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ABSTRACT

The objective of this paper is to measure the deposition of solid impurity on finished cotton fabric with DMDHEU resin treated during washing cycles of cotton fabric treated with different softeners. Fabrics will be softened with the conventional esterquat and with aminosilicone products having different molecular weights and mixtures of both. The anionic surfactant sodium dodecyl benzenesulphonate and non-ionic surfactant fatty alcohol oxyethylenated were used. Both separately and in different proportions during the deposition trials were used. In general, the deposition values decrease on increasing the quantity of non-ionic surfactant in the mixture and with the finished cotton fabric increase this one after the applicability of the softeners tested alone and with their mixtures.

Key words: Deposition, washing, finish, softeners, cotton, surfactants, mixtures of surfactants and mixtures of softeners,

1. INTRODUCCION

Fabric softeners offer textiles added properties which are desirable to consumers; such properties include improvements to the handle of the fabric, and making fabrics softer and smoother, while improving flexibility, compressibility and elastic recuperation(1)(2).

Resins are applied to cotton fabric finishing processes, in order to improve their crease recovery; this results in a substantial loss of fabric smoothness in addition to a loss in the
mechanical properties of the fabric. Such problems make it appropriate to use softeners on fabrics (3).

Many studies about the influence of the softeners on the textile properties have been published, the majority of them centred on the influence of the softeners on the handle of the fabric, water absorption, static electricity, abrasion, whiteness, flame retardancy, creasy recovery, the easy ironing effect, durable press, and soil release (4)(5)(6)(7). They do not deal with fabric depositions of solid impurities.

The objective of this work is to analyse the deposition behaviour of solid impurities on finished cotton fabric with DMDHEU resin treated with different cationic softeners Esterquat and ammoniosilicone softeners of different molecular weights were applied to the finished cotton fabric, separately and in mixtures Deposition trials of carbon black during washing cycles and in presence of anionic and non-ionic surfactant and their mixtures were performed. This anionic surfactant sodium dodecil benzenesulphonate and non-ionic surfactant fatty alcohol oxyethylenated were used. Deposition trials of carbon black during washing cycles and in presence of anionic and non-ionic surfactant and their mixtures were performed.

2. EXPERIMENTAL PART.

2.1 Materials.

2.1.1 Fabrics.

The following fabric was used
Cotton fabric 100% having plain structure and scoured and chemically bleached (without optical bleaching). Its characteristics are

a) weight: 176 g. m\(^{-2}\)
b) warp: 34.5 Tex 1/c and weft: 46.3 Tex 1/c
c) fabric density: warp: 28 yarns.cm\(^{-1}\) and weft: 24 yarns.cm\(^{-1}\)

2.1.2 Chemicals.

2.1.2.1 Surfactants.

The following surfactants were used:

Anionic surfactants: Sodium dodecyl bezenesulphonate (SDBS) reagent for analysis supplied by Sigma.

b) Non-ionic surfactant: Fatty alcohol with 6 m.O:E. (Synperonic 91/6) supplied by ICI-Mevisa S.A.(Spain), analitycal purity higher than 99.7 %. Hydroxyl number 134 mg KOH.g\(^{-1}\)

2.1.2.2 Softeners

The softeners employed in microemulsion were the following:

a) Distearylamidoethylmethylimidazoline mehylsulphate (DSEAMIMS) Rewoquat W 75 H supplied by Witco S.A. (Barcelona): 75 % active material, average m.w. 732 The microemulsion had the following initial concentration: 34.1 g.L\(^{-1}\)
b) NI 3474 de Hansa Finish, copolymer consisting of polydimethyl siloxanes and organic quaternary group with molecular weight of 6.000 g.mol\(^{-1}\). Solid quantity of the polymer: 95%. The microemulsion had the following initial concentration(8): 34.4 g.L\(^{-1}\).

c) NI 6922 de Hansa Finish, copolymer consisting of polymethylsiloxanes and organic quaternary groups with molecular weight of 2.500 g.mol\(^{-1}\). Solid quantity of the polymer: 50%. The microemulsion had the following initial concentration(8): 34.4 g.L\(^{-1}\).

The particle size of the microemulsions products was less than 100 nm, determined on a Zetasizer III light scattering apparatus (Malvern, UK).

2.1.2.3 Finish

Cotton was finished with the resin 1,3 dimethyl 4,5 dihydroxyethylen urea (DMDHEU), Fixapret CP, supplied by BASF

2.1.2.4 Soils.

Carbon black Raven 1040, made by Columbian was used with a particle diameter of 29 nm and a surface area of 85 m\(^2\) g\(^{-1}\) of negative charge. For its application in washing it was adequately dispersed in 2-propanol with an ultrasonic shaker.

2.1.3 Water hardness.
For washings, water of 20º hf was prepared by using distilled water and filtered in reverse osmosis filter. and salts of MgCl₂.6 H₂O with purity of 99% supplied by Panreac and CaCl₂ with 95% purity supplied by Panreac were added (9).

2.2 Apparatus

2.2.1 Deposition trials.

The deposition trials during washing were performed in, a Launder-Ometer model LHT of Atlas Electric Device Co. USA was used.

2.2.2 Reflectance measurements

Fabric measurements reflectance were measured in a spectrophotometer White-Eye 3000 supplied by Macbeth (England).

2.3 Procedures.

2.3.1 Application of the finishing resin

As a finish, 100 g.L⁻¹ the reactive resin was applied to a sample material in the presence of 20 g.L⁻¹ of magnesium chloride. This resin was applied in a padder 95% squeezing and then it was dried and cured at 160ºC during 4 minutes.

2.3.2 Application of the softener
A perforated stainless steel hollow cylinder 16 cm long and 2.5 cm in diameter was introduced into the flask, around which was wrapped the cotton fabric. The cylinder was attached to a stirrer (type RW 20DZM, Ika-Werk, Germany) and along with the fabrics rotated at a constant speed of 149 r.p.m. in the softening bath.

The sorption of the softeners on finished cotton fabrics, was under the following conditions:

a) The total concentration of the softeners used was 0.3 % o.w.f.

b) Temperature 40ºC.

c) Liquor ratio: 15:1.

d) pH= 6

e) Time : 20 min.

The exhaustion of the softener remaining in the bath was determined after each sorption trial. This was done by a two-phase evaluation method (10), averaging at least three measures. The exhaustion for Rewoquat W 75 H was about 85 % and for aminosilicone about 75 %.

The softeners Rewoquat, HF 3474 and HF 6922 were used alone and with mixtures of both. The mixtures of Rewoquat with HF 3474 and mixtures of Rewoquat with HF 6922 were in the percentage proportions: 0.75:0.25; 0.5:0.5 and 0.25:0.75 respectively. Rewoquat W 75 H (cationic) was applied on the fabric in first place and later the aminosilicone (non-ionic), each one in the indicated proportions at the same total concentration of softeners.

2.3.3 Deposition methods

Deposition was carried out according to the specifications of ISO Standard 105-106 /DAD

1. Fabric specimens (10 x 4) cm were cut. The washing temperature was 30ºC, and the
process time was 30 minutes. The volume in the washing bath was 150 ml. The amount of
carbon black introduced in the washing was 10 mg, adequately dispersed in 2-propanol.
The water hardness was of 20ºhf.

2.3.4 Assessment of the degree of soiling

The degree of soiling was determined by the Florio and Merserau (11) equation, from the
triistimulus values resulting from the fabric reflectances before and after washing. Such
equation is the following:

\[ \Delta C = \sqrt{\left( X_s - X_p \right)^2 + \left( Y_s - Y_p \right)^2 + \left( Z_s - Z_p \right)^2} \]

where \( X_p, Y_p \) and \( Z_p \) are the triistimulus values of the white sample before the washing
and \( X_s, Y_s \) and \( Z_s \) the triistimulus values of the sample after the deposition or soiling test.
These values are averages of four reflectance readings, the sample being rotated through
90º before each reading.

2.4 Testing.

The deposition of carbon black in washing of the cited fabrics at 40ºC, with the total
concentration of the surfactants of 2 x 10^{-3} M was carried out. In the detergency testing
the same total concentrations of the surfactants were used.

Each deposition result was the average of two identical tests, carried out, after the addition
of carbon black to the washing bath, the fabric was previously immersed in the test
solution for 40 minutes.
The ratios between the surfactants in the proportions of anionic with non-ionic mixtures were the following: 1:0 ; 0,75:0.25; 0.5:0.5 ; 0.3: 0.7 ; 0.1: 0.9 and 0:1.

3. RESULTS AND DISCUSSION

3.1 Lineal relationship between the degree of soiling and the amount of carbon black deposited on fabrics.

The lineal relationship between the degree of soiling was evaluated by the Florio and Merserau equation(11) applied to the reflectance of fabrics and the logarithms of the amounts of carbon black deposited expressed as mg C. m⁻² were found. A total concentration of the surfactant of 2 x 10⁻³ M was maintained for a washing time, enough to ensure a total deposition of all the carbon black on the fabric after removal of all aqueous solution by boiling. The lineal adjustments obtained are shown in Table I.

3.2 Deposition of carbon black on finished cotton fabrics with DMDHEU and treated with different softeners such as esterquat(rewoquat) and aminosilicones (HF 3474 and HF 6922) in presence of SDBS and AE-6

The results corresponding to the amount of carbon black deposited at 40°C on the finished cotton fabric with DMDHEU and different softeners in presence of total concentration of 2x 10⁻³ M of the surfactants SDBS (anionic) and AE-6(non-ionic) alone and the mixtures of both are indicated in Figures 1 to 10. The mixtures of both surfactants were : 1:0 ; 0.75:0.25 ; 0.5:0.5;0.3:0.7;0.1:0.9 and 0:1. The water hardness was 20°hf.

a) With the softeners on the finished cotton applied alone
The deposition values for the finished cotton fabric without softeners are indicated in Figure 1. This fabric treated with the softeners Rewoquat W 75 H, and aminosilicones HF 3474 and HF 6922 at 0.3% o.w.f. are indicated in Figures 2, 3 and 4 respectively. The mixtures of both surfactants were: 1:0 ; 0.75:0.25 ; 0.5:0.5; 0.3:0.7; 0.1:0.9 and 0:1. The water hardness was 20ºhf.

------Figures 1 -2-3 and 4

In Figures 1 to 4, in general, the deposition values on the fabrics treated with resin DMDHEU and with the softeners alone, Rewoquat W 75 H, HF 3474 and HF 6922, are the highest in presence of anionic surfactant alone. For the mixtures, in general, the deposition values decrease on increasing the quantity of non-ionic surfactant in these ones. For the non-ionic surfactant alone the deposition values were, in general, the lowest ones.

After the softeners treatments of the cotton finished fabric, the deposition values increase for all the softeners tested and their mixtures.

Aminosilicone treatments HF 6922 and HF 3474 produce higher deposition values than Esterquat W 75 H, motivated by more hydrophobicity behaviour by organosilicones of the finished cotton fabric (12).

In general, the deposition values for finished cotton with aminosilicone HF 6922, of low molecular weight show the highest values in presence of anionic surfactant and slightly higher than aminosilicone HF 3474 of higher molecular weight for the surfactant mixtures.

a) **With the mixture of softeners applied on the finished cotton**
The deposition values for the mixtures of softeners applied on cotton fabric finished are indicated in Figure 5 to 10. Mixtures of Rewoquat with aminosilicone HF 3474 with total concentration of 0.3% o.w.f are indicated for the proportions of 0.75:0.25 ; 0.5:0.5 ; 0.25:0.75 respectively in the Figures 5, 6 and 7. The same ones for the mixtures of rewoquat with aminosilicone HF 6922 are indicated in Figures 8, 9 and 10.

In Figures 5 to 10, for mixtures of the softeners of Rewoquat W 75 H with aminosilicone HF 3474 and with HF 6922, show the same behaviour as the softeners alone. Higher deposition values in presence of anionic surfactant alone SDBS and lower ones for non-ionic surfactant AE-6, and, in general intermediate ones values for the mixtures of both surfactants tested, are obtained.

The deposition values of the Rewoquat W 75 H increase when it was applied in mixtures with aminosilicone HF 3474 and HF 6922 (Fig. 5 to 10)

On increasing the quantity of aminosilicone HF 3474 and their mixture with Rewoquat W 75 H, Figures 5, 6 and 7 or for aminosilicones HF 6922, Figures 8, 9 and 10, increase of the deposition values for the surfactants used alone and in mixtures, are obtained

The aminosilicone HF 3474 (of low molecular weight), in mixtures with Rewoquat W 75 H, in general, show slightly higher deposition values than the corresponding values obtained with aminosilicone HF 6922 (of low molecular weight) in the same proportion mixture.
4. CONCLUSIONS

With respect to the deposition at 40ºC of carbon black during washing with mixtures of surfactants onto finished cotton fabric with DMDHEU and treated with the softeners at the total concentration of $2 \times 10^{-3}$ M of Rewoquat W 75 H and aminosilicones (HF 3474 and HF 6922) having different molecular weights, alone and with mixtures The following conclusions can be drawn. These ones tested were Rewoquat/aminosilicone 0,75:0,25 ; 0,5:0,5 and 0,25 : 0,75. The DBSS and AE-6, alone and in mixtures were used for deposition trials. :

1º) A linear relationship between the degree of soiling and the logarithm of the concentration of carbon black deposited (mg C.m$^{-2}$) for cotton finished with DMDHEU and treated with Rewoquat W 75 H, aminosilicones HF 3474 and HF 6922, each one alone in presence of the surfactants used, was obtained.

2º) Anionic surfactant SDBS shows higher deposition values than non-ionic one.

3º) In general, the deposition values decrease on increasing the quantity of non-ionic surfactant in the mixture.

4º) In general, the deposition values of the finished cotton fabric increase the after the applicability of the softeners tested alone and with their mixtures.

5º) The deposition values for finished cotton with HF 6922, aminosilicone of low molecular weight, were higher than HF 3474 aminosilicone of high molecular weight.
6º) The Rewoquat W 75 H gave the deposition values lower than aminosilicones and HF 3474 and HF 6922.

7º) For mixtures of softeners Rewoquat W 75 H with HF 3474 and HF 6922 show the same behaviour as softeners alone, in consequence, the deposition values are higher for anionic and lower for non-ionic surfactants, and mixtures of them gave intermediate values.

8º) In general, the deposition values increase with respect Rewoquat W 75 H when aminosilicone (HF 3474 and HF 6922) are added to the mixture of softeners.

9º) On increasing the quantity of aminosilicones in the mixtures of softeners applied increases the deposition values, increase.

10º) In general, the aminosilicone HF 3474 of low molecular weight added to the mixture with Rewoquat W 75 H, shows slightly higher deposition values than HF 6922 aminosilicone of higher molecular weight.

5. BIBLIOGRAPHY


9) ISO Standard 2714(E)(1990)


Table I

Lineal adjustments between the degree of soiling and the logarithm of the concentration of carbon black deposited (x=\log \Delta C) on fabrics tested.

<table>
<thead>
<tr>
<th>Fabrics</th>
<th>Lineal adjustments</th>
<th>Correlation coefficients</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton + DMDHEU</td>
<td>Y = 26,577 x + 11,084</td>
<td>0.9805</td>
</tr>
<tr>
<td>Cotton+DMDHEU+REWOQUAT</td>
<td>Y = 36,147 x +4,0628</td>
<td>0.9812</td>
</tr>
<tr>
<td>Cotton +DMDHEU +HF 3474</td>
<td>Y =39,938 x + 5,8285</td>
<td>0.9823</td>
</tr>
<tr>
<td>Cotton +DMDHEU +HF 6922</td>
<td>Y = 40,778 x + 4,3032</td>
<td>0.9871</td>
</tr>
</tbody>
</table>
Fig. 1 Quantity of carbon black deposited at 40°C in function of molar proportion of SDBS/AE-6 for finished cotton fabric with DMDHEU (water at 20°C).
Fig. 2 Quantity of carbon black deposited at 40ºC in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and treated with 0.3% o.w.f Rewoquat W 75 H (water at 20ºhf)
Fig. 3 Quantity of carbon black deposited at 40°C in function of molar proportions of SDBS/AE-6 for finished cotton fabric with DMDHEU and treated with 0.3% o.w.f. of aminosilicone HF 3474 (water at 20°C)
Fig. 4 Quantity of carbon black deposited at 40°C in function of molar proportions of SDBS/AE-6 for finished cotton-fabric with DMDHEU and treated with 0.3 % o.w.f. of aminosilicone HF 6922 (water at 20ºhf)
Fig. 5 Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3% o.w.f of Rewoquat/aminosilicone HF 3474: 0.75:0.25. (water at 20°hf).
Fig. 6 Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3 % o.w.f. of Rewoquat/aminosilicone HF 3474: 0.5:0.5. (water at 20ºhf).
Fig. 7 Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3% o.w.f of Rewoquat/aminosilicone HF 3474: 0.25:0.75. (water at 20°C).
**Fig. 8** Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3 % o.w.f. of Rewoquat/aminosilicone HF 6922 0.75:0.25. (water at 20ºC).
Fig. 9 Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3% o.w.f. of Rewoquat/aminosilicone HF6922 0.5:0.50. (water at 20°hf).
Fig. 10 Quantity of carbon black deposited at 40°C in function of molar proportion SDBS/AE-6 for finished cotton fabric with DMDHEU and with 0.3 %o.w.f. of Rewoquat/aminosilicone HF 6922 0,25:0,75. (water at 20ºhf).