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Multifunctional finish and cotton cellulose fabric

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ABSTRACT

Some commercial textile softeners were incorporated during the finishing of cotton fabrics with citric acid (CA) leading to alteration in performance properties of that fabrics. The obtained results showed that finishing of these fabrics with 80 g/l of aqueous CA in presence of 20 g/l of silicone micro emulsion (SME) followed by drying at 85 °C for 3 min, cured at 180 °C for 90 s resulted in crosslinking as well as an improvement in some performance properties of those fabrics such as nitrogen content, resiliency, tear strength and softness of the finished fabrics accompanied with decreasing of the whiteness indices and absorbency of that fabrics. Moreover, infrared spectrum as well as TGA analysis of CA/SME finished fabrics was investigated. Furthermore, the CA/SME finished fabrics were able to keep the Rosemary, Jasmine, Lavender and vanillin perfumes upon storing up to 3 months or after ten repeating washing cycles.

1. Introduction

Crosslinking of cotton fabric by polycarboxylic acids such as citric acid have proven to be the most effective substitutes for the formaldehyde-releasing N-methylol compounds (Hebeish, Hashem, Abdel-Rahman, & El-Hilw, 2006; Kim, Jang, & Ko, 2000). However, crosslinking of cotton fabrics with polycarboxylic acids improves the wrinkle resistance; it reduces the mechanical strength and yellows the treated fabrics (Bajaj, 2002; Hebeish et al., 2006; Ibrahim, Abo-Shosha, Elnagdy, & Gaffar, 2004; Kim et al., 2000; Zhou, Yang, & Lickfield, 2004). Several studies have been made to decrease these disadvantages (Hebeish et al., 2006; Ibrahim, Abo-Shosha, et al., 2004).

On the other hand, softeners have been reported to produce the softest possible hand to improve crease recovery, tear & abrasion resistance and they are excellent for improving sewing properties of fabrics (Habereder, 2002; Sand, Brückmann, & Zyschka, 2000; Thoss, Hesse, Höcker, Wagner, & Lange, 2003). Because of these functional reasons, softener chemicals are included in nearly every finishing formulation applied to fabrics (Tomasino, 1992).

Moreover, finishing of textiles with fragrances has been carried out for many years in the form of fabric conditioners in washing and during tumble-drying; all are designed to impart a fresh aroma to the textile. However, no matter the quality of the technology used to impart the fragrance, the effect is relatively short-lived. Numerous attempts have made to add fragrances directly to fiber and fabrics but all fail to survive one or two wash cycles. Only through microencapsulation, fragrances are able to remain on a garment during a significant part of its lifetime. Using this technology, many companies have manufactured new fabrics that emit the natural aroma of flowers, fruit, herbs and perfumes for up to 25 wash cycles and on the shelf, the finish will remain ready for action for between 3 and 5 years (Nelson, 2002).

Keeping in mind that background, the present work is considered a trial to reduce the extent of the disadvantages of crosslinking of cotton fabrics with citric acid as well as to impart perfume finishing for those fabrics.

2. Experiment

2.1. Materials

Mill-scoured-bleached cotton fabric of 147 g/m^2 was used. Citric acid (CA) and sodium hypophosphite monohydrate (SHP) were of reagent grade. Non-ionic wetting agent (Egyptol®), supplied by the Egyptian Company for Starch and Yeast and Detergents – Alexandria – Egypt. The commercial softeners: Basosoft® SWK (weakly cationic mixture of a fatty acid condensation product and polyethylene wax, supplied by Basf), Leomin® NI-ET (nonionic, hydrophilic, and supplied by Clariant) and Silicon-SLH® (micro silicon emulsion) (SME) – supplied by (Texchem Egypt Co., Ltd) were used. Four commercial oily perfumes made in France were used which are jasmine, lavender, Rosemary and vanillin.

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Table 1
Effect of softener type on some performance properties of treated cotton fabrics.

Softner Type	% N	WRA (w + f)°	TS (Retained %)	SR	WI	W (S)
Untreated	0	135	100	15.36	114	2.1
Control	0	183	68.6	16.25	105	1.5
Leomin® NI	0	195	72.8	15.18	106	2.5
Basosoft® SWK	0.05	209	76.1	14.73	101	34.1
Silicon® SLH	0.04	223	78.3	14.13	99	74.3

[Softener], 20 g/l; [CA], 80 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s. Control, cotton sample estrified with CA in absence of any of softeners.

2.2. Fabric treatments

2.2.1. Finishing of cotton fabrics with CA/SHP and softeners

Fabrics were boiled in an aqueous solution containing 2 g/l non ionic detergent for 30 min, thoroughly rinsed and dried at ambient conditions. After that, fabrics were padded in finishing baths containing different concentrations of CA/SHP and/or different types of textile softeners to a wet pick up of 90%, followed by drying at 85 °C/3 min, and cured at 180 °C for 90 s in a circulating air oven. The cured cotton fabrics were then rinsed with distilled water at 50 °C for 30 min, dried at ambient room temperature prior to evaluation.

2.2.2. Finishing of cotton fabrics with perfumes

CA/SHP and/or softeners finished fabrics were padded in an alcoholic solution of perfume oil (200 g/l) to a wet pick up of 100% followed by drying at ambient conditions for 2 h before testing.

2.3. Testing and analysis

Nitrogen content was determined according to Kjeldahl method (Vogel, 1975). Carboxyl content was determined according to the Cirino method (Cirino, 1975). Dry wrinkle recovery angles (WRA) were determined by the ASTM method D-1296-67 using the iron recovery apparatus, type FF-07 (Metrimpex). The Elmendorf tear strength (TS) was determined, in the warp direction, according to ASTM: D-1424. CIE Whiteness Index (WI) was measured according to AATCC test method 110-1989 using a Milton Roy Color Mate spectrophotometer. Surface Roughness (SR) was determined according to JIS 94 Standard test method using the Surface Roughness Measuring Instrument, SE 1700a. The absorbency test (W) was carried out according to AATCC test method 79-1992. The ability of the fabric to keep fragrance upon storing under ambient conditions in stagnant air for 1 week, 1 month, 2 months and 3 months was determined by:

- Evaluating the % loss in weight gained after the desired time as follows: % loss in weight = $100(B - C)/(B - A)$where A is the weight of CA/SME finished fabric before padding in the perfume oil solution, B is the weight of CA/SME finished fabric after padding in the perfume oil solution and C is the weight of CA/SME finished fabric upon storing.
- Evaluating the scent intensity of the perfumed fabrics through comparing the scent intensity of the perfumed fabrics stored in air for a known time to that of a similar sample stored in a refrigerator in a zip-locked plastic bag. Comparison was achieved independently by three persons, using the smelling sense, and the average of their estimation was recorded.

  Durability to wash was tested by washing the perfumed fabrics for 1, 5 and 10 washing cycles using 1 g/l nonionic wetting agent in automatic washing machine (Fresh L-411A) for 10 min at 30 °C followed by evaluating the scent intensity of those fabrics.

3. Results and discussion

It has been established that cotton fabrics can be crosslinked with polycarboxylic acids through an anhydride intermediate mechanism (Cirino & Rowland, 1976) in the presence of sodium hypophosphite monohydrate (SHP) as a catalyst, accompanied with two main disadvantages which are the yellowness as well as the reduction in the mechanical properties of the finished fabrics. Incorporation of textile softeners, as additives, in the CA/SHP finishing bath may alter the performance properties of the crosslinked fabrics. The main task of the present work is to evaluate the impact of these softeners, as additives in the CA/SHP finishing baths, on some performance properties of the finished fabrics. Moreover, factors affecting the finishing process, such as softener type and concentration as well as, CA concentration have been studied. Results are obtained along with their appropriate following discussion:

3.1. Effect of softener type

Table 1 shows the effect of incorporation of different textile softeners (20 g/l) in the CA finishing bath (80 g/l), of cotton fabrics. It is clear that incorporation of such softeners in the CA/SHP finishing bath is accompanied by a noticeable increase in nitrogen content as well as resiliency, except with Leomin® NI, tear strength, and softness of the finished fabrics along with decreasing of whiteness and absorbency of that fabrics occur. The altering in magnitude of the aforementioned properties is governed by nature of the softening agent and follow the decreasing order: Silicon® SLH (SME) > Basosoft® SWK > Leomin® NI which reflect the differences among the aforementioned softeners in the chemical composition, mode of interaction, location and extent of surface modification, along with a reduction of interfibers and interyarns friction. Consequently, affecting the resiliency, tear strength, smoothness, whiteness indices and absorbency (Welch & Andrews, 1994) of the finished fabrics. On the other hand, the decrease in the extent of ester crosslinking of the finished fabric in presence of Leomin® NI may be related to an increase in the viscosity of the finishing bath, thereby hindering the diffusion and penetration of finishing agent within the fabric and hence altering the values of the aforementioned properties (Ibrahim, Fahmy, Hassan, & Mohamed, 2005).

3.2. Effect of silicon® SLH (SME) concentration

Table 2 shows the effect of SME concentration on the performance properties of the finished fabrics. It is clear that by increasing the concentration of SME (0–30 g/l) in the finishing bath, results in an enhancement in the nitrogen content, resiliency, tear strength as well as softness of the finished fabrics accompanied with decreasing of the whiteness indices and absorbency of those fabrics. This can be attributed to the enhancement of the thickness of silicone film deposited onto the fabric structure and hence, altering the aforementioned performance properties. Moreover, the lowering in the whiteness indices of the finished fabrics in presence of SME, compared to that finished in its absence may be attributed to the

Table 2
Effect of SME concentration on some performance properties of treated cotton fabrics.

SME (g/l)	% *N*	WRA (*w*+*f*)°	TS Retained (%)	SR	WI	*W* (*S*)
Control	0	195	68.6	16.25	105	1.5
10	0.02	207	75.1	15.43	101	26.2
20	0.04	223	78.3	14.13	99	74.3
30	0.07	228	80.1	12.59	98	87.5

[CA], 80 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s. Control, cotton sample estrified with CA in absence of SME.

Table 3
Effect of CA concentration on some performance properties of treated cotton fabrics.

CA (g/l)	% *N*	WRA (*w*+*f*)°	TS Retained (%)	SR	WI	*W* (*S*)
Control	0.07	184	95.6	12.07	107	80.6
50	0.05	198	89.4	12.69	103	78.3
65	0.05	212	82.6	13.54	100	76.1
80	0.04	223	78.3	14.13	99	74.3

[SME], 20 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s. Control, cotton sample treated with 20 g/l SME.

thermal curing of the treated fabrics with SME softener, with its cationic sites, which leads to oxidative decomposition of the amino group forming chromophoric groups (Ahmed, Fahmy, & Abd El-Aziz, 2011). On the other hand, the resiliency of the finished fabric at concentration of 20 g/l of SME reaches higher value than the other SME concentrations which may be attributed to the formation of ionic bonds between the single ended carboxyl groups of the finished fabric and the cationic centers of the SME softener (Bajaj, 2002).

3.3. Effect of CA concentration

Table 3 shows the effect of citric acid concentrations (50–80 g/l) on the performance properties of treated cotton fabrics. For a given set of conditions, it is clear that: (i) finishing of cotton samples with SME softener, in absence of citric acid, increases the nitrogen content, resiliency, softness of treated samples along with decreasing the tear strength, whiteness indices and absorbency of those fabrics, and (ii) raising the CA concentration from 50 to 80 g/l in the finishing bath containing SME softener, 20 g/l, enhances the nitrogen content, resiliency and the absorbency of the treated fabrics along with the reduction in the tear strength, softness and whiteness indices of those fabrics which could be related to increasing the extent of the ester crosslinking of the cellulosic hydroxyl groups. Moreover, increasing the concentration of citric acid in the finishing bath results in increasing of hydrophilicity of the treated fabric via increasing the single ended carboxyl groups bound to the fabric structure (Fahmy, 2004; Fahmy & Fouda, 2008; Hashem, Ibrahim, El-Shafei, Refaie, & Hauser, 2009).

3.4. Characterization of CA/SME finished fabric

The cotton fabric finished with citric acid (80 g/l) in combination with SME (20 g/l) was characterized by investigating its FTIR spectra as well as thermal gravimetric analysis.

3.4.1. FT-IR spectra

Fig. 1 shows the FT-IR spectra of the aforementioned CA/SME finished fabrics. It is obvious that the most characteristic peaks in Fig. 1 are 3347 cm^{-1} confirming the presence of alcoholic –OH stretching, 2901 confirming aliphatic –CH2 stretching, 1163 cm^{-1} confirming –C–O–C– asymmetric bridge stretching, 1031 cm^{-1} confirming –C–O stretching, 1730 cm^{-1} confirming the presence of –C=O stretching of carboxylic acid, 2927 cm^{-1} corresponding to C–H stretching of methyl or methylene group, 1640 cm^{-1} corresponding to an ester carbonyl band of citric acid, 1262 cm^{-1} corresponding to Si–CH3 symmetric deformation and 1636 cm^{-1} assigned to the vibration absorption of amine C–N bond.

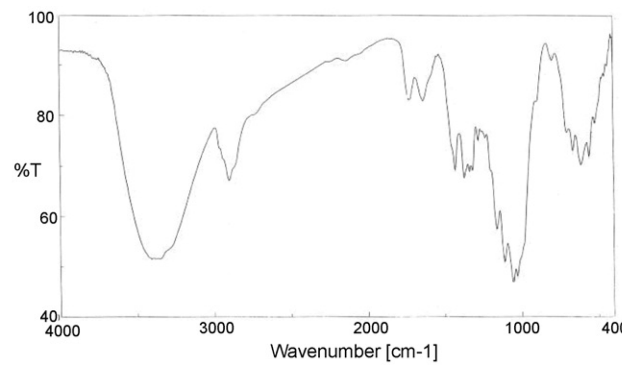

Fig. 1. FT-IR spectra of CA/SME finished cotton fabric.

3.4.2. Thermal gravimetric analysis

Fig. 2 shows three curves representing the thermal gravimetric curves (TGA) of untreated, CA and CA/SME finished cotton fabrics. It is obvious that the TGA of each curve consists of three parts; the first one is a dehydration stage which starts from 50 °C and ended at 230.8, 213.6 and 187.8 °C with a loss of weight 2.909, 4.448 and 3.495% for untreated, CA and CA/SME finished cotton fabrics, respectively. The second part is a thermal degradation stage resulting from pyrolysis which starts from 230.8, 213.6 and 187.8 °C and ended at 322.7, 314.1 and 305.4 °C with loss of weight of

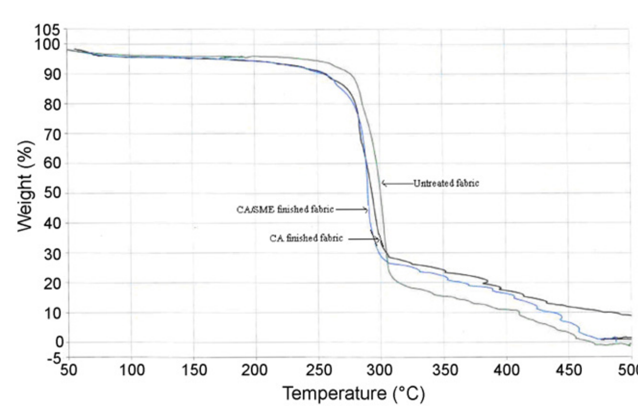

Fig. 2. Thermal gravimetric analysis of untreated, CA and CA/SME finished cotton fabrics.

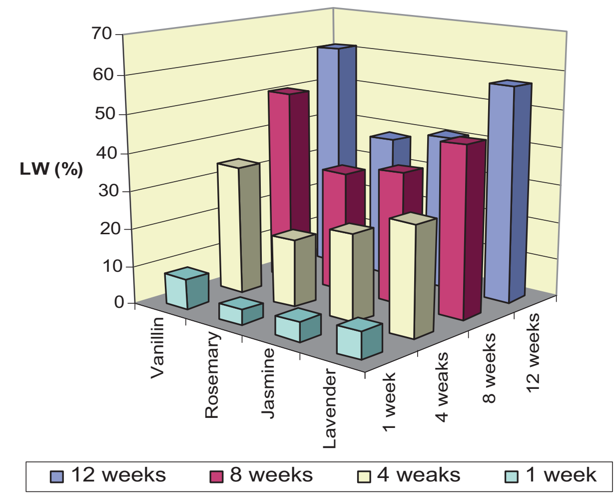

Fig. 3. Effect of perfume type on the percent loss in its weight upon storing the perfumed CA/SME finished fabrics. [SME], 20 g/l; [CA], 80 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s; padding in an alcoholic solution of perfume oil (200 g/l) to a wet pick-up of 100% and air drying for 2 h; LW (%), percent loss in the weight of perfume oil.

76.868, 66.037 and 67.758% for untreated, CA and CA/SME finished cotton fabrics, respectively. The third part represents the conversion of the remaining materials to carbon residues which ends at 470.7, 445.0 and 480.5 °C with weight loss of 19.723, 27.274 and 25.842% for untreated, CA and CA/SME finished cotton fabrics, respectively. From the above discussion, it can be concluded that the residual weights at the end of the combustion process are 0.5, 2.241 and 2.905% at final degradation temperatures of 470.7, 445.0 and 480.5 °C for untreated, CA and CA/SME finished cotton fabrics, respectively, reflecting the thermal stability of CA/SME finished fabric compared to that of CA finished fabric.

3.5. *Perfume finishing of CA/SME finished fabric*

3.5.1. *Effect of perfume type*

Fig. 3 and Table 4 show the effect of perfume type on both the percent loss in weight as well as scent intensity, respectively, of that perfumes upon storing the perfumed fabrics. It is obvious that:

(i) The ability of the CA/SME finished fabrics to keep the aforementioned fragrances decreases by increasing the storing time up to 3 months.
(ii) The ability of the perfumed CA/SME finished fabrics to keep perfumes can be arranged, in terms of the percent loss of their weight, as the following: Rosemary > Jasmine > Lavender > vanillin

Table 4
Effect of perfume type on its scent intensity upon storing the perfumed CA/SME finished fabrics.

Perfume type	Storing time			
	1 week	1 month	2 months	3 months
	Scent intensity			
Lavender	+++++	+++	++	+
Jasmine	+++++	++++	++	+
Rosemary	+++++	++++	++	+
Vanillin	+++++	+++	+	Nil

[SME], 20 g/l; [CA], 80 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s; padding in an alcoholic solution of perfume oil (200 g/l) to a wet pick-up of 100% and air drying for 2 h. +++++, express very strong; ++++, express strong; +++, express common; ++, express weak; +, express very weak.

Table 5
Effect of the repeated washing on the scent intensities of CA/SME perfumed-finished fabrics with different perfumes.

Perfume type	Scent intensity		
	Washing cycle		
	1	5	10
Lavender	++++	+	Nil
Jasmine	++++	++	+
Rosemary	++++	++	+
Vanillin	+++	+	Nil

[SME] 20 g/l; [CA], 80 g/l; CA/SHP molal ratio, 1; wet pick up, 80%; drying, 85 °C/3 min; curing, 180 °C/90 s; padding in an alcoholic solution of perfume oil (200 g/l) to a wet pick-up of 100% and air drying for 2 h. +++++, express very strong; ++++, express strong; +++, express common; ++, express weak; +, express very weak.

This may be attributed to the action of the oleophobic segments of SME softener to solubilize and/or encapsulate the Jasmine oil, then release it slowly so that its smell can be sensed (Ibrahim, El-Sayed, Fahmy, Hasabo, & Abo-Shosha, 2004). It must be noted that the perfumes scent disappear when the percent loss in its weight reaches 75% which can be attributed to the composition of a perfume oil; that is an artificial oil is usually composed of a base oil (usually of no fragrance and of relatively high vaporization point) in which other volatile oils (responsible for fragrance and volatile easily) are dissolved (Ibrahim, El-Sayed, et al., 2004).

3.5.2. *Durability of perfumed CA/SME finished fabrics*

Table 5 shows the effect of washing cycles on the scent intensity of the CA/SME perfumed-finished fabrics with Lavender, Jasmine, Rosemary and vanillin fragrances. It is clear that:

(i) The loss in scent intensity of these perfumes increases by increasing the number of washing cycles.
(ii) These perfumes can be arranged in terms of the ability of perfumed fabrics to keep them with repeated washing (up to 10 washing cycles) as follows:

Rosemary ≈ Jasmine > Lavender ≈ vanillin

These results reflect the ability of the CA/SME finished fabrics to keep and release these fragrances in spite of that number of washing cycles.

4. Conclusion

1. Finishing of cotton fabrics with CA/SHP in presence of softeners increases the nitrogen content as well as resiliency, except with Leomin NI; tearing strength, and softness of the finished fabrics along with decreasing of whiteness, absorbency of those fabrics compared with the only CA finished fabrics occur.
2. The altering in magnitude of aforementioned properties can be arranged, according to the softener type, in the following decreasing order: Silicon® SLH > Basosoft® SWK > Leomin® NI.
3. Increasing the concentration of SME (0–30 g/l), in the finishing bath in presence of 80 g/l CA, results in an enhancement in nitrogen content, resiliency, tearing strength as well as softness of the finished fabrics accompanied with decreasing of whiteness indices and absorbency of those fabrics.
4. Raising the CA concentration from 50 to 80 g/l in the finishing bath containing SME softener, 20 g/l, enhances the nitrogen content, resiliency and the absorbency of the treated fabrics along with the reduction in tearing strength, softness and whiteness indices of those fabrics.
5. The CA/SME finished fabrics were able to keep the perfumes upon storing, than the CA treated and untreated samples.

6. The ability of the CA/SME finished fabrics to keep fragrance decreases by prolonging storing time up to 3 months.
7. The ability of the CA/SME finished fabrics to keep perfumes can be arranged according the fragrance type, in terms of the percent loss of their weight, as follows:

   Rosemary > Jasmine > Lavender > vanillin.

8. The loss in scent intensity of the aforementioned perfumes increases by increasing the number of washing cycles.

   These perfumes can be arranged in terms of the ability of perfumed fabrics to keep them with repeated washing (up to 10 washing cycles) as follows:

   Rosemary ≈ Jasmine > Lavender ≈ vanillin

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