Application of Polyurethane/Citric Acid/Silicone Softener Composite on Cotton/Polyester Knitted Fabric Producing Durable Soft and Smooth Surface

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Received 4 December 2010; accepted 23 July 2011
DOI 10.1002/app.35399
Published online 29 November 2011 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: Application of softeners on fabrics can usually increase the fabric pilling tendency and it is difficult to obtain a soft handle fabric without pilling during wearing. This research was conducted to use various chemicals to reduce pilling with reasonable softness on the cotton/polyester knitted fabric. Diverse composites of the water-based polyurethane resin (PU), citric acid (CA) as a crosslinking agent and silicone-based softener were selected and applied on the fabric through conventional pad-dry-cure method. The characteristics of the treated fabrics including pilling rate, pilling density, water droplet adsorption time, bending length, crease recovery angle, tensile strength, and water contact angle were examined and reported. Application of the polyurethane resin along with citric acid reduced the fabric pilling. However, co-application of resin, CA, and softener improved the fabric crease recovery angle, bending length, and water droplet adsorption time. The preferred formulation was 20 g L^{-1} CA, 25 g L^{-1}PU resin, and 20 g L^{-1} silicone softener. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 4141–4148, 2012

Key words: polyurethane; cross-linking; silicone softener; pilling; softness

INTRODUCTION

Fabric pilling has been a great concern as it spoils fabric surface appearance. The problem of pilling became even more serious with synthetic fibers such as polyester especially in a blend with some fiber of lower tensile strength.[1–4] Gintis and Mead studied the pilling tendency of different synthetic fibers. They revealed that the most pronounced effect on pilling tendency of polyester fibers is obtained by changes in abrasion resistance, which affected the rate of pill wear off.[5] Sivakumar and Pillay found that increasing the polyester fiber content in a polyester /cotton blend fabric increases the pilling.[6] Various factors contributed to pilling including: fiber characteristics, yarn construction, fabric construction, fabric finishing such as singeing, heat setting and enzymatic treatment, humidity, rinse, and softener.[4]

Formaldehyde-based resins used in wash and wear finishing can improve adhesion of fibers in the yarn and reduce pilling.[4–7] These resins may release formaldehyde during application, wearing and washing; however, there are various methods to reduce the formaldehyde release.[8] Also, cellulase can act on the cellulosic fabrics and improve fabric handle and bending along with producing brighter color.[4,8–11]

Using resin on fabric surface can add stiffness to it while this may be undesirable on some fabrics.[4] Therefore, fabric softeners are used in textile wet processing as additives and in home laundering to improve fabric handle. However, these softeners adhere to the fiber surface usually through a weak electrical attraction without any chemical bonds, causing poor washing durability. On the other hand, durable silicone-based fabric softeners render additional performance properties to cotton fabric such as improved wrinkle recovery and crease resistance and improved wear comfort with a smooth handle.[2,12,13]

Tanveer Hussain et al. indicated that softeners based on nonionic organo-modified silicone microemulsion and amino functional polysiloxane decrease the pilling but softeners based on a blend of nonionic surfactants and fatty acid esters, nonionic water-dispersible esters, polyethylene emulsion, and cationic fatty amides have no significant influence on fabric pilling.[14] Also, the size of softener droplet in emulsion is an important factor in fabric pilling. Particle size of macroemulsion silicone softeners is 150–250 nm while the particle size of microemulsion silicone softener is lower than 30 nm.[15] The softener droplets in microemulsion silicone softeners are smaller and allow them to penetrate the inner structure of the yarn or the fabric while macroemulsion

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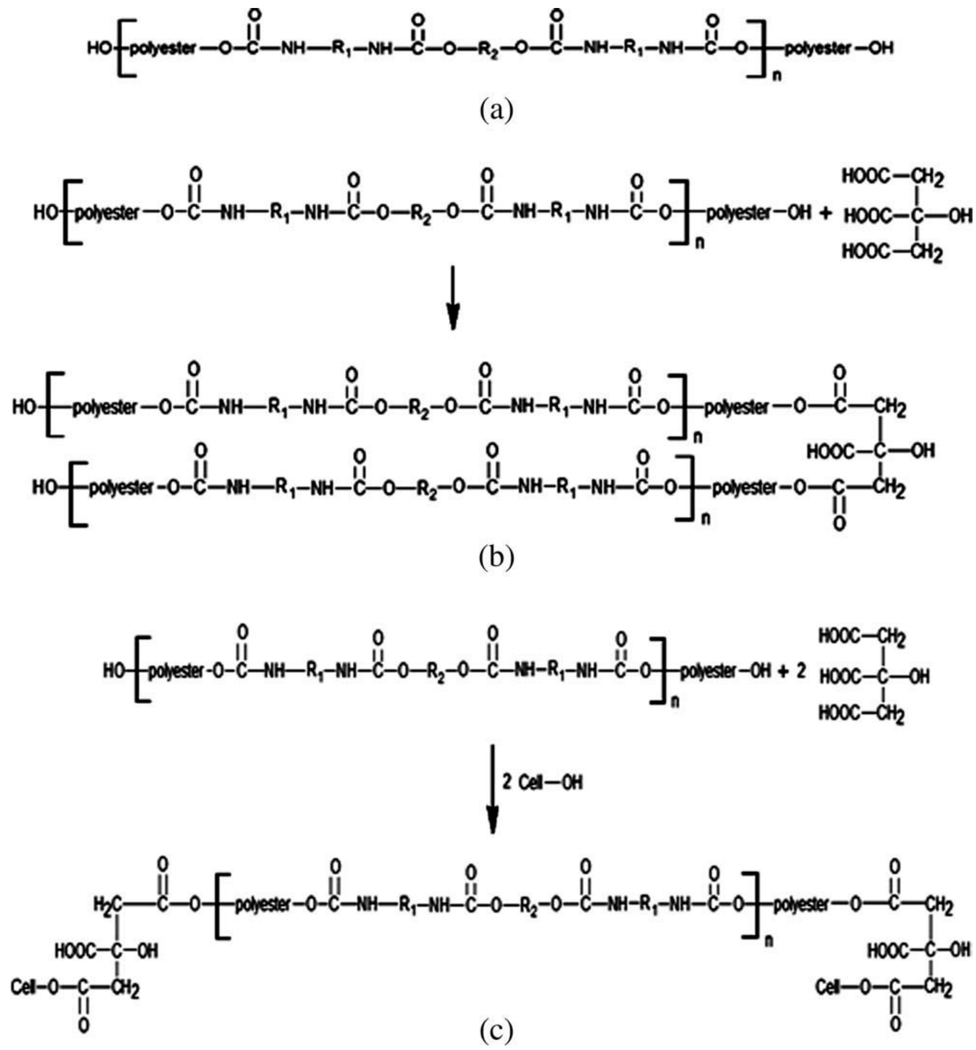


Scheme 1 (a) Water dispersible polyester polyurethane resin, (b) binding of polyurethane resin to citric acid, and (c) binding of polyurethane resin to cellulose by means of citric acid.

silicone softeners can only load on the yarn or fabric surfaces. It is also known that pilling is formed by entangled fiber ends; however, nanosilicone softener increases the slipperiness of fiber surface and entangling of fiber ends becomes difficult. Thus, nanosilicone treatment improves the pilling resistance of fabrics.[16,17]

Montazer et al. have already reported the use of caustic soda along with sodium hydrosulphite to reduce pilling performance of the cotton/polyester woven fabric without yellowing.[18] We have also indicated the application of different blends of aminoplast resins and cross-linking agents on the viscose/polyester knitted fabric obtained permanent anti-pilling fabric.[19] In this study, the composite of polyurethane resin, a carboxylic acid based cross-linker and silicone-based softener was applied on the fabric to obtain a durable soft cotton/polyester knitted fabric with decreased pilling.

EXPERIMENTS

Materials

The 195 g/m^2 knitted fabric containing 35% cotton and 65% polyester was purchased from Aali baft Co. (Iran). Water-based polyester polyurethane resin namely Asupret E-Pol purchased from Asutex CO (Spain) (Scheme 1a). Ultravon GPN based on sodium alkyl aryl sulphonate and ethoxylated fatty alcohol was gifted from Ciba (Switzerland); Adrasil AF microemulsion polymer-based on nonionic silicon softener was gifted from Arghavan resin Co. (Iran), citric acid (CA), and sodium hypophosphite (SHP) were supplied from Merck Chemical Co. (Germany).

Instruments

The Martindale (Shirley) 2002 was used to measure pilling rate according to ASTM D 4970-05. The

TABLE I
Prepration of Samples

Sample no.	CA (g/L)	SHP (g/L)	Polyurethane resin (g/L)	Silicone softener (g/L)
Control	–	–	–	–
1	10	–	–	–
2	20	–	–	–
3	30	–	–	–
4	40	–	–	–
5	50	–	–	–
6	10	6	–	–
7	20	12	–	–
8	30	18	–	–
9	40	24	–	–
10	50	30	–	–
11	–	–	25	–
12	–	–	50	–
13	–	–	75	–
14	–	–	100	–
15	20	–	25	–
16	10	–	25	20
17	10	–	50	20
18	10	–	75	20
19	10	–	100	20
20	20	–	25	20
21	20	–	50	20
22	20	–	75	20
23	20	–	100	20

Shirley Stiffness tester was employed to examine the bending length according to ASTM D 1388-96. The Shirley crease recovery angle, SEM XL30 (Germany) from Philips, pad mangle from Konrad Peter A.G Liestal, Instron (England) and Oven (Germany) from Binder were also used. Contact angle was also measured by Kruss K100-SF tensiometer (Germany).

Methods

The fabric samples were first scoured with 1 g L^{-1} Ultravon GPN at 80°C for 30 min, rinsed with cold water, and dried at 100°C for 10 min. Different concentrations of polyurethane resin, citric acid, and softener were prepared in water as indicated in Table I. The samples were then treated with the various aqueous solutions and padded with 80% wet-pick-up [eq. (1)], dried at 100°C followed by curing at 150°C for 4 min.

$$\text{Wet}-\text{pick}-\text{up}\% = \frac{W_2 - W_1}{W_1} \times 100 \qquad (1)$$

in which W_1 and W_2 are the weight of fabric before treatment and after padding, respectively. Add-on % was determined according to the eq. (2).

$$\text{Add}-\text{on}\% = \frac{W_3 - W_1}{W_3} \times 100 \qquad (2)$$

in which W_3 is the weight of fabric after treatment (after curing).

RESULTS AND DISCUSSION

The test results on the treated and untreated fabric samples reported in Tables II–V.

Add-on %

Increasing the concentration of the cross-linking agent (CA) in the pad bath resulted in intensifying add-on percentages. Also, increasing of the polyurethane resin content in the pad bath had a considerable influence on the add-on percentage. This means that increasing of both polyurethane resin concentration and CA in the finishing bath helps to build up higher resin and CA on the fabric surface.

Bending length

Bending length is one of the determining factors in assessing the fabric handle. A decrease in the bending length leads to improve the fabric drape and produces a desirable fabric handle. The results in Table II indicate that the bending length of the fabrics treated with polyurethane resin (without softener) slightly changed and made the harsh handle. In order to improve the handle of the treated fabric, a silicone-based softener was employed. It is well known that silicone softeners provide very high softness, special unique hand, high lubricity, and good sew ability, elastic resilience, crease recovery, abrasion resistance, and tear strength. Softeners formed cross-linked films.[8] Using softener in the treatment bath has significant influence on the bending length of the treated samples. This is because of the low molecular rotational energy of silicone-based softeners.[8,18] It is well known that use of resins and CA on cotton fabric leads to fabric stiffening. Adding silicone-based softener to the bath containing polyurethane resin and CA prevented the hardening effects of them on the fabric. The chemical structure of the silicone-based softener with siloxane backbone is shown in Scheme 2.

Crease resistance

It was expected that adding cross-linking agent (CA) to the finishing bath helps to improve the fabric crease recovery angle. However, the results of fabric crease recovery angle measurements indicated that the presence of citric acid (CA) with or without SHP has no significant influence on the fabric crease recovery angle. This can be attributed to the loose construction of the knitted fabric and the low cotton content of the fabric. However, the fabric crease recovery angle became higher by adding polyurethane resin to the fabric. Further, the influence of the silicone softener on the fabric creases recovery angle

TABLE II
Characteristics of the Samples

Sample no.	Add on (%)	Bending length (cm)		Crease recovery angle (°)	Water droplet absorption time (sec)		Pilling rate	Pill density (pill/cm^2)	
		Course	Wale	Course + wale	Mean	SD		Mean	SD
Control	-	1.50	2.06	218	0	0	1–2	17.6	2.3
1	0.25	1.55	1.92	222	0	0	2–3	8.5	1.5
2	0.46	1.53	1.97	220	0	0	2–3	6.5	1.3
3	1.15	1.53	1.94	217	0	0	2–3	8.0	1.6
4	2.0	1.43	1.85	220	0	0	2–3	7.5	1.4
5	2.8	1.44	1.80	224	0	0	2–3	7.5	1.5
6	0.3	1.54	1.98	223	0	0	2	10	1.5
7	1.2	1.51	1.90	225	0	0	2–3	9.5	1.6
8	1.5	1.46	1.78	224	0	0	2–3	8.5	1.4
9	2.9	1.60	1.92	226	0	0	2–3	8.0	1.5
10	3.1	1.50	1.84	225	0	0	2–3	7.5	1.3
11	1.5	1.56	2.46	241	3.0	0.4	2–3	9.5	1.7
12	2.1	1.73	2.50	264	4.5	1.9	3	5.5	1.2
13	2.2	1.73	2.50	269	5.5	0.7	3	5.5	1.5
14	3.0	1.90	2.52	289	12.5	1.1	3	5.0	1.3
15	2.1	1.66	2.15	251	23.0	2.5	4	3.2	1.4
16	2.4	1.58	2.03	267	81	4.3	2–3	6.2	1.4
17	2.6	1.55	2.00	266	100.5	4.6	2–3	7.0	1.6
18	2.9	1.59	2.08	267	122.5	7.3	2–3	7.0	1.2
19	3.5	1.57	2.14	275	158	7.5	3	4.6	1.5
20	3.4	1.53	2.00	266	69	4.9	4–5	1.4	1.2
21	3.6	1.58	2.08	244	93	6.0	3	5.4	1.6
22	3.7	1.65	2.09	259	98	5.5	4	3.4	1.5
23	3.9	1.60	2.10	256	110	6.7	4–5	2.0	1.4

was not significant too. In general, using three compounds including polyurethane resin, CA, and silicone-based softeners on the fabric helps to increase the fabric crease recovery angle and prevent the fabric from creasing.

Water droplet adsorption time

The water droplet can be adsorbed into the untreated and CA-treated fabrics very quickly and the time of droplet adsorption on these fabrics was close to zero second. The treatment of the fabric with the polyurethane resin changed the water adsorption properties of the fabrics. The resin covers the fabric surfaces and blocks the hydrophilic groups and covers some of the voids between fibers and yarns, creating a polyurethane film on the fabric surface. Accordingly, the accessibility of water to the fabric surface decreased, which caused a small increase in the water droplet absorption time. The increase of water droplet adsorption time in samples 11 to 14 can be related to the high polyurethane resin content. The water droplet adsorption time of sample 15 increased due to the considerable reaction between polyurethane resin, fiber, and acid (Scheme 1), leading to lower accessibility of water to hydrophilic groups. It can be seen in Table II that the water droplet adsorption time increased in the fabric samples treated by higher amount of polyurethane resin along with certain amount of citric acid. It is well known that the hydrophilicity of the fabric is related to the existence of hydrophilic groups, micropores and free surface energy.[8] Increasing the presence of the hydrophilic groups and micropores can help to improve the water absorption and wicking. Also, increasing of resin content with the softener led to an increase in the water droplet absorption

TABLE III
Tensile Strength, Strain and Water Contact Angle of the Sample 2 and 20 and Control

Sample no.	Maximum load (N)		Strain (%)		Water contact angle (°)
	Mean	SD	Mean	SD	
Control	696.41	32.64	60.01	1.64	80.00
2	669.12	16.65	64.43	1.53	75.70
20	619.47	25.72	70.58	2.42	88.04

TABLE IV
Bending Rigidity of Sample 15, 20 and Control

Sample no.	Bending rigidity (mg cm)	
	Course	Wale
Control	65	170
15	91	197
20	72	161

TABLE V
The EDX Analysis Results for Control Sample and Sample 20 (Before and After 10 Washing Cycles)

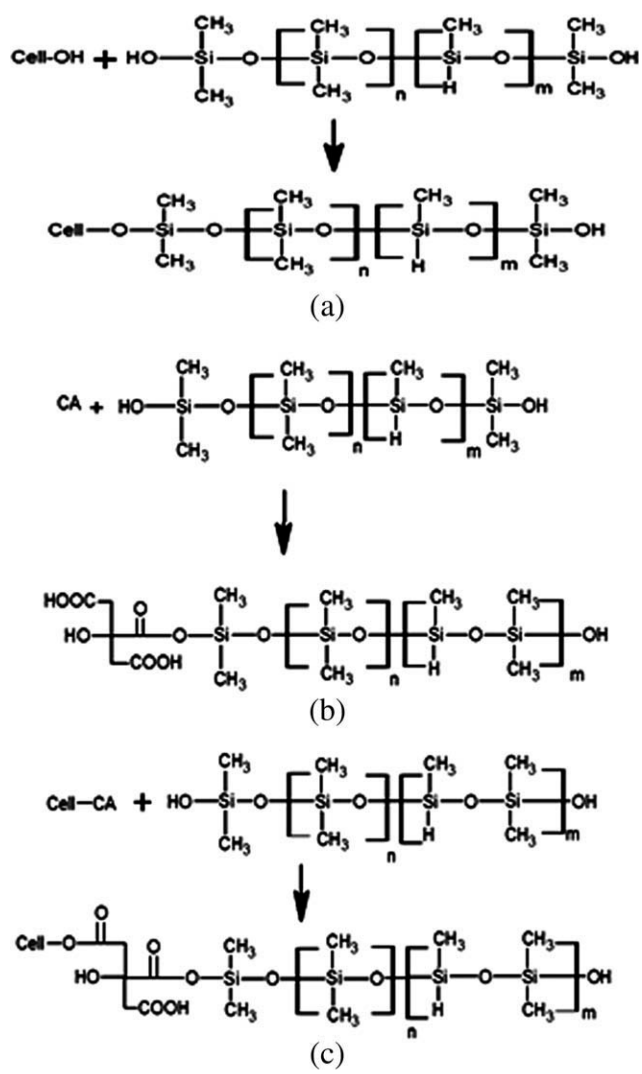
Sample no.	Atomic %	
	Au	Si
Control	100	0.00
20	90.18	9.82
20 (after 10 washing cycles)	93.84	6.16

time. Since the resin can coat the hydrophilic groups of the fabric and reduce the micro pores, it can increase the water droplet absorption time. Further, employing softener on the fabric, which decreases the surface tension of the fabric, will result in water adsorption time increment. It can be seen that increasing of citric acid concentration caused a reduction in the water droplet adsorption time of the sample 16 to 23. This can be attributed to the presence of hydrophilic groups of CA on the fabric surface.[20] However, the influence of CA on water droplet adsorption was different for various fabrics depending upon the composition of the bath. Overall, in co-application of resin, CA, and silicon-based softener, using low CA concentration decreased the hydrophilicity and high CA concentration increased the available hydrophilic groups on fabric surface compared with the low concentration. This can be due to the cross-linking influences of the CA in a way that in low concentration of CA, the carboxylic groups esterifies as a result of cross-linking with other hydroxyl groups but at high concentration of CA, some of the carboxylic groups remaining free and unreacted caused water droplet adsorption. Moreover, the standard deviation (SD) was calculated and reported in Table II.

Pilling rate and pill density

The fabric surface can form pills as a consequence of friction with other surfaces. The results in Table II revealed that the pill density was approximately proportional with the pilling rate. Higher pilling rate indicates a lower pill density. However, the pilling density is more accurate. Table II also indicates that the pilling rates are higher for those samples treated with CA. The low pilling performance of CA-treated samples can be attributed to the irreversible acid degradation.[21] The acid degradation caused weak anchor fibers that can be removed easily, leading to higher pilling rate. However, this was not enough and still anchors fibers remained on the fabric surfaces. It can be seen that increasing of acid concentration above 20 g L^{-1} has no significant influence on the fabric pilling rate. This may be as a result of achieving the same cellulose degradation effect at lower concentrations of CA.

The polycarboxylic acids can crosslink the cotton fibers through esterification reactions.[21,10,22,23] The results of samples 6 to 10 confirmed that SHP has no influence on the fabric pilling rate and pill density. Thus, presumably CA can crosslink the cellulosic chains of cotton without SHP at high temperature or the crosslinking of cotton by CA does not have a significant effect on the fabric pilling performance and acid degradation of the cotton fibers is an important factor. The results of tensile strength in Table II indicated the reduction in the tensile of the treated fabrics that can be due to the acid degradation. Also, it should be considered that the cotton content of the fabric is low and the effect of CA as a cross-linking agent on the fabric pilling rate and pill density are not considerable. Thus, the surface coating with polymeric resin is more helpful to reduce pill density. For this reason, the polyurethane resin (E-POL) which is an appropriate coating with supreme adhesion to the fabric surface in different concentrations was employed. In principle,



Scheme 2 (a) Reaction of cellulose with silicone softener, (b) reaction of citric acid with silicone softener, and (c) reaction of silicone softener with cellulose by means of citric acid.

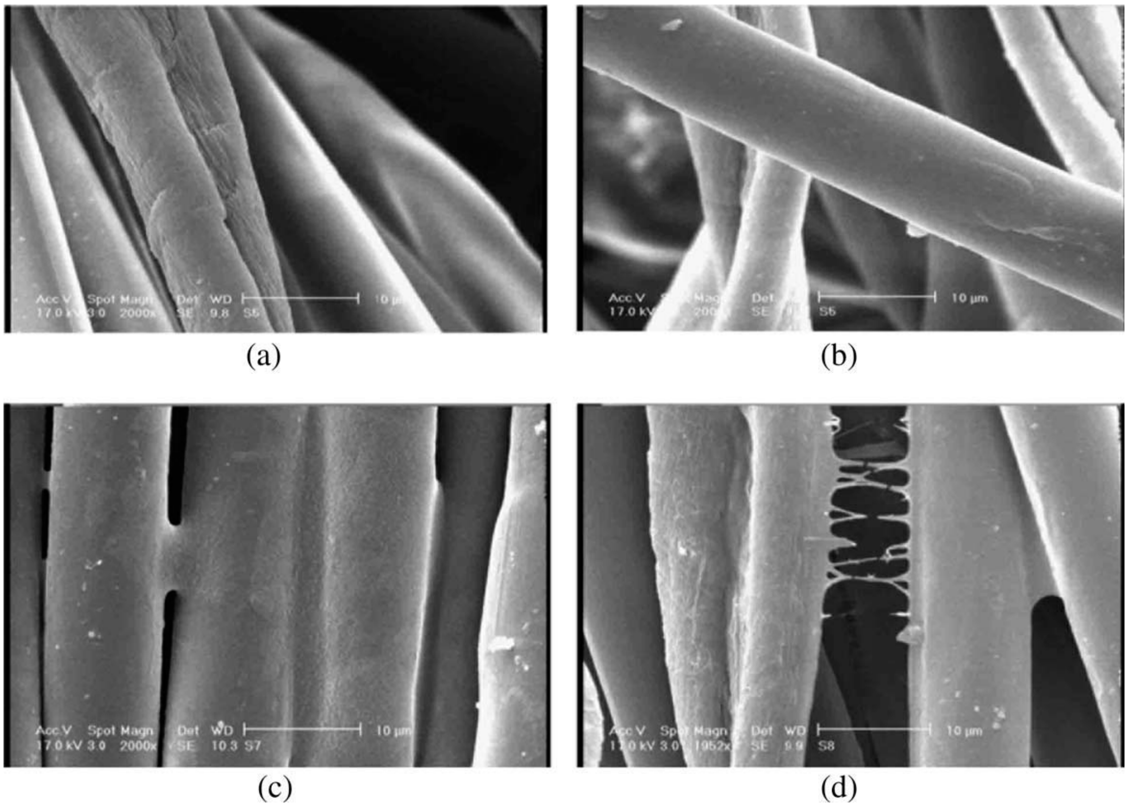


Figure 1 SEM images of control sample (a) cotton (×2000), (b) polyester (×2000), (c) sample 20 before washing (×2000), and (d) sample 20 after 10 washing cycles (×2000).

citric acid may react with a polyurethane resin by the ways of reacting with free OH, urethane bonding (if the reaction condition is favorable), or physically bond with the polyurethane chain. Thus, the polyurethane resin not only may coat the fabric surface and bind the fibers together but also may react with citric acid [Scheme 1(b)] and also may attached to the cellulose through CA cross-linking [Scheme 1(c)].[22] This causes grafting and crosslinking of polyurethane, CA, and cellulosic chains and binding the fibers together, decreasing fabric pill density (Scheme 1).

The pilling rate was improved for the samples treated with the resin alone; however, this was not enough for clothing and still some pills can appear on the fabric surface. The results of the samples treated with blend of resin, softener, and CA indicated that increasing CA concentrations leads to reduce pill density. By comparing the samples treated with and without softener, it can be realized that the presence of the softener caused an increase in the fabric pilling rate. Considering the fact that formation of the pill occurs in several steps, the softener has influence in two steps. First, the silicone softener can act as a resin and react with cellulose and citric acid [Scheme 2(a-c)] to bind the fibers in the fabric prevented the outgoing of the fibers from the fabric surface. Second, it helps to reduce the friction between the fibers and other surfaces consequently preventing the entanglements of the fibers and pill formation.[15] Because the softeners frequently increase the flexibility of the fibers, they result in an increase in longevity of the pills on the fabric surface. However, in these experiments, the presence of the resin and citric acid reduced this effect. It can be concluded that the resin concentrations used in samples 20, 22, and 23 were enough to create a good adhesion between the fibers and hold hairs in fabric construction together, preventing pill formation as a consequence of friction with other surfaces. The above results revealed that the sample 20 was the best sample with regard to the softness and antipilling. In order to evaluate the washing fastness of the treated sample, sample 20 was washed 10 cycles at 60°C for 20 min, and then pilling test was carried out on the washed sample. The pill density of sample 20 after 10 washing cycles was 3 with pilling rate of 4. This showed the reasonable fastness for antipilling performance of the treated sample. Further examinations such as SEM micrographs and EDX analysis have been also carried out on this sample.

Tensile strength and strain

The fabric tensile strength was measured in the wale direction for the selected fabric samples. The results for the samples 2, 20, and control are indicated in Table III. The higher tensile strength value belonged to the untreated sample. The application of the resin and citric acid on the cotton/polyester knitted fabric reduced the tensile strength. This can be considered due to the crosslinking of the cellulosic chains and thus internal stress concentration. Furthermore, the reduction of tensile strength can be attributed to the acid degradation of cellulose content.[15,22] This helps to break the fibers easier and reduce the fabric pill density. The tensile strength of sample 20 was lower than sample 2 and its strength reduced around 10% compared to untreated sample. This can be related to the random binding of the fibers together in the fabric construction, leading to prevent slippage and easy breakdown with a lower tensile strength of fibers as a consequence of stress concentration.

The highest strain value belonged to sample 20, as it had the least tensile strength. The application of resins on the surfaces of cotton/polyester fabrics increased the strains. However, the effect of resins on tensile strength was not significant. Overall, the treatment of resin on the cotton/polyester fabric did not have much influence on the mechanical properties of the fabric. Thus, the reduction of pill density is mainly due to the adhesion of fibers and yarns together in the fabric construction.

Water contact angle

The results in Table III revealed that the presence of citric acid on the fabrics led to produce a fabric with higher hydrophilicity than the control fabric. It has been reported that the treatment of cotton fabrics with polycarboxylic acids leaves the fabric with more carboxyl groups on the surface, absorbing higher amount of water,[19] but the polyurethane resin reduced the hydrophilicity of the fabric. Thus, the contact angle increased for those treated fabrics with polyurethane resin.

Qualitative analysis of handle

Qualitative analysis is one of the methods used to evaluate the fabric handle.[2] Fifteen assessors were used in order to rank the softness of the two different fabrics including sample 15 without softener and sample 20 with softener. All the assessors evaluated that sample 20 is softer than sample 15. In order to carry out a quantitative assessment, we evaluated the bending rigidity of the two samples from eq. (3). From Table IV it is obvious that despite the higher add on % of sample 20, it is softer than sample 15. Also, the results show that in wale direction the bending rigidity of sample 20 is lower than control sample. Thus, the sample 20 is soft enough.

$$G = WC^3 \times 10^3 \text{mg} \cdot \text{cm} \qquad (3)$$

where G is bending rigidity, W is fabric weight, and C is bending length.

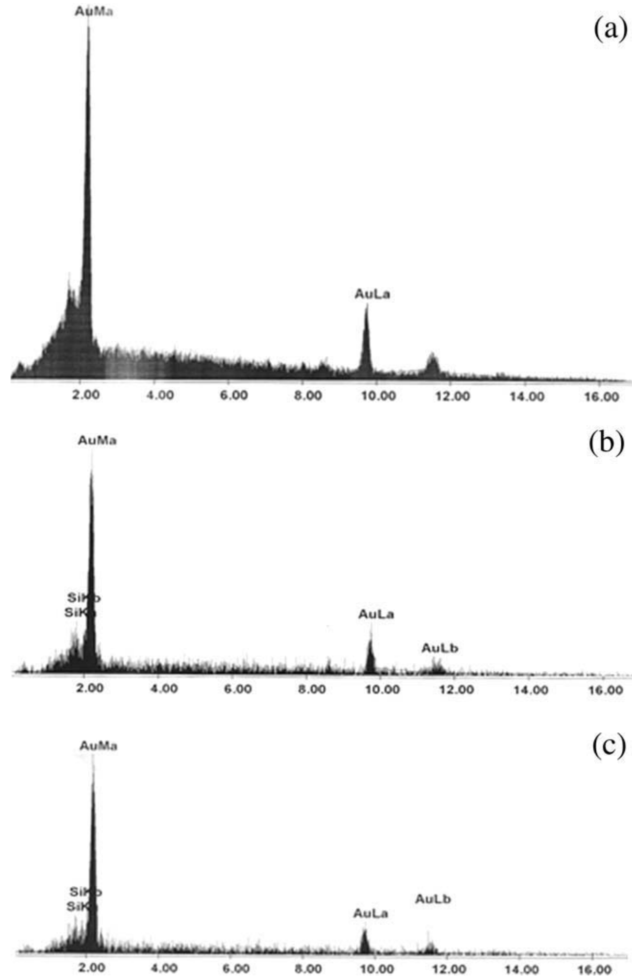


Figure 2 EDX analysis of (a) sample control, (b) sample 20, (c) sample 20 after 10 washing cycles.

SEM images

The SEM picture of the control sample is shown in Figure 1(a,b). It can be seen that the untreated sample has a clean surface without any additives on the surface. It can be also observed that the fibers in sample 20 [Fig. 1(c)], which has been treated with resin, softener, and citric acid, are greatly adhered together. The SEM pictures were indicated the presence of polyurethane resin on the surface of the fibers. The picture in Figure 1(c) also reveals that considerable amounts of resin on the surface of the fibers bind them together, preventing them from coming out the fabric surface and creating fabric pills. In addition, the SEM pictures in Figure 1(d) confirmed that the resin has remained on the fiber surface even after 10 washing cycle. On these bases, this sample can stand against cyclic washings.

EDX analysis

EDX analysis accomplished for illustrating the presence of silicone softener on the control sample and sample 20 (before and after 10 washing cycles). The percentage of Si with respect to Au is evaluated. The results are shown in Figure 2 and Table V. No Si was recorded for the control sample [Fig. 2(a)]. The presence of Si on the silicone treated samples was confirmed and also the presence of Si on the sample 20 even after 10 washing cycles was approved [Fig. 2(b,c)].

CONCLUSION

In this research, a simple method for producing cotton/polyester knitted fabric with a durable soft and smooth surface was introduced. An optimized combination of a water-based polyurethane resin, a carboxylic acid cross-linker along with a silicone based softener was introduced. The optimized formulation obtained was 25 g L^{-1} E-POL, 20g L^{-1} citric acid, and 20 g L^{-1} silicone softener (Adrasil AF). This leads to producing a fabric with acceptable low pilling performance and desirable handle properties. The obtained properties were durable to repeated washings, and the treated fabrics had a better resistance against creasing.

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