

Physical Properties of Human Hair Dyed Using Nonionic Surfactants

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The influence of different nonionic and slightly cationic surfactants used only in bleaching of hairs before dyeing with C.I. Acid Blue 80 or only in dyeing on tensile stress/strain properties of dyed human hair was examined. The pretreatment of hair fibres before dyeing was carried out in alkali media with hydrogen peroxide in the presence or in the absence of surfactants. The results have shown that when the bleaching of hairs was accomplished in the absence of surfactants, but in the subsequent low temperature dyeing of hair fibres were applied nonionic surfactants, the rate of dye adsorption onto the fibres was enhanced. When polyoxyethylene (25) cocamine or ethoxylated (32) castor oil surfactants or ethoxylated (EO 12) coconut acids fatty amide were applied only in the bleaching of hair fibres, the dyeability was enhanced and the tensile characteristic values of dyed hairs were increased as well.

Keywords: bleaching, dyeing, hair fibres, physical properties, surfactants.

1. INTRODUCTION

The mechanical properties of α -keratin fibres such as hair fibres and wools are primarily related to the two components of the elongated cortical cells, the highly ordered intermediate filaments (microfibrils) which contain the α -helices, and the matrix in which the intermediate filaments are embedded [1]. Chemical treatment, bleaching and dyeing is known to be one of hair cuticle and cortex damage producing and properties impairing factors [2, 3]. Bhushan *et al* [4] using nanoindentation and SEM for nanomechanical characterization of human hair have shown that the chemical damage and conditioner treatment causes the hardness and elastic modulus of hair surface to decrease within a depth of less than 1.5 μm . The decrease of mechanical properties was assumed to be due to the sorption of individual conditioner molecules into outer part of cuticle.

Hydrogen peroxide is widely used for the oxidative hair coloring or bleaching. It easily penetrates into the hair fibre and being capable of non-specific interactions with cortex and cuticle proteins results in irreversible cleavage of cystine crosslinks [5, 6]. The treatment, which includes oxidative steps, causes tensile damage and modifies the surface of hair, introducing negative charges as a result of oxidation of cystine to cysteic acid [7].

Doering *et al* [5] have suggested that preventing hair damage at the molecular level during oxidation coloring could be successfully rendered by the use of organic disulphides such as α -lipoic acid which is not a powerful antioxidant. The results of this study indicate the possibility by the way of molecular screening to reduce the hair cystine crosslinks splitting. Specifically, the effect of hair protection could be achieved due to a lower accessibility of cystine crosslinks to the oxidative agents. However, it's not easy though to achieve the positive effect

of cystine crosslinks protection by reason of the extraction of soluble materials from the hair occurring during the treatments with aqueous solutions of chemical agents. Scanavez *et al* [8] have demonstrated that daily care damages human hair and renders cavities, or holes in the endocuticle. In the cortex, cavities develop in the intermacromolecular cement and in the cell membrane complex. Displacement, cracking and cleavage of cuticle cells was also observed.

Gummer [9] has shown that the high-sulphur proteins of cuticle, usually considered as highly cross-linked and inaccessible, are also easily penetrated for the delivery of industrial and cosmetic materials.

Rele *et al* [10] have investigated the effect of some cosmetic chemicals on the damage of hair fibres pretreated with aqueous solutions of different nonionic or ionic compounds. The authors have hypothesized that coconut oil used as a prewash penetrates endocuticular material in the intercuticular region and reduces its swelling propensity. It has been also shown that the above indicated prewashing could be a measure of the prevention hair surface cuticle lifting and of the reduction of its breaking under exerted external forces. Gamez-Garcia has investigated the behaviour of swollen hair fibers under strain-cycling conditions and has found that cuticle damage of type decementation and buckling can be prevented by using appropriate hair swelling agents [11].

The dependence of physico-mechanical properties of keratin fibres upon their structure changes occurring during pretreatment and dyeing was investigated by Joko *et al* [12, 13]. The authors have presumed that the destruction of α -crystallite, which results on the fission of microfibril-matrix crosslinkages such as hydrogen bonding, salt linkage, and disulphide bond, and this cleavage may increase the dyeing sites and facilitate dye penetration [13]. In our previous research the effect of a nonylphenylethoxylate-based nonionic surfactant on the kinetic and thermodynamic properties in wool fiber dyeing with acid dye and on physical properties of dyed wool fabric was studied

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[14, 15]. The presumption was made that one of the action mechanisms of nonionic product incorporating ethoxylated alkylphenols on dyeing is the modification of hydrophobic domains of cellular membrane complex in wool fibre. It was also observed that a mixture of short chain ethoxylated alkylphenols revealed wool fibre protective properties when it is used in dyeing and reduced permanent set of fabric [15]. The pretreatment of wool fibre with aqueous solution of nonionic nonylphenoethoxylate-based surfactant as compared with anionic sodium laurylsulphonate was assessed to be beneficial in the case of exposure of wool fabric to UV irradiation [16]. Unfortunately, nonylphenoethoxylates and the compounds of their chemical transformation are considered to be more toxic and estrogenic as compared with surfactants based on linear ethoxylates.

This investigation is aimed at presenting the beneficial effects of linear nonionic or slightly cationic surfactants in preventing hair damage when they are used as auxiliaries in bleaching with hydrogen peroxide or in subsequent dyeing.

2. EXPERIMENTAL

2.1. Methods and Materials

Hair samples. Hair fibres samples were used from Lithuanian women who had not been exposed to chemical treatment (bleaching or perming). Pretreatment of hair fibres samples (tresses) by washing or bleaching was performed as described below.

Surfactants. Nonionics commercial product based on nonylphenoethoxylate Lanasan LT (Clariant) (NPE), ethoxylated castor oil Berol 199 (Akzo Nobel) (REO 32), ethoxylated coconut acids fatty amide Bermodol SPS 2532 (Akzo Nobel) (SPS 2532), slightly cationic polyoxyethylene cocamine Ethomeen C/25 (Akzo Nobel) (C/25), zwitterionic cocamidopropylbetaine (CAPB) were used as received.

2.2. Procedures

Washing. Untreated hair fibres samples were washed in solution containing 1.5 g/dm³ of nonionic ethoxylated (C₁₂-C₁₆) fatty alcohol Berol 175 and 0.8 g/dm³ of sodium carbonate. pH of solution was 10.7. Washing was carried out for 10 min at the temperature of 55 °C. Finally, the samples were rinsed subsequently with hot (warmed-up) and cold demineralized water.

Bleaching. Hair fibres were bleached using a solution of hydrogen peroxide (6 %) in the presence of respective surfactants (*ca* 0.18 mmol·dm⁻³), or without them. pH 10 of bleaching solution was adjusted with NH₄OH and the treatment was carried out during 1 h at 60 °C. The tress was then rinsed with 2 % aqueous acetic acid solution and then with demineralized water until pH was neutral. Excess water was removed by placing the tress onto a paper towel. The tress was dried up under ambient temperature in open air (20 °C ± 2 °C).

Dyeing. C.I. Acid Orange 7 dye applied for hair dyeing was purchased from Sigma Co (Germany) and used in this study without further purification. Dyeing of pretreated samples was carried out using laboratory

apparatus ISAL AG BASEL with steel cylinders to girdle the samples. The mass of hair fibres samples was 0.25 g. The samples before dyeing were soaked off in 0.1 M acetic acid-sodium acetate buffer solution (pH 4.2) for 1 h. After that the samples were drained with filter paper and put into the steel cylinders for dyeing. The samples were dyed in dye solution of 0.57 mmol·dm⁻³ concentration. Dyeing was carried out for 1 h in the absence or in the presence of surfactants. The liquor-to-hair ratio was 100:1 and the temperature was 60 °C. The dyed samples were rinsed for 5 s–7 s in demineralized water, squeezed and dried in open air.

Dye exhaustion was calculated from the difference in dye absorbance depending on solution concentration before and after dyeing. The absorption measurements were performed on a Specord UV-VIS spectrophotometer with 10 mm quartz cell. The percentage of dyebath exhaustion *E* (%) was calculated according to:

$$E = \left(\frac{A_0 - A_t}{A_0} \right) \times 100, \quad (1)$$

where *A*₀ and *A*_{*t*} are the absorbances at λ = 488 nm of dye in the initial dye bath and that of residual dye in the dye bath respectively.

2.3. Mechanical tests

Tensile force-extension tests for swollen in water hair fibres were performed and curves were drawn by using a Mesdan-Lab strength tester equipment (LST EN ISO 2062 2000). The average of results obtained by measurement of ten hairs from every sample of tress was taken in consideration. The values of measured breaking force are employed as fiber damage test [3]. The swollen fibers were obtained by immersing in distilled water at room temperature for 24 h. The hair fibres used were about 15 cm long snippets. The distance between clips in the tester was 10 cm and the extension speed was 135 mm/min. The measurements were performed ten times for every examined sample of hair the fibres. The average values of breaking force and elongation at break were taken into account and used in this study. Variation coefficient of the breaking force and elongation at break in all measurements did not exceed 5 %.

3. RESULTS AND DISCUSSION

To demonstrate the effectiveness of nonionic surfactants in terms of reduction of hair damages occurring in bleaching and dyeing we present results in which the change of mechanical properties of hair fibres is shown. Some surfactants are shown to increase the strength of hair as well as dye adsorption.

The loss in strength was estimated as the level of hair damage. The mechanical tests were performed for dyed hair fibres samples, which before dyeing were bleached in the presence of surfactants or dyed in the presence of surfactants. The loss in strength was evaluated for all series of assays and the results were compared with the corresponding untreated or treated without using of surfactants. For the most important cases the tensile load-extension tests were performed. The parameter ,load to

break' or load-extension characteristics for hairs are often measured under conditions, which should be similar to those occurred on the head [17, 18]. Commonly the term 'force' instead of term 'load' is used. Thus in this study we measured the parameters named as breaking force, elongation at break and the character of tensile force-extension curves.

3.1. Characterization of hair fibers pretreated before dyeing

The application of nonionic surfactants ethoxylated (C_{12} - C_{16}) fatty alcohol (REO 7.5) or nonylphenoethoxylate (NPE) as dyeing auxiliaries as show the results presented in Figure 1 enhance dye adsorption (Fig. 1, a) at low temperature (60 °C) of dyeing and render a higher values of breaking force (Fig. 1, b) for dyed hair fibres as compared with those for dyed without auxiliary.

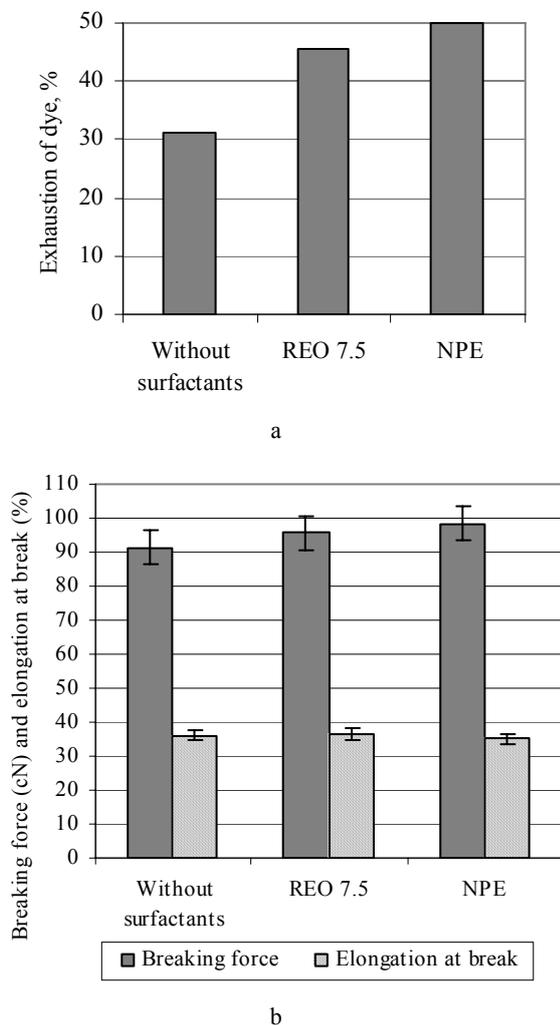


Fig. 1. Influence of nonionic surfactants on dye sorption (a) and breaking force characteristics (b) of dyed hair fibres, which before dyeing were only washed

As it can be seen from the results presented in Fig. 1, when NPE was added to the dyeing solution the dye exhaustion was higher and the loss of fibre strength during dyeing was somewhat lower as compared with those observed when REO 7.5 was used.

Whereas the control of hair fibres damage level was of our interest, for the treated samples in this study we carried out mechanical tests.

The results presented in Fig. 2 show that the breaking force values for bleached hair fibres was lower, as compared with those for the untreated or washed hair fibres.

The tensile force-extension curves (Fig. 3) of untreated, washed or bleached hair fibres had generally specific features for keratin fibres and consisted of Elastic (Hookean) (A-B), Yield (B-C) and Post-yield (C-D) regions. The comparison of tensile force-extension curves show that the transition from the elastic region into the yield region for the washed and especially for the bleached fibres is somewhat steeper as compared with that of untreated hair fibres. This could be related to the change of α -keratin fibres cohesion discussed by Wortman and Zahn [19]. The results presented in Figs. 2 and 3 allow us to assume that the split of cystine cross-linkages, salt links and hydrogen bonds could take place during oxidative treatment as it is denoted elsewhere [20–22]. The Yield region (B-C) in Fig. 3 represents the beginning of $\alpha \leftrightarrow \beta$ transformation, which according to Feughelman [23] is the result of the disruption of salt links and hydrogen bonds. In our study for the bleached hair fibres the yield region (B-C) (Fig. 3) represents a lower extension comparing with those for untreated or washed hair fibres. Herle [24] explained the yield as a crystal transition in the fibrils of wool from the helically coiled α -lattice to the longer extended straight-chain β -lattice.

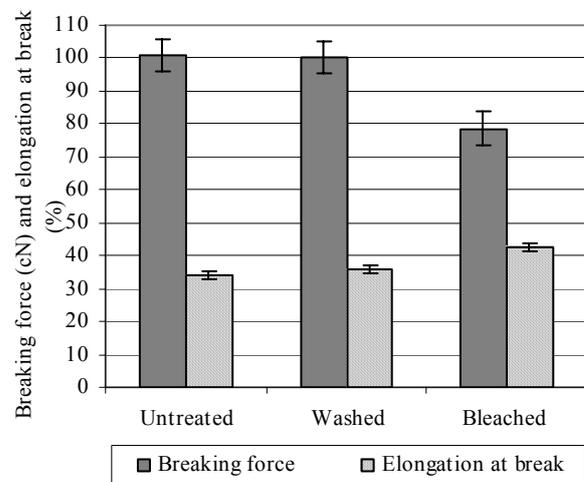


Fig. 2. Breaking force and elongation at break of hair fiber pretreated before dyeing

The comparison of tensile force-extension curves presented in Fig. 3 for untreated fibres with those for washed ones leads to the assumption that during the washing, the hydrogen bonds within the hair could be disrupted or the net of hydrogen bonds is lessened. The data are in agreement with Woodruff's idea [17] that most of the bonds present in dry hair are lessened in the presence of water. According to the discussion made by Wortmann and Zhan [19] in the Post-Yield region C-D a complete $\alpha \leftrightarrow \beta$ transformation occurs and the polypeptide chains are completely unfolded in this region [17]. The

shape of the curve in the region C-D which is generally assumed to be defined by cystine cross-linkages, as we can see in Fig. 3 for bleached hair sample is less sharp as compared with those for untreated and washed hair samples. The results of these tensile force-extension tests allow to presume that a significant part of disulphide bonds in the bleached hair is disrupted. The comparison of tensile force-extension curves for untreated hair fibres with those for washed or bleached fibres leads to the assumption that the pretreatment before dyeing, such as washing or bleaching, influences on its physical properties. Bleaching with hydrogen peroxide considerably reduces the strength of hair fibres and changes their tensile force-extension behaviour.

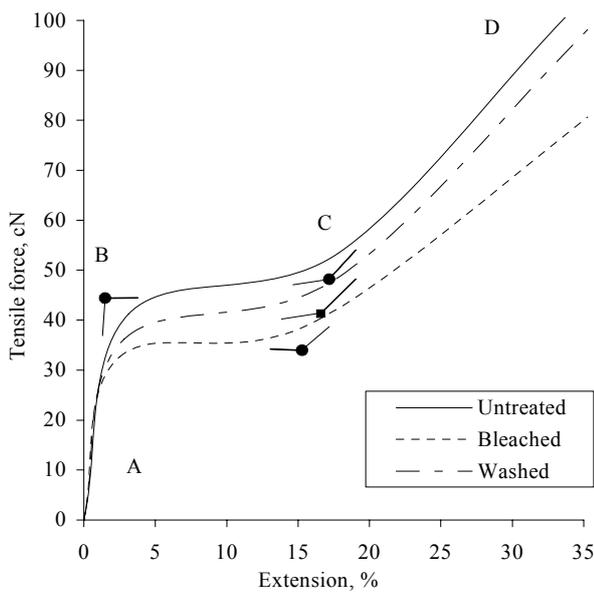


Fig. 3. Tensile force-extension curves of untreated, washed or bleached hair fibres

3.2. Effect of surfactants used in bleaching solution before dyeing on tensile properties

It is known that chemical and physical properties of keratin fibres are influenced by the presence of surfactants and the research in the development of bleaching with hydrogen peroxide has been oriented into the ways promising the maximum of whiteness with minimal damage to fibre [21, 22]. The series of investigations has been accomplished to compare the effects of sodium alkylsulphates and cationic surfactants, which were added to the bleaching solution. It was found that alkylsulphates with the longer hydrophobic chain reduced the damage of fibre occurring during bleaching [21]. The effect was explained by the hydrophobic interaction between the fibre and surfactant and the shielding action of absorbed surfactant leading to the reduction of disulphide linkages splitting and related to this chemical change the reduction of fibre damage.

In our study it was of interest to introduce the surfactants possibly displaying distinct behavior in the interaction with hair fibre and/or with anionic dye. Thus, for the investigation of hair fibres bleaching with hydrogen peroxide in the presence of surfactant we have selected a

slightly cationic polyoxyethylene cocamine C/25 (C/25), and zwitterionic – cocamidopropylbetaine (CAPB). In this series of assays bleached fibres were dyed without surfactants. The measurements of dye exhaustion and mechanical testing were performed.

Fig. 4 presents the tensile force-extension curves obtained for hair fibres, which were bleached in the presence of NPE, C/25 and CAPB and afterwards the dyeing in the absence of surfactants was performed. The comparison of tensile force-extension curves shows that the extent of slope in the Elastic region (A-B) for the fibres bleached in the presence of cocamidopropylbetaine (CAPB) was lower as compared with that for the fibres bleached with C/25, NPE or control assay.

The results presented in Fig. 5 show that the values of breaking force of bleached hair fibres with the addition of zwitterionic surfactants CAPB decreased more significantly as compared with those for C/25 or REO 32. This indicates that the impairment of tensile properties of hair fibres occurring due to the damage, when the nonionic surfactants (REO 32 or C/25) were applied was lower as compared with that observed for zwitterionic surfactant.

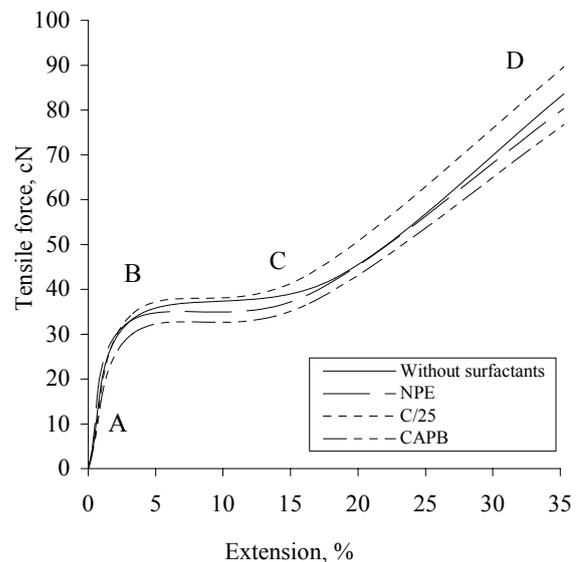


Fig. 4. Tensile force-extension curves for dyed hair fibres which were bleached in the presence of surfactants

The data obtained for hair fibres bleached in the presence of ethoxylated castor oil (REO 32) as well as of ethoxylated coconut acids fatty amide (SPS 2532), and afterwards were dyed without surfactants (Fig. 5) show that the influence of SPS 2532 on the breaking force of fibres was insignificant comparing with the results obtained with REO 32. However, relatively high values of breaking force of fibres in the case of polyoxyethylene cocamine (C/25) indicates that the beneficial effect in preventing fibre damage during bleaching with hydrogen peroxide can be considered as exceptionally good.

The tensile force-strain diagram presented in Figure 4 shows that for the sample of hair fibres bleached in the presence of C/25, the Elastic (A-B), and Yield (B-C) regions in the curves are less extended as compared with those for the sample treated with hydrogen peroxide in the absence of surfactants.

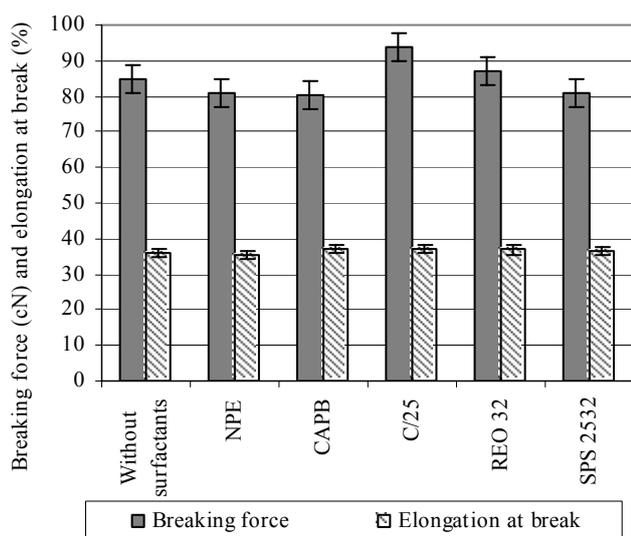


Fig. 5. Mechanical properties of hair fibres dyed, when surfactants were used only in bleaching

The results presented in Table 1 show, that when the surfactants were added to the bleaching solutions the following dyeing in all cases resulted in the enhanced adsorption of dye onto the fibres. However the extent of dye exhaustion was significantly lower as compared with that observed for only washed fibres (Fig. 1, a). This observation is consistent with the published data [6, 21, 22]. The difference in intensity of dye adsorption onto the bleached and unbleached fibres can apparently be explained by the higher amount of cisteic acid groups in bleached fibres which leads to stronger electrostatic repulsion of anionic dye [6].

Table 1. Dye exhaustion by hairs bleached in the presence of surfactants

Surfactants	Dye exhaustion, %
Without surfactants	22.8
NPE	27.8
CAPB	27.4
C/25	30.3
REO 32	29.8
SPS 2532	29.2

The different tensile force-extension behavior of the hair fibres bleached in the presence of C/25 (Fig. 4) and a higher dye sorption onto the fibre (Table 1) as compared with those for assay without surfactant, can be explained by the reduction of disulphide links split occurring due to the shelding action of adsorbed slightly anionic surfactant. Such effect can consequently reduce the formation of cistine and cisteic acid and herewith provide a lower electronegative charge density as well as a lower electrostatic repulsion of anionic dye from the hair fibre.

3.3. Physical properties of hair fibres dyed in the presence of surfactants

The results of mechanical tests for hair fibres bleached and afterwards dyed with acid dye in the presence of

surfactants are shown in Fig. 6. The results allow to assume that the effect of surfactants on fibre damage in dyeing solution depends on the origin of nonionic surfactants, since the breaking force values of dyed hair fibres as well as the elongation at break were significantly distinct (Fig. 6).

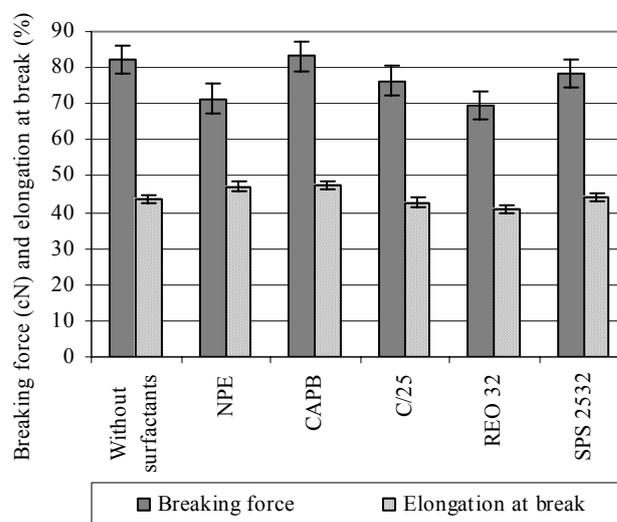


Fig. 6. The effect of surfactants used as dyeing auxiliaries on breaking force and elongation at break of dyed hair fibres

It is surprising that the effect of surfactants on mechanical properties of fibres was absolutely distinct and dependent on the technique of their application in bleaching or in dyeing. Generally, the breaking force values of hair fibres dyed in the presence of surfactants (Fig. 6) as compared with those for bleached in the presence of surfactants and dyed samples (Fig. 5) were lower with the exception of the case with CAPB. It is worthy to note, that the tensile characteristics values, which were established for hair fibres dyed in the presence of CAPB, were higher.

The results of dye exhaustion measurements are presented in Table 2.

Table 2. Influence of surfactants on dye exhaustion in dyeing of bleached without surfactants hair fibres

Surfactants	Dye exhaustion, %
Without surfactants	35.2
NPE	42.1
CAPB	31.1
C/25	39.3
REO 32	38.4
SPS 2532	38.7

The data obtained in this study allow us to assert, that nonionic surfactants used in dyeing enhanced anionic dye exhaustion, while in the case of zwitterionic CAPB dye adsorption was somewhat lower. The values of dye exhaustion for the latter as well as for nonionic surfactants were higher compared with those of dyeing in the absence of surfactants (Table 1). However the breaking force of fibres when the nonionic surfactants were used in dyeing decreased (Fig. 6). Comparison of the results presented in

Fig. 1, b, 5 and 6 allow us to assume, that when slightly cationic polyoxyethylene cocamine C/25 or nonionic alkoxyethoxylate surfactants was used as auxiliaries in hair fibres bleaching before dyeing an effect of fibre protection was observed.

4. CONCLUSIONS

We examined the effect of nonionic and slightly cationic surfactants on damage of hair fibres during bleaching and on acid dye exhaustion in dyeing. The results obtained show that the slightly cationic polyoxyethylene (25) cocamine or ethoxylated coconut fatty acids amide when used as auxiliaries in dyeing at 60 °C improve dye exhaustion. The examination of mechanical properties revealed that the values of breaking force of hair fibres dyed in the presence of polyoxyethylene (25) cocamine or ethoxylated coconut fatty acids amide were higher than those of dyed in the presence of nonylphenolethoxylate. Polyoxyethylene (25) cocamine used in the bleaching solution reduced the damage of hair fibres and enhanced dye exhaustion in dyeing in the same way as when it was used as the dyeing auxiliary.

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