Optimization of Easy-Care Finishing of Cotton/Polyester Blend Fabric

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Summary: The objective of the given study was to obtain eco-friendly finishes and optimized application parameters of easy-care finishing. Easy-care finishing of cotton/polyester blend fabric was done at different pH values and resin concentrations using three different types of cross-linking finishing resins: modified dihydroxyethylene urea (I), modified dimethyloldihydroxyethylene urea (II), and modified *N*-methyloldihydroxyethylene urea (III). Optimum mechanical properties (wrinkle recovery angle WRA; 252 (W+F^o) and tensile strength TS; 2143 N) were observed at 30 g/l of resin I and 5.0 pH. At these conditions resin I showed lowest free formaldehyde content (3.07 mg/kg). Formaldehyde (HCHO) was extracted, derivatized, and estimated as DNPH-formaldehyde using HPLC through a C18 column. No appreciable difference in easy-care properties were observed when cotton/polyester blend fabric was finished, in optimized conditions, with resins II and III, except slightly increased HCHO.

Keywords: Polyester, Cross-linking finishing resins, DNPH-formaldehyde, HPLC.

Introduction

Cellulose-containing fabrics finished to easy-care properties with durable-press finishing are reported eco-friendly [1]. In last few decades the interest of buyer in wrinkle-resistant apparel has diverted the producers' attention to use easy-care finishes; however, studies have shown that the mechanical properties of cellulosic and cellulosic blend fabric finished with easy-care resins are influenced [2, 3]. Finishing is very important for the fabric attraction and market acceptance. Finishing trends nowadays are dictated by the consumers' requirements and purposes for which fabric is finished, and must be regulated by eco-friendship, biodegradability and durability [4]. Urethane prepolymers and diisocyanates are employed as HCHO-free, durable-press finishing agents for polyester, cotton, and their blends [5] and cotton blend textile fabrics show dimensional rigidity in the dry form but shrinking and wrinkling in the wet state. This happens because water forms cross-linked structures *via* hydrogen bonding. Dimensional stability in cellulosic fabric is due to the hydrogen bonded cross-linked structures. Thus for durable press characteristics it is essential to form such cross-linking, which are not easily replaced by water and laundering.

Easy care finishes such as formaldehyde based resins, methylol compounds, etc. are widely used finishing agents in textile industries to get wrinkle-resistant cotton and cotton/polyester blends. Cotton and cotton/polyester blends finished with formaldehyde based resins have great concerns due to the release of free formaldehyde, therefore, finishing conditions and techniques are continuously a subject of study [6-15]. Dimethyloldihydroxyethylene urea (DMDHEU) has been used to stabilize the wood (a cellulosic complex) from weathering [16, 17]. Textile substrates finished with formaldehyde based finishing agents are known to cause allergy, due to the release of free formaldehyde [18, 19]. Formaldehyde is a suspected carcinogen and an irritant to the eyes and lungs when in high concentrations [20]. Toxicology of formaldehyde has been extensively studied [21], leading to establish regulations and introduction of certain limits of formaldehyde content in textile substrates. With new cross-linking agents it is possible to achieve formaldehyde content less than 100 mg/kg [22]. European Standard (Oeko-Tex 100) has established the limits of concentration of HCHO, which should not be more than 75 mg/kg for the fabrics touched directly by skin and 300 mg/kg for the fabrics not touched directly by skin and for fabrics used in interior decoration [23].

In finishing of cellulosic and cellulosic blend fabrics, cross-linking agent DMDHEU penetrates into the fibers and reacts readily with the hydroxyl groups of adjacent cellulose chain. This produces crosslinks and masks the free hydroxyl moieties, which reduces the shrinkage and swelling and thus improves the crease resistant properties of fibers. On the other hand easy-care finishing imparts negative effect on the mechanical properties of the

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finished fabric e.g., tensile strength. Incorporation of some polymer additives in finishing process of cellulosic and cellulosic blend fabric along with DMDHEU increases the tensile strength, nitrogen content, and acid dye ability of the fabric [1, 24-26].

Recently, traditional formaldehyde based resins have been replaced with non-formaldehyde finishes, namely, polycarboxylic acids, such as 1,2,3,4-butanetetracarboxylic acid (BTCA) and citric acid (CA), and dialdehydes, e.g., glyoxal, and glutaraldehyde. The disadvantages of using these finishing agents for cotton fabrics have also been reported [2, 27-30].

The reaction modifying the cotton/polyester blend fibers by DMDHEU is well known, where in the curing stage hydroxyl group of DMDHEU and cellulose reacts together with the elimination of water. The ether cross linkages so formed stiffens the cellulose to resist the wrinkle formation at the cost of mechanical strength [24].

To make the finished cotton blend fabrics more acceptable to consumers, producers, environment experts, and economists, various works have been done in this direction [6-15]. None of these studies have simultaneously reported the physical analyses (tensile strength, wrinkle recovery angle, and SEM analyses), chemical analyses (free formaldehyde using HPLC), along with the optimization of the finishing conditions (pH variation in the resin's application bath and varying the concentration of resins), applying three resins of different chemistry.

Results and Discussion

In the current study types and concentrations of resins and the application parameters of finishing bath were varied, during resin finishing of cotton/polyester blend fabric. Each application experiment was followed by the chemical analysis to monitor the conditions, which produces minimum release of formaldehyde content. Similarly mechanical properties were also monitored. The variables included pH and concentration of resin in finishing bath. The target was to find a compromising increase in wrinkle-resistance and tensile strength with low free HCHO, with which the finished cotton/polyester blend fabric will meet market values ecologically and economically.

Effect of Variation of pH and Resin Concentration

Table-1, 2 and 3 present the release of formaldehyde content, wrinkle recovery angle and tensile strength of the finished cotton/polyester blend fabrics with resin I, II and III respectively, when pH and resin concentrations in finishing formulation were varied. The data indicated that the treatment of cotton/polyester blend fabric with resin I in slightly acidic medium (pH 5.0-6.0); and resin II and III at pH 5.0 gave low formaldehyde content in finished fabric with reasonable tensile strength. However, the wrinkle recovery angle did not show noticeable changes. It was noticed that cotton/polyester blend fabrics finished with resin I, II, and III at pH lower than 5.0 gave high formaldehyde content and lower value of tensile strength and wrinkle recovery angle. The concentration of resins were also varied and monitored to find the cost effective range of resin's concentration in finishing bath, so that fabrics remain fit for market requirements and eco-friendship. As a result 30 g/l concentration of resins I, II, and III gave lowest formaldehyde content with reasonable wrinkle recovery angle and tensile strength.

Table-1: Properties of fabric treated with resin-I.

Conditions	Entry	Concentration	pH	Free HCHO	WRA	TS
units		(g/l)		(mg/kg)	(W+F°)	(N)
pH variation	1	30	4.0	4.67	250	2126
	2	30	5.0	3.07	252	2143
	3	30	6.0	3.46	252	2153
	4	30	7.0	3.84	251	2165
Concentration variation	5	Untreated	5.0	2.32	210	2232
	6	30	5.0	3.07	252	2143
	7	40	5.0	4.72	261	2125
	8	50	5.0	7.87	267	2113
	9	60	5.0	10.94	274	2099
	10	70	5.0	16.60	279	2090

All results are average of three replicates. WRA; wrinkle recovery angle, TS; tensile strength.

Table-2: Properties of fabric treated with resin-II.

Conditions	Entry	Concentration	pH	Free HCHO	WRA	TS
units		(g/l)		(mg/kg)	(W+F°)	(N)
pH variation	1	30	4.0	21.48	244	2149
	2	30	5.0	16.33	247	2164
	3	30	6.0	20.41	248	2172
	4	30	7.0	22.56	247	2182
Concentration variation	5	Untreated	5.0	2.32	210	2232
	6	30	5.0	16.33	247	2164
	7	40	5.0	19.60	257	2151
	8	50	5.0	20.37	265	2137
	9	60	5.0	25.74	269	2122
	10	70	5.0	27.31	275	2109

All results are average of three replicates. WRA; wrinkle recovery angle, TS; tensile strength.

Table-3: Properties of fabric treated with resin-III.

Conditions	Entry	Concentration	pH	Free HCHO	WRA	TS
units		(g/l)		(mg/kg)	(W+F°)	(N)
pH variation	1	30	4.0	19.48	243	2156
	2	30	5.0	18.68	245	2172
	3	30	6.0	19.08	244	2178
	4	30	7.0	20.18	244	2187
Concentration variation	5	Untreated	5.0	2.32	210	2232
	6	30	5.0	18.68	245	2172
	7	40	5.0	21.02	256	2159
	8	50	5.0	22.31	262	2142
	9	60	5.0	23.93	266	2131
	10	70	5.0	25.64	273	2115

All results are average of three replicates. WRA; wrinkle recovery angle, TS; tensile strength.

Optimization of the Finishing Conditions

Wrinkle recovery angle of treated cotton/polyester blend fabric increases with increasing concentration of resins in finishing bath at pH 5.0. On the other hand, concentration above 30 g/l causes an increase in the release of formaldehyde content. The pH 5.0-6.0 and concentration 30-60 g/l of resin I in finishing bath was found to be the best for easy-care finishing of cotton/polyester blend fabrics. Similarly, cotton/polyester blend fabrics finished with resins II and III at concentration ranging 30-60 g/l gave considerable improvement in wrinkle recovery angle. An increase in the free formaldehyde content was also observed. The best conditions of finishing bath, when resins II and III used, were found to be 30-40 g/l at pH 5.0.

SEM Evaluation

The SEM technique was used to examine the surface morphology of fiber from untreated and resin finished fabric. The micrographs showed that twisting around single fiber of untreated fabric was in greater extent when compared with the resin finished cotton/polyester blend fabric (Fig. 1). This provides the evidence that cross-linking agent has formed the desired cross-linked structure with the free hydroxyl groups of cellulose chains, thus are quite stable to wrinkling or twisting. These SEM micrographs were also compared with the analytical data of wrinkle recovery angle measured by wrinkle recovery tester. The value of wrinkle recovery angle of untreated and resin finished fabric (Table-1, Entries 5-10) is in support with the SEM micrographs (Fig. 1). The relative degree of wrinkling in resin treated fabric was in the order of: resin I < resin II < resin III.

Experimental

Materials and Chemicals

Bleached cotton/polyester (50:50) blend fabric (88 g/m^2), provided by Mustaqeem Dyeing and Printing Industry, Karachi, was used throughout this study. Fabric was not subjected to any finishing process except those discussed in finishing process.

Three commercial grade finishing resins were obtained either from local textile industry or suppliers. These were; modified dihydroxyethylene urea, modified dimethyloldihydroxyethylene urea, and modified *N*-methyloldihydroxyethylene urea. These finishing resins herein after are named as resin I, II, and III respectively. Sodium thiosulfate pentahydrate, potassium dichromate, iodine, starch, potassium iodide, sodium bicarbonate, sulfuric acid, sodium hydroxide, acetic acid, magnesium chloride, sodium dodecylsulfonate, ortho-phosphoric acid, 2,4-dinitrophenylhydrazine (Art. 803080), and acetonitrile (Art. 1.00030) used were of analytical grade purchased from E. Merck, Germany, while formaldehyde (Art. A2617-R) analytical grade was obtained from Avonchem, UK. HPLC grade water was obtained in house from Pure Lab Option-S 7/15, Elga, UK.

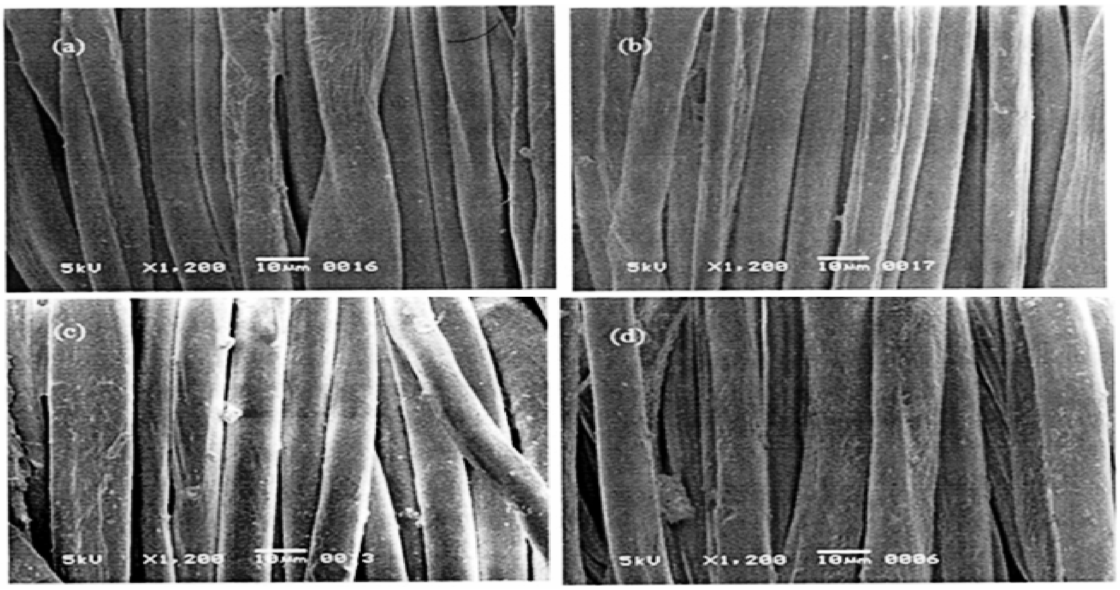

Fig. 1: SEM micrographs of fibers from (a), untreated and resin finished cotton/polyester blend fabric treated with (b) resin-I, (c) resin-II, (d) resin-III.

Equipments

General Equipments

Temperature control shaking was done in water bath BS code AAH 44112K, Jeio Tech, Korea; analytical balance GR-300, A and D®, Japan and pH-meter 3510, Jenway, UK, were used in general.

Fabric Processing Equipments

Finishing was carried out using a laboratory scale vertical padder, Rapid, UK; finished fabrics were cured on a curing machine R-3 NO 825, Rapid, UK.

Analytical Equipments

Analyses of extracted formaldehyde were performed either using Hitachi HPLC (Japan) L-6200A system coupled with L-4000A UV detector and AS-2000A autosampler or Lab Alliance HPLC (USA), hooked with EZ Chrom Elite Software Ver. 3.1.6 (USA), equipped with an analytical column Kromasil 100 C18 (5µm, 0.46 × 25 cm, Teknokroma, Spain); RP18 guard column (7 µm, 0.4 × 3 cm, LKB-Produkter AB, Bromma, Sweden); Grad 2500 Plus pump; Degasys DG 2410 degasser and UV 6000 LP Spectra System diode-array detector. Crease recovery tester M 003A, SDL, UK and tensile strength tester LRX Plus, LLOYD, UK, were used to measure the wrinkle recovery angle and tensile strength of finished fabrics.

Finishing Process

Bleached cotton/polyester blend fabrics were immersed individually in finishing formulation baths containing varying amounts (30, 40, 50, 60, and 70 g/l) of resin I, II, and III at pH 5.0, in the presence of a catalyst $MgCl_2$ (5 g/l) and nonionic wetting agent (2 g/l). 30 g/l of resins I, II, and III were also applied at different pH values (4.0, 5.0, 6.0, and 7.0), maintained with acetic acid. The padded fabrics with 80 % liquor pick-up were squeezed, dried, and cured at 160 ^{o}C for 3 min.

Wrinkle Recovery and Tensile Strength Testing

Standard methods AATCC-66 (2003) and ISO-13934-1 (1999) were used to evaluate wrinkle recovery angle (WRA) and Tensile strength (TS) of finished cotton/polyester blend fabric respectively.

Scanning Electron Microscopy (SEM)

The microscopic study of fibers from untreated and resin finished cotton/polyester blend fabric was carried out using analytical scanning electron microscope JSM-6380A, Jeol, Japan. The samples were coated with gold using an ion sputtering device JFC-1500, Jeol, Japan and micrographs were taken at an accelerating voltage of 5 kV.

Formaldehyde Extraction

Extraction of formaldehyde was done, under temperature control system for 60 ± 5 min, using shaking water bath at 40 ± 0.5 ^{o}C. 50 ml sodium dodecylsulfonate (0.1 %) solution was used to extract formaldehyde from 2 ± 0.1 g small specimen of finished fabrics. Extracts were filtered on whatman 41 filter paper and cooled down to room temperature.

HPLC Analyses

Recrystallized DNPH from acetonitrile-methanol (70:30 v/v) was used in derivatization. 5.0 ml filtrate followed by 4.0 ml acetonitrile and 0.5 ml DNPH (0.3 %) solution were measured and the volume was made up to 10.0 ml. Reaction mixture was shaken smoothly and gently and allowed to stand for 60 ± 5 min. Detection wave length and flow rate of mobile phase acetonitrile-water (60:40 v/v) was set at 360 nm and 1.0 ml/min respectively. 20 µl of standard and sample DNPH-HCHO derivatives were injected and quantified by external standard method.

Conclusion

An attempt has been made to achieve durable press, wrinkle-resistant, eco-friendly cotton/polyester blend fabric. Free HCHO is directly related to the concentration of resin applied to the fabric. Therefore, to find out the optimum value, pH of finishing bath was varied using minimum (30 g/l) resin in finishing bath. The effect of change in pH was observed in terms of mechanical properties (wrinkle recovery angle and tensile strength) and free HCHO content. Slightly acidic condition in finishing bath (pH 5.0) produces low formaldehyde content and reasonable tensile strength as compared to strong acidic condition. Varying concentration of resin at pH 5.0 showed that cotton/polyester blend fabric cross-linked with 30-40 g/l aqueous solution of resins I, II, and III produced better performance properties such as wrinkle recovery angle, tensile strength and optimized formaldehyde content. Cotton/polyester blend fabric treated with resin I are more eco-friendly with slightly low tensile strength while mechanical properties of fabrics treated with resins II and III were good with slightly elevated free HCHO.

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