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EFFECTS OF CHEMICAL STRUCTURE OF SILICONE POLYETHERS USED AS FABRIC SOFTENER ADDITIVES ON SELECTED UTILITY PROPERTIES OF COTTON FABRIC

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Abstract:

The study addressed the effect of the structure of silicone polyethers on selected functional properties of cotton fabric rinsed in conditioners containing the additives under study. Fabric softener formulations containing two comb-structured compounds (PEG/PPG-14/0 Dimethicone and PEG/PPG-20/20 Dimethicone) and one block-structured compound (Bis-PEG/PPG-20/20 Dimethicone) were developed. Cotton fabric rinsed in conditioners containing silicone glycols was not found to be affected by yellowing. However, differences were noted in the softening ability and re-wettability of rinsed fabrics due to diverse structures of the additives used. The most desirable soft hand effect was observed after cotton rinsing in fabric softeners containing the block-structured compound Bis-PEG/PPG-20/20 Dimethicone. In contrast, the highest fabric re-wettability was shown for the conditioner enriched with a comb-structured compound (PEG/PPG-20/20 Dimethicone). The study results demonstrate that the prototypical fabric softeners containing silicone derivatives have a potential to provide quality characteristic required by consumers of this product group.

Keywords:

cotton fabric, adsorption capacity on fibers, fabric softeners, silicone polyethers

1. Introduction

Fabric softeners are used during the rinse cycle, which is the final stage of the laundry process. Their purpose is to provide fabrics with a range of beneficial qualities, for example reduce fabric roughness (so-called “soft hand”), eliminate static electricity build-up, improve the ironing effect and ensure a pleasant scent. In addition, fabric softeners are expected to be easily dispersible in water and should not cause fabric yellowing and corrosion of structural elements in washing machines.[1–9] The functions of fabric softeners determine their names. The products are also referred to as anti-static agents or fabric softeners. All these names are used in subsequent sections of the study interchangeably.

Fabric softeners usually have the form of aqueous dispersions of cationic surfactants, such as quaternary ammonium salts (quats), pyridine and imidazole salts. An important group of compounds used as additives in fabric conditioners are silicones, mainly those including a quaternary nitrogen atom in their structures. The presence of a nitrogen atom gives silicones a cationic nature, and thus, increases their capacity to become adsorbed on the surface of fabrics such as cotton. Many silicone types have been studied as potential additives to liquid fabric conditioners.[4,6,9–13]

Bereck et al.[11] evaluated the effect of silicone molecule structure, type and number of substituents and length of the siloxane chain on fabric softness, hydrophobization and yellowing. The authors studied a number of additive groups including polydimethylsiloxanes (PDMS), aminosilicones, epoxysilicones, silicone polyethers (polyetherpolysiloxanes, silicone glycols), cyclohexylaminosilicones, aminoethyloaminopropylosilicones.

The effects of silicone nano- and microemulsions on the properties of polyester fibers were investigated by Parvinzadeh and Hajiraissi.[4] Their study focused on evaluating the degree of whiteness of polyester fabrics subjected to antistatic agents containing silicone emulsions.

Research conducted by Habereder, Becker et al.[10–11] and Chinta et al.[12] explored the topic of hydrophobization of fabrics rinsed in conditioners containing aminosilicones at various concentrations. Furthermore, Becker, Haas and Kugler[13] investigated the decrease in friction coefficient at the fiber-fiber interface.

The studies outlined above were concerned primarily with silicones, that is, compounds with poor solubility in water, which are used as a base in fabric softener formulations. This may generate problems related to the manufacturing technology and markedly increase production costs because of the need to incorporate additional components enhancing the water solubility of these compounds. As a follow-up to previous research conducted worldwide on silicone derivatives used as fabric softener additives, the present study proposes the application of silicone polyethers in two structural forms: comb-like and block-like. In order to ensure the solubility of silicones in water, polyoxyethylene chains (formed by the binding of ethylene oxide molecules) are introduced into their structures. In addition, the presence of polyoxypropylene chains (arising by the addition of propylene oxide molecules) in the molecule increases the hydrophobicity of the compound.[14–16] Consequently, the proposed compounds combine hydrophobic properties that are characteristic of polydimethylsiloxanes with hydrophilic properties resulting from the presence of polyoxyethylene chains.

The present study sought to evaluate the effect of the chemical structure of silicone polyethers used as fabric softener additives on the fabric-softening ability and re-wettability, and the degree of whiteness. Also, an attempt was undertaken to assess the potential for film formation on the surface of cotton fibers by silicone polyethers selected for testing.

2. Experimental

2.1. Materials

Reagents and materials

The study material consisted of model fabric softeners formulated with the addition of silicone polyethers originating from a reaction between oxyalkylated allyl alcohol and polydimethylsiloxane containing a polysiloxane chain activated by Si-H groups, supplied by Evonik Industries AG.

The silicone compounds used in tests differ in the number of mers in the silicone chain (m, n), the number of ethylene oxide groups (x) and propylene oxide groups (y). Two of the compounds used are characterized by a comb-like structure (Fig. 1) and one has a block-like structure (Fig. 2). The following designations are used in the text: PEG – polyoxyethylene chain, PPG – polyoxypropylene chain, Dimethicone – polysiloxane chain. PEG/PPG designations are followed by the number of moles of ethylene oxide and propylene oxide bound to a given molecule.

The following compounds with a comb-like structure were used: PEG/PPG-14/0 Dimethicone (commercial name: Abil 8843, degree of polymerization m = 13, n = 5), PEG/PPG-20/20 Dimethicone (commercial name: Abil 8863, m = 32, n = 6), and Bis-PEG/PPG-20/20 Dimethicone (commercial name: Abil 8832, m = 62), characterized by a block-like structure.

Also, the following materials were used in the study: Bisacyloxyethylhydroxyethylmethylammoniummethosulphates (commercial name: Praepagen TQ from Clariant, containing 90% of cationic surfactant and 10% of isopropyl alcohol), sodium dodecylbenzenesulfonate (commercial name: Pasta ABS Na from Brenntag), sodium hydrogen carbonate (commercial name: sodium bicarbonate from Eurochem BDG Sp. z o.o.), ethylenediaminetetraacetic acid tetrasodium salt (commercial name: Ergon B from P.P.H. Polskie Odczynniki Chemiczne Gliwice), mixture of polyoxyethylene alkyl amines containing 8 moles of ethylene oxide (commercial name: Rokamin SR8 from: Zakłady Chemiczne Rokita S.A.), polyoxyethylene glycol (commercial name: Polikol 1500 from Zakłady Chemiczne Rokita S.A.), nonylphenol ethoxylated with 8 moles of ethylene oxide (commercial name: Rokafenol N8 from Zakłady Chemiczne Rokita S.A.).

The tests were performed using a cotton fabric without any additives or modifications: NORIS cotton, 1917-221-400-364, fabric batch 13/160 (m/width), manufactured by Tkalnia Bielawa "Bieltex" Spółka z o.o.

Properties of cotton fabric used in tests are: weight 133 g/m2, yarn linear density (warp 25, weft 30), number of threds/10cm (warp 256, weft 192), tensile strength (warp 34.4, weft 34.5), shrinkability (warp 5.0, weft 3.2).

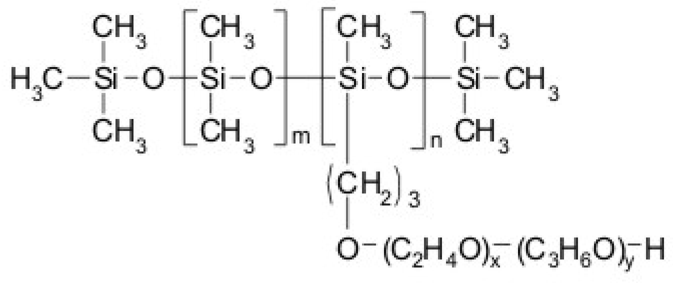

Figure 1. Structural formula of silicone glycol with a comb-like structure

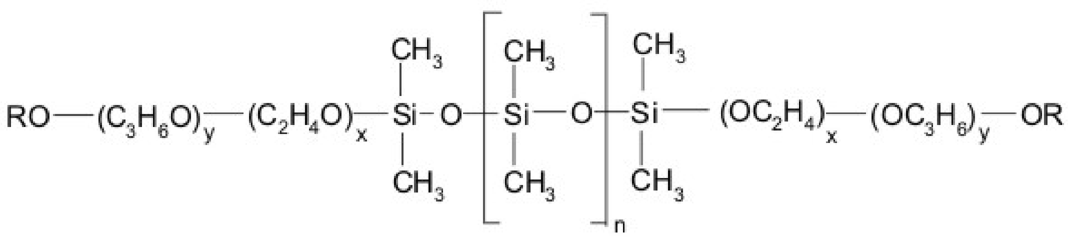

Figure 2. Structural formula of silicone glycol with a block-like structure

New fabric softeners

Based on the literature data and own experience,[3–5,9–11,17–21] model fabric softener formulations were developed, differing in the type of silicone polyether used. The compositions are listed in Table 1.

Preparation of fabric softeners:

A total of four fabric softener formulations were developed. Their compositions are listed in Table 1. The method of fabric softener preparation involved adding an appropriate amount of a cationic surface active agent to water at a temperature of approximately 50ºC. After the solution was cooled, an appropriate silicone polyether and a preservative were added to the solution. The formulation was mixed until complete homogenization.

2.2. Methods

Fabric preparation for tests

Prior to testing, the cotton fabric was washed in order to remove fabric finishers. The fabric samples were washed at a temperature of 100ºC for 15 minutes in a bath containing 3 g of sodium dodecylbenzenesulfonate (expressed as 100% of the active substance) and 2 g of sodium hydrogen carbonate in 1 l of distilled water. After washing, the fabric was rinsed five times in distilled water. In the next step, water was removed by manual squeezing and the fabric was left to dry. After that, fabric samples of specific dimensions were prepared for testing.

Softening properties of fabric softeners

The aim of the test was to compare the softening properties of new fabric softeners with standard formulations using the method of fabric hand evaluation. Based on a subjective evaluation of fabric softness, 1 point was awarded to the fabric sample in the pair, which—in the tester's opinion—was softer. If both test samples were considered equally soft, each of them was given 0.5 point. The maximum number that could be obtained for the fabric softener under study was 50. Detailed methodology has been described in the literature.[22]

Re-wettability

The method was based on measuring relative wetting height in cotton fabric samples rinsed in a solution of the fabric softener under study in comparison to the control sample (fabric rinsed in distilled water).

Re-wettability X [%] was calculated from the formula:

$$X = \frac{h}{h_0} \cdot 100,$$

where:
h – height of water front in fabric sample rinsed in fabric softener under study [mm],
h_0 – height of water front in control fabric sample [mm].
Detailed methodology has been described in the literature.[23]

Degree of fabric whiteness

The method was based on leucometric measurements of changes in the degree of whiteness occurring in fabrics rinsed in a solution of the prototypical fabric softener compared to a fabric rinsed in distilled water. The degree of fabric whiteness was measured with a leucometer (Color 02) after the instrument was calibrated using white and black standards. Detailed methodology has been described in the literature.[24]

Adsorption of silicone polyethers on cotton fibers

The topography of the fabrics rinsed in 1% aqueous solutions of polyetherpolysiloxanes and in distilled water was assessed. Measurements were performed with S-2460N Hitachi scanning electron microscope (SEM) with low vacuum mode, and energy-dispersive (EDS) detector from Noran, fitted with a Norvar window and SiLi crystal, with a resolution of 133 eV, electronically coupled to the microscope. The images were obtained at the magnification of 1,000x.

All the results shown in the charts are averaged values of independent measurement series defined for a given test. The limits of the confidence interval for the measured parameters were calculated using Student's t-test. For the confidence level of 0.90, intervals representing the measurement error were defined.

Table 1. Compositions of model fabric softeners

Ingredients [INCI]	**Concentration [wt. %]**			
	PS0	**PS1**	**PS2**	**PS3**
Bisacyloxyethyl hydroxyethyl methylammonium methosulphates	5,55			
PEG/PPG-14/0 Dimethicone	-	1	-	-
PEG/PPG-20/20 Dimethicone	-	-	1	-
Bis-PEG/PPG-20/20 Dimethicone	-	-	-	1
Benzisothiazolinone and Methylisothiazolinone	0,01			
Aqua	to 100			

3. Results and discussion

Degree of fabric softness

Fibers that make up fabrics are exposed to considerable damage during the laundry process. A prominent role is played by friction processes occurring between individual fibers and between the washing machine drum and fabric fibers. An additional factor is chemical damage of fibers, which arises from interactions with solutions often containing aggressive surfactants and having relatively high pH levels. Consequently, after the laundry process, the fabric fiber surface is frequently destroyed, heterogeneous, with multiple damaged microfibers sticking up from the surface. What is more, the ingredients of laundry washing agents may accumulate on the surface of washed fabrics. The phenomenon is referred to as incrustation, and its most common cause is the build-up of a deposit on the fiber surface, consisting of water insoluble calcium soaps. Since the surface of such deposits is very rough, the fabric does not have a soft hand.[8] The hand of the fabrics rinsed in new liquid fabric conditioners containing silicone glycols was evaluated in accordance with the methodology set out in the PN-86C-4833/02 standard, with results shown in Fig. 3.

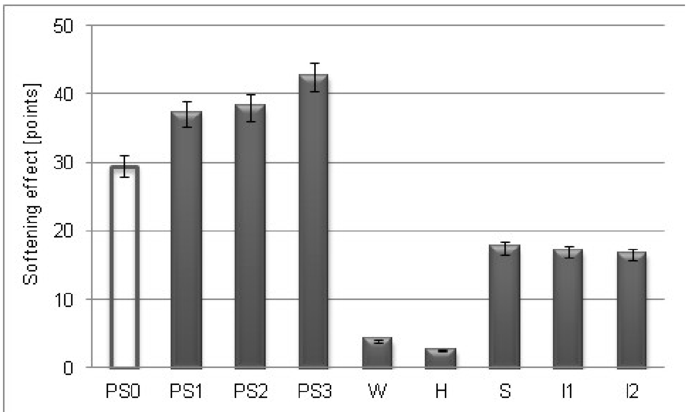

Figure 3. Degree of softness of fabrics rinsed in aqueous solutions of fabric softeners: baseline (PS0), with comb-structured silicone polyethers (PS1, PS2) and with block-structured silicone polyethers (PS3)

Designations of samples rinsed in: W – distilled water, H – hard standard, S – soft standard, I1 – intermediate standard 1, I2 – intermediate standard 2

Figure 3 shows the results of measurements performed to determine the degree of softness in a cotton fabric rinsed in standard formulations and in tested fabric softeners. The degrees of softness determined in the cotton fabric rinsed in normative standards were: 4 points (sample rinsed in distilled water), 2.5 points (cotton rinsed in the hard standard), 17.5 points (fabric rinsed in the soft standard), 17 and 16.5 points (samples rinsed in intermediate standards).

The degree of softness determined for the fabric rinsed in an aqueous solution of a silicone-free fabric softener (PS0) was 29.5. The scores obtained for the fabric conditioners containing comb-structured organosilicone derivatives (PS1, PS2) were 37 and 38 points, respectively, and were thus nearly 10 times higher than for the samples rinsed in water, over 15 times higher than the scores obtained for cotton subjected to the effect of the hard standard and approximately twice as high as for the samples rinsed in the soft and intermediate standards. The most desirable softness, amounting to 42.5 points, was observed when the cotton fabric surface was modified with a fabric softener containing a block-structured silicone (PS3).

A comparison of different formulations failed to reveal a significant effect induced by the presence of a propylene oxide group in the structure of comb-like silicones (PS1 and PS2). However, an important factor from the viewpoint of the softening effect seems to be the structural form of silicone polyethers. The fabric softeners containing comb-structured silicones induced a lower level of soft hand compared to the formulation enriched with a block-structured compound.

Bereck et al.[11] studied soft fabric hand depending on a range of factors including the silicone molecule structure, type and number of substituents, length of the siloxane chain and affinity to the fiber surface. Based on the collected data, the authors ordered the compounds under study depending on their fabric softening ability. The following sequence was obtained:

polydimethylsiloxanes (PDMS) < aminosilicones < epoxysilicones < silicone glycols < cyclohexylaminosilicones < aminoethyloaminopropylosilicones.

The fact that PDMS is associated with a low level of fabric softness is most likely an effect of weak interactions between the molecules and the fiber surface, and consequently, an uneven distribution of the compound on the fiber. The level of softness in derivatives containing a nitrogen atom in their structure depends most likely on the number of nitrogen atoms in the molecule. Based on the literature[10–11] it can be postulated that the softness of fabrics rises as a function of increasing number of nitrogen atoms in the molecule of the silicone derivative.

Re-wettability

The surface of fibers subjected to the effect of fabric softeners usually becomes hydrophobic, which reduces the ability of fabrics to absorb water. The property should be considered depending on the intended application of rinsed fabrics.[12] For example, good water absorbing capacity is a desirable property in towels. In contrast, some fabrics are intentionally hydrophobized in order to protect them from moisture. An analysis was performed for the wettability of fabrics, the surface of which was modified by rinsing in developed fabric softeners containing silicone polyethers (Figure 4).

The incorporation of silicone derivatives into the fabric conditioner formulation increases the efficiency of cotton fabric re-wettability with water (Fig. 4). The sample rinsed in the fabric softener without any addition of silicone polyether exhibited the re-wettability level of 62%. The use of proposed compounds in the formulation causes an increase in the measured value by up to one-fourth (PS2) compared to cotton rinsed in the baseline softener (PS0).

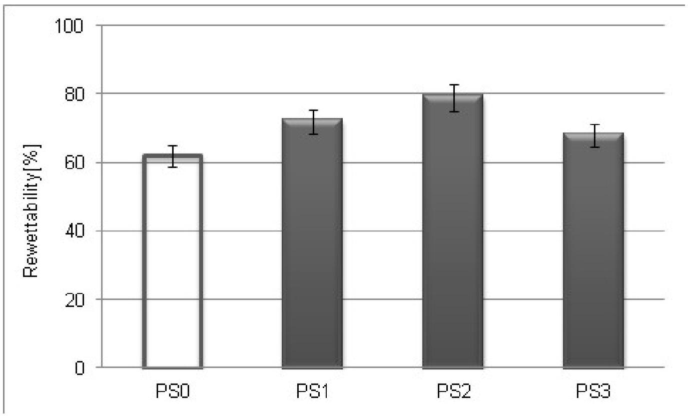

Figure 4. Re-wettability of fabrics rinsed in aqueous solutions of fabric softeners: baseline (PS0), with comb-structured silicone polyethers (PS1, PS2) and with block-structured silicone polyethers (PS3)

Generally, there was a tendency for re-wettability to increase after the application of all new fabric softeners enriched with silicone glycols. Importantly, based on the results obtained in the study, it can be claimed that the presence of a polyoxypropylene chain (PS2) in the molecular structure has a greater impact on the measured parameter in comparison with a silicone containing only a polyoxyethylene chain (PS1). The collected data provide grounds to conclude that a significant role is played by the spatial structure of the compound and the size of its molecule (molecular mass). Re-wettability determined in fabric softeners containing silicones with a comb-like structure (PS1, PS2) was in the range of 72–80%. In contrast, the incorporation of a block-structured silicone (PS3) into the formulation produced the re-wettability level of 68%. It can therefore be postulated that the measured value is also affected by the molecular structure.

It needs to be noted that the application of silicone polyethers, regardless of their structural form, increases the hydrophilicity of fibers compared to the sample of fabric rinsed in a fabric softener containing exclusively a cationic SAA (PS0).

The wettability mechanism in conditioner-rinsed fibers can be explained on the basis of literature data.[9–11] According to Habereder, Bereck et al.,[10–12] the fabric softener ingredients become adsorbed on the fiber surface and form a film that would make it more difficult for the fabric to become soiled. Polydimethylsiloxanes form thick “bundles” on the fibers. Consequently, the fabric exhibits a limited hydrophobic effect.[10] Aminosilicones and their derivatives reduce fabric wettability, and the severity of the effect depends on the number of groups containing a nitrogen atom. Following the incorporation of silicones containing quaternary nitrogen atoms into the fabric softener formulations, fabric wettability depends to a large extent on the molecular mass of the polymer (length of the siloxane chain), the number and arrangement of substituents, and the presence of other atoms in the molecule. Fabric wettability decreases with an increase in the length of the siloxane chain and grows in proportion to increases in the number of hydrophilic substituents.

Also, Chinta et al.[12] compared the water absorption capacity in softeners containing, among others, various silicone derivatives. Their experiments show that an increase in the concentration of silicone additives in the form of aminosilicones correlates with an extended period of water absorption by cotton fibers. In fabric softeners containing hydrophilic silicones, the tendency was similar; however, the time of water absorption by cotton fabric was shorter compared to other silicones used.

<u>Degree of fabric whiteness</u>

A desirable property of liquid antistatic agents is the absence of the fabric yellowing effect after drying. Fabric yellowing noted after the application of fabric softeners is most likely a result of the presence of cationic surface active agents containing unsaturated bonds in their structures. Fat derivatives of this type are highly prone to oxidation processes, for example, during storage or in response to high temperatures (e.g., during ironing).

The degree of whiteness (DW) of cotton rinsed in the original fabric softeners with and without the addition of silicone polyethers was assessed by means of leucometric measurements. Correlations were determined between the chemical structure of silicones present in the original fabric softeners and their impact on the degree of whiteness in rinsed cotton fabrics. Test results are shown in Fig. 5.

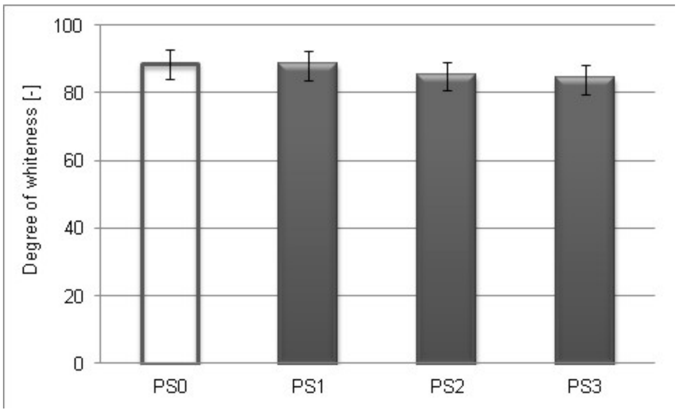

Figure 5. Degree of whiteness of fabrics rinsed in aqueous solutions of fabric softeners: baseline (PS0), with comb-structured silicone polyethers (PS1, PS2) and with block-structured silicone polyethers (PS3)

Based on the test results, it was concluded that the addition of three types of silicone polyethers with different chemical structures (comb-like, block-like) to liquid fabric softeners has no significant impact on the change in the degree of whiteness of rinsed cotton. The DW values obtained for the fabric rinsed in the silicone-free conditioner (PS0) and for the sample rinsed in the conditioner containing PEG/PPG-14/0 Dimethicone (PS1) were nearly identical (approximately 88). High DW levels (about 84) in fabrics were also observed after the application of fabric softeners containing PEG/PPG-20/20 Dimethicone (PS2) and Bis-PEG/PPG-20/20 Dimethicone.

Based on these results, it can be claimed that the rinsing of cotton fabric samples in the new liquid fabric conditioners containing silicone polyethers caused no fiber yellowing.

Parvinzadeh and Hajiraissi studied the degree of fabric whiteness after the application of silicone nano- and microemulsions on polyester fibers.[4] Their research showed that the silicone emulsions studied could make polyester fibers turn grey.

The literature reports[10] also indicate that elevated temperatures cause the yellowing of fabrics subjected to the activity of aminosilicones. The degree of fabric yellowing depends on the type and number of substituents, and on temperature. For example, a fabric rinsed in a solution of aminoethyloaminopropylosilicone is more prone to yellowing than a fabric rinsed in a solution of aminopropylosilicone. The application of silicone glycols as fabric softener ingredients has also been found to potentially reduce fabric yellowing.

Adsorption of silicone polyethers on cotton fibers

Scanning electron microscope was used for evaluating the topography of cotton fibers subjected to aqueous solutions of silicone glycols. The purpose was to document the formation of film on the surface of cotton fibers, which could confirm the adsorption of silicone glycol molecules. Images of cotton fibers subjected to 1% aqueous solutions of silicone glycols are shown in Fig. 6.

Cotton fibers are arranged in the form of ribbons with a spiral twist. Their outer edges are curled. The surface of fibers subjected to water and aqueous solutions of silicone glycols is similar (Figs. 6A–D). However, the surface of fibers rinsed in aqueous solutions of polyetherpolysiloxanes was shown to exhibit "infiltrates" forming a specific layer of "grease". The finding can be attributed to the adsorption of silicone molecules from solutions on the fiber surface. The fibers appear linked by distinct bridge-like structures. The effect is particularly prominent in fibers subjected to 1% solution of PEG/PPG-14/0 Dimethicone. Similar findings were obtained by Montazer and Hashemikia for a fabric rinsed in a solution of conditioners containing silicone derivatives.[6]

The adsorption of silicone glycol molecules on the fibers can be explained on the example of cationic surfactants that are commonly added to fabric softeners. Typical compounds used as fabric softener ingredients (cationic SAAs) may become adsorbed on the fibers through electrostatic interactions and the so-called "hydrophobic effect". In cationic SAAs, the adsorption of molecules on the fiber surface takes place via an interaction in which hydrophobic aliphatic chains of cationic SAA molecules are adsorbed perpendicularly to the fiber.[9]

A different type of process accounts for the adsorption of surfactants via hydrophobic interactions.[9] Cationic surfactants are characterized by limited solubility in water due to the fact that their molecules consist of long hydrophobic chains. Consequently, they are adsorbed on the fiber surface in such a manner as to reduce the area of contact with the aqueous environment. The fiber surface is considered here as an interfacial surface on which hydrophobic surfactant molecules are accumulated. The same explanation applies to the adsorption of silicone SAA on fibers.[9,15] Silicone chains will be arranged in parallel to the fiber surface so as to ensure that the degree of contact with the fiber surface is as great as possible.

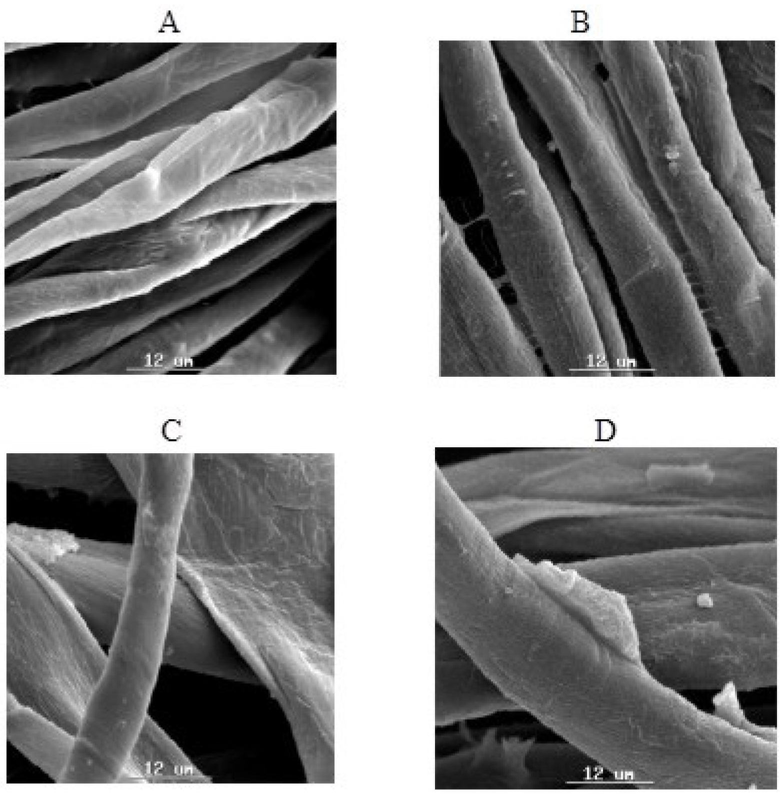

Figure 6. Photographs of cotton fabric subjected to: (A) water and 1% aqueous solutions of the following compounds: (B) PEG/PPG-14/0 Dimethicone, (C) PEG/PPG-20/20 Dimethicone, (D) Bis-PEG/PPG-20/20 Dimethicone. Images obtained with scanning electron microscope (SEM). 2000x magnification

4. Conclusions

The study results demonstrate that there is a large potential for using silicone derivatives as active additives of fabric softeners. The structure of the studied compounds was shown to have an impact on improving the selected utility fabric properties, as compared to the fabrics rinsed in additive-free fabric softeners. The most beneficial cotton fabric softening ability was demonstrated for the formulations enriched with the block-structured compound Bis-PEG/PPG-20/20 Dimethicone. In contrast, the highest re-wettability was shown for the conditioner containing a comb-structured compound (PEG/PPG-20/20 Dimethicone). The application of silicone derivatives in fabric conditioners was not shown to cause any fabric yellowing effect. SEM images obtained in the study also point to the possibility of film formation on the surface of cotton fibers. It is postulated that the adsorption of silicone glycol molecules on the fiber surface may be involved in this case.

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