve
Disperse Red 74	UT	5.25	5.14	73.34	4	3	4	4	4	4	4	4	4	0	0
Dis	Т	6.08	8.91		4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	22	19
erse ue	UT	5.03	23.79		3-4	4	3-4	4	3-4	3-4	4	4	4	0	0
Disperse Blue 183	Т	6.21	28.00	17.69	4-5	4-5	4-5	4-5	4-5	4-5	5	4-5	4-5	28	26

Post-finishing: Invasan® (10 g/L); nonionic wetting agent (2g/L); pH (5); wet pick-up (80%); thermofixation at 150 °C/3 min. N%: nitrogen content; K/S: color strength; WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; LF: light fastness; ZI: zone of inhibition; UT: untreated; T:aftertreated with Triclosan; Alt: alteration; C: staining on cotton

#### 3.3.2. Post-finished with AgNPs/HBPAA hybrid

Table 2 shows the effect of post-treatment using AgNPs /HBPAA hybrid solution (10g/L) on printing and antibacterial properties of the developed substrates. Post- finishing of the printed substrates with AgNPs/HBPAA hybrid increases both N% and K/S values along with enhancing the tested fastness properties of the finished prints i.e. wash, perspiration, rubbing and light fastness properties. The enhancement in these properties is governed by the type of dye, location, distribution, and extant of fixation of the used hybrid onto the printed samples.

This improvement confirmed interactions of the modified wool/polyester structure active sites, i.e. -βCD moieties, -OH, , -COOH and -NH<sub>2</sub> groups, with the loaded AgNPs/HBPAA, (-NH, -NH<sub>2</sub>) groups and the encapsulated AgNPs by hydrogen bonding, electrostatic interactions and/or complex formation. On the other hand, post-finishing with the prepared hybrid showed a significant improvement in the antibacterial efficacy of the treated samples. Wool/polyester prints post finished AgNPs/HBPAA showed better antibacterial capability against S. aureus than E. coli bacteria. Additionally, the imparted antibacterial functionality of AgNPs/HBPAA-loaded prints is due to the

interaction of AgNPs with sulfur-or phosphorouscontaining protein, causing DNA damages which in turn has negative influence on the respiratory process and cell division, thereby changing the metabolism of microorganism, preventing its growth and leading to the death of the cell [22, 33-37].

## 3.4. SEM images and EDX analysis

Fig. 4 represents the SEM images and EDX spectra of the disperse printed wool/polyester pre-modified with MCT-βCD Fig. 4 (a,b) and post-finished with AgNPs/HBPAA composite Fig. 4 (c,d) respectively.

EDX spectra, Fig. 4b, showed elements of carbon, oxygen and nitrogen as well as Sulphur that related to the wool/polyester component and the used disperse dye, while EDX spectra of post-finished with AgNPs/HBPAA hybrid, Fig. 4d, displayed additional peak of silver element which in turn confirmed the existence of AgNPs onto the finished wool/PET fabric sample.

Table 2

Effect of post-treatment using Ag-NP's /HBPAA hybrid on disperse printing and antibacterial finishing of polyester/wool blend

Dyestuff	Substrate	% N	K/S	Incr. in K/S (%)	WF		RF			I	PF		LF	Antibac al Activ (ZI, m	tivity
					4.74	Alt C	Dry	Wet	Acidic		Alkaline				
					Alt				Alt	С	Alt	С		G +ve	G –ve
Disperse Red	UT	5.25	5.14	69.26	4	3	4	4	4	4	4	4	4	0	0
	Т	6.13	8.70		4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	20	18
Disperse Blue 183	UT	5.03	23.79	16.98	3-4	4	3-4	4	3-4	3-4	4	4	4	0	0
	Т	6.10	27.83		4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	20	19

Post-finishing: Ag-NP's/HBAA hybrid (10 g/L); nonionic wetting agent (2g/L); pH (5); wet pick-up (80%); thermofixation at 150 °C/3 min. N%: nitrogen content; K/S: color strength; WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; LF: light fastness; ZI: zone of inhibition; UT: untreated; T:aftertreated with hybrid; Alt: alteration; C: staining on cotton.

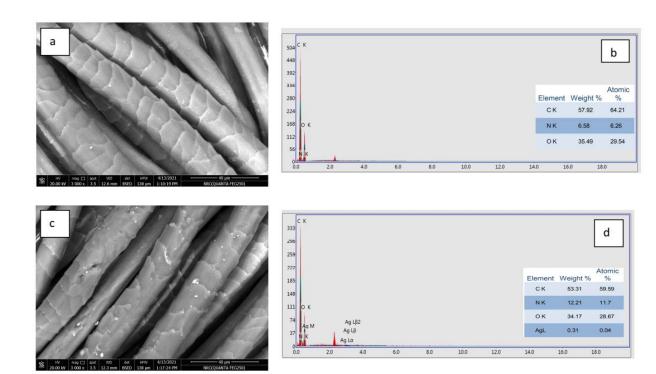


Fig 4. SEM image and EDX spectrum of: Untreated wool/polyester (a,b) and  $\beta$ CD-loaded  $\rightarrow$  Disperse printed  $\rightarrow$  Ag-NP's /HBPAA hybrid post-treated (c, d) respectively.

#### 4. Conclusions

In this work, a new approach for disperse printing and antibacterial finishing of wool/polyester (50/50) fabric was successfully developed. The suggested method comprised the following steps: grafting of MCT- $\beta$ CD  $\rightarrow$  disperse printing  $\rightarrow$  post-finishing with the nominated antibacterial agents namely triclosan and AgNPs/HBPAA. Pre-modification of the blended substrate with MCT-βCD followed by post disperse printing with subsequent antibacterial functional finishing had a positive impacts on both coloration and functionalization properties. Moreover, SEM images and EDX spectra for selected fabric sample confirmed the changes in surface morphology as well as the existence of Ag element compared with unfinished fabric samples.

Conclusively, this suggested sequence of treatment is considered to be a facile and effective approach to obtain high quality disperse printed wool/polyester fabric with outstanding durable antibacterial functionality against the pathogenic bacteria.

## **Conflicts of interest**

There are no conflicts to declare.

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