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# Effect of the Interaction between Acid Dye and Nonionic Surfactants on the Adsorption of Dye in Wool Fibre

**Abstract**
*The binding behaviour of 1.4-diamine anthraquinone derivative disulphonic CI Acid Blue 80 dye with nonionic surfactants based on ethoxylated alkylamines and fatty alcohols or alkylphenol (NPE) in aqueous solutions was investigated by means of conductivity measurements as well as by absorption spectroscopy. When the concentration of the surfactants was lower than the critical micellar concentration and the dye/surfactant molar ratio was ca. 1:1, the interaction of the dye with ethoxylated fatty alcohols or NPE was significant and dependent on temperature. It was established that the extent of interaction between the dye and surfactant is in correlation with the results of dye exhaustion from a dyebath as well as the colour strength of dyed wool fibre.*

**Key words:** *nonionic surfactants; interaction; acid dye; exhaustion; wool fibre.*

## Introduction

The factors considered, which in a certain way influence the value of the dye diffusion coefficient and, therefore, determine the rate of dyeing fibres, are the molecular size, affinity, the presence of aggregates, the interaction between all the chemical species present in both phases, as well as the temperature and changes in the physical structure of the substrate [1].

In the case of dyes containing larger or less hydrated molecules, when dyeing is proceeded at low temperatures, the dyeing process is problematic since the uneven and ring dyeing of fibres take place. In the presence of surfactants, considerable changes in the state of the dye in the solution, and morphological changes within the cuticle-cellular membrane complex (CMC) are observed. These changes influence both the thermodynamics and kinetics of dyeing [2-4].

Some studies have indicated [5] that alkoxyethylated nonylphenols have a significant effect on the cellular membrane complex (CMC) of wool fibre, and therefore the absorption of dyes is enhanced at low temperatures. Nonionic alkylphenolethoxylate type surfactants are also known as adducts, forming complexes with wool fibre by means of hydrophobic interaction between hydrophobic sites [6]. However, alkylaminoethoxylates, which are also ascribed to the class of nonionic surfactants, are assessed in a different manner. It is assumed that, due to the combination of nonionic and cationic properties, alkylaminoethoxylates are able to form complexes with acid dyes, facilitating their solubilisation, and as a result these compounds provide a leveling dyeing of wool [3].

J-L. Mieusset *et al.* [7] showed that quick initial surfactant adsorption by wool occurred in the case of dyeing with a 1:2 metal-complex dye regardless of the manner of surfactant application. Surfactant adsorption was followed by a pronounced desorption with the increase in temperature and dye exhaustion [7].

On the other hand, it is known from the relevant literature [8] that a gradual decomposition of the complexes between the leveling agent and dye could occur when the dyeing temperature is increased. Therefore of interest is the stability of alkylamine ethoxylate and anionic dye complexes at different temperatures and the influence of the complexes on dye adsorption on wool fibre.

In the previous research work, we examined the dyeing properties of acid milling dye vis-à-vis wool fibre in the presence of two different nonionic surfactants [9]. The nonionic surfactant based on polyethoxylated alkylphenol showed enhanced dyeing and colour characteristics at low temperatures, whereas the alkylaminoethoxylated product enhanced the dyeing rate at higher temperatures.

In the present paper we report on the interaction of acid dye with different nonionic surfactants in aqueous solutions and the influence of these surfactants on dye uptake by wool fibre at low temperatures.
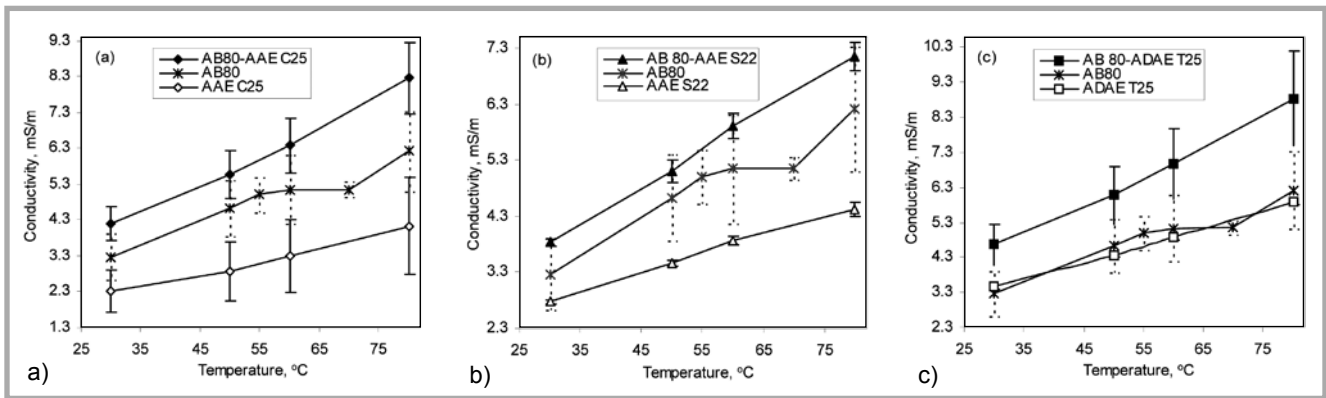
## Experimental

### Materials

Anthraquinone disulphonated dye CI Acid Blue 80 (AB80) was supplied by Clariant and purified by reprecipitation: after dissolving in hot dimethylformamide, it was precipitated in acetone and then filtered. The fabric used for dyeing experiments was Merino wool fabric corresponding to ISO 105/F: 1985 (E).

The commercial nonionic surface-active substances used in this research work are ethoxylated alkylnonylphenol (Lanasan LT, Clariant; active content 83%), an alkyl amine ethoxylate and glycolic ether mixture (Rucogal MSC, Rudolf Chemie; active content 38%), and fatty alcohol ethoxylate (Felosan RG, CHT; active content 80%). Lanasan LT (NPE) is suited to be used in low temperature wool dyeing. Rucogal MSC (AAE MSC) is intended for use in wool and polyamide leveling dyeing**.** Felosan RG (REO 9) is developed and produced by Chemische Fabrik Tubingen (CHT) as a surfactant candidate for substituting highly toxic (but still used in large quantities in textile processes) ethoxylated alkylphenols (NPE) [10].

Alkoxylated alcohols and alkylamines surfactants obtained from Akzo Nobel Surface Chemistry were selected as products of high active substance content with a definite degree of ethoxylation. Ethoxylated alkylamines Ethomeen S/17 (AAE S17), Ethomeen S/22 (AAE S22) and Ethomeen C/25 (AAE C25) are, respectively, polyoxyethylene(7)oleylamine, polyoxyethylene (12)oleylamine and polyoxyethylene(15) cocamine.

Ethoxylated alkyldiamines Ethoduomeen T/25 (ADAE T25) and Ethoduomeen T/13 (ADAE T13), the chemical formulas of which are N, N′, N′-polyoxyethylene(15)-N-tallow-1.3-diamino-propane and N, N′, N′-tris (2-hydroxyethyl)-N-tallow-

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**Figure 1.** *Influence of temperature on the conductivity of AB80 solution containing a) AAE C25, b) AAE S22, c) ADAE T25 surfactant:* $c_d = 0.18$ *mmol* $dm^{-3}$.

1.3-diaminopropane, respectively, were also used in this study. All the ethoxylated alkyl amines and alkyl diamines contained 100% active substances. These surfactants are characterised as being mildly cationic in an acid media, naturally becoming more nonionic with increasing ethylene oxide content. Alkoxylated alcohol Berol 175 (Akzo Nobel; active content 90%) (REO 7.5) (hydrophobe – $C_{12} – C_{16}$) with 7.5 EO and HLB 12.5 (active content 90%) was selected as a principal surfactant, representing linear ethoxylated alcohol groups.

**Measurements**

The surfactants were added to the dye solutions and the concentrations used were c.a. 0.18 (lower than the critical micellar concentration) and 1.7 mmol $dm^{-3}$ of the dye solution examined.

Dye/surfactant solutions were prepared in advance by mixing necessary amounts of solutions of dye and surfactants to obtain the required concentration of the solution. The molar ratio of dye and surfactant $[C]_d:[C]_{surf}$ in all the essays was ca.1:1. The value of pH of the solutions

was 4.2 (adjusted using acetic acid solution). Distilled water was used for the preparation of the solutions.

The temperature of the solutions during conductivity measurements was in the range of 30 to 80 °C. It was controlled by thermostatic heating. Before the conductivity measurements, the solutions were stored for 1 hour at the measurement temperature in a water thermostat. The electric conductivity was measured on a N5721M conductometer with a CDC-344 conductivity cell and a T-801 temperature sensor. The nominal constant of the conductivity cell was 3.16 $cm^{-1}$. The measurement procedure was repeated 3 times for each solution.

The degree of dye/surfactant interaction was measured by determining the electric conductivity of the dye solutions in the presence of surfactants using the method proposed by D. Jocic [11]. The results of the conductivity measurement were formulated by the conductance ratio (*CR*), which is considered as a measure of the degree of dye/surfactant interaction. The conductance ratio was calculated by Equation 1:

$$CR = \frac{K_{DS} - K_S}{K_D} \qquad (1)$$

where $K_{DS}$ is the electric conductivity of the dye/surfactant solution, mS/m, $K_S$ is the electric conductivity of the solution of surfactant only, and $K_D$ is the electric conductivity of the solution containing only dye.

Using the above mentioned methodology [11], it was considered that there was no interaction between the dye and surfactant when the conductance ratio *CR* value is 1, and the lower the *CR*, the more pronounced the interaction between the dye and surfactant is.

Alternatively, the ultraviolet-visible (UV-vis) spectra recorded for the prepared dye/surfactant solutions were examined. The visible absorption spectra of the aqueous dye/surfactant solutions with dye and surfactant concentrations of 0.18 and 1.7 mmol $dm^{-3}$ and temperatures of 30 and 60 °C were measured using a GILFORD UV-vis Spectrofotometer, which had five quartz cells (10.0 mm in light pass length).
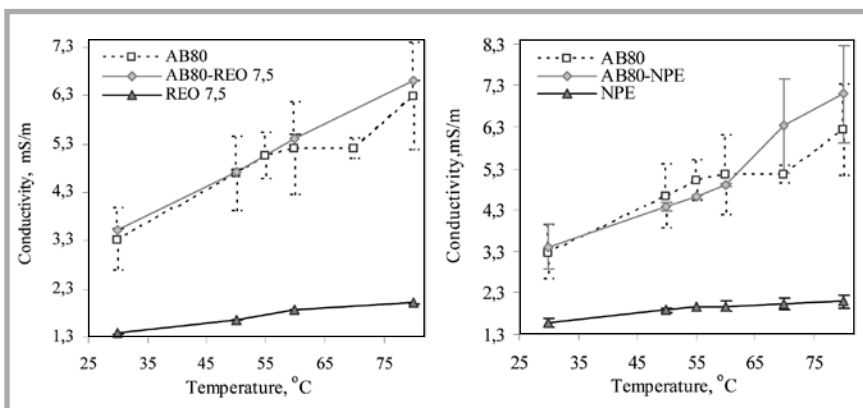


**Figure 2.** *Influence of temperature on the conductivity of dye solution (* $c_d = 0,18$ *mmol* $dm^{-3}$*) containing REO 7.5 (a) and NPE (b) surfactants.*
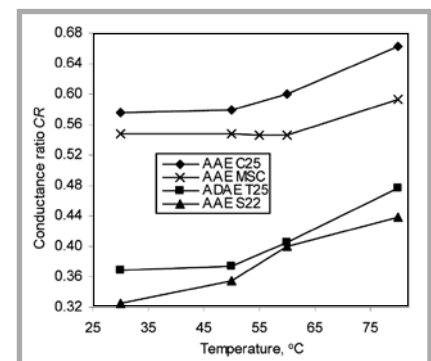


**Figure 3.** *Influence of temperature on the conductance ratio (CR) for AB80 solutions containing alkylamine ethoxylates (AAE) surfactants,* $c_d = 0.18$ *mmol* $dm^{-3}$.

## Dyeing procedures

Dyeings were carried out using ISAL AG BASEL laboratory apparatus with steel cylinders to girdle the samples. The fabric samples were of 10 x 20 cm with a mass of 1.0 g. The dye solution was buffered at pH 4.2 ± 0.05 with a 0.1 M acetate buffer. In all dyeings the liquor-to-fiber ratio was 200:1, the dye concentration was ca. 0.18 mmol dm$^{-3}$ and the temperature 60 °C or 85 °C. Dyeings were carried out for 1 h in the absence or in the presence of surfactants, the concentration of which was ca. 0.18 mmol dm$^{-3}$. At the end of the dyeing, the samples were rinsed thoroughly in water and dried in the open air. Dye concentration in the dyebath was measured at the beginning of the dyeing and after 60 min.

## Dyeing characteristics

Dye absorption was calculated from the difference in dye concentration before and after dyeing. The dye concentration in liquor was measured in a 10 mm cell using a Spectronic Unicom Genesys TM spectrophotometer. On completion of the dyeing, the wool fabric samples were removed from the liquor, rinsed in water to remove any loosely adsorbed dye and dried in air.

The percentage of dyebath exhaustion $E$ was calculated using Equation 2:

$$E = \left( \frac{A_0 - A_d}{A_0} \right) * 100 \qquad (2)$$

where $A_o$ and $A_d$ are the adsorbances at 590 nm ($\lambda_{max}$) of the dye in the initial dyebath and of the residual dye in the dyebath after dyeing, respectively.

For the dyed fabric samples, light diffuse reflectance measurements were performed using a Spektroflash SF 450 PLUS spectrophotometer (Datacolor), and colour yield values were calculated according to the Kubelka-Munk function ($K/S$) by Equation 3 [12]:

$$K / S = \frac{\left(1 - R\right)^2}{2R} \qquad (3)$$

where $K$ is the absorbance, $S$ is the scattering, $R$ is the reflectance.

## ■ Results and discussion

### Conductivity and conductance ratio for the determination of dye/surfactant interaction

**Figures 1.a, 1.b, 1.c** and **2.a, 2.b** show the conductivity values versus the temperature for AB80 dye/surfactant solutions compared with corresponding plots for the solutions of the dye and of the surfactant only. The plots show that in the range of 30 to 50 (60) ºC, cocamine ethoxylate (AAE C25) **(Figure 1.a)**, oleylamine ethoxylate (AAE S22) (**Figure 1.b**) and particularly tallow diamine ethoxylate (ADAE T25) (**Figure 1.c**) increase the conductivity of the dye solution, while alkoxylated fatty alcohol REO 7.5 and NPE do not change it significantly **(Figure 2)**. However, at a temperature above 60 °C, the conductivity values of the dye solutions, especially in the presence of NPE, were significantly higher compared with those observed for temperatures below 60 ºC.

**Figures 3** and **4.a, 4.b** show the effect of temperature on the extent of the conductance ratio for dye solutions containing the distinct nonionic surfactants. Curves representing the variation in the conductance ratio allow to assume that the extent of dye/surfactant interaction is significantly higher (for a lower conductance ratio ) for AAE S22 and ADAE T25 (**Figure 3**) compared with that for REO 7.5 and NPE (**Figure 4.a**). Cocoamine ethoxylate AAE C25 is the exception as it manifests a relatively weak interaction with the dye. A more or less regular change in the conductance ratio $CR$ with the temperature, as is evident from the plots presented in **Figures 3** and **4**, allows to draw conclusions about a strong decrease in the extent of dye/surfactant interaction when the temperature of the solution increases from 50 to 60 ºC. However, this is not the case for NPE (**Figure 4.a** and **4.b**), since the ability of this surfactant to interact with dye seems more likely to be dependent on the temperature. The complexity of the behaviour of NPE vis-a-vis dye molecules (**Figure 4.a** and **4.b**)

as regards the variation in temperature could be due to the specific composition of this surfactant, which is presumably composed of a short-chain ethoxylated alkylnonylphenol and a longer-chain dispersing agent [3].

The results presented in **Figures 4.a and 4.b** show that, having increased the concentration of surfactants in the dye solution ten times, the values of the conductance ratio $CR$ increase significantly, which indicates a lowering of the extent of interaction between the dye and surfactants.

### UV-vis spectra of dye/surfactant solutions

The UV-vis spectrum of AB80 dye in an aqueous medium at pH 4.2 and 30 °C has $\lambda_{max}$ at 582.0 nm and 626.25 nm (**Figure 5**, see page 120). The spectral changes observed upon addition of nonionic surfactants are illustrated in **Figure 5** and **Table 1** see page 120. A significant reduction in the visible light absorbance of the main bands of AB80 is observed when the equivalent molar concentration of AAE MSC or AAE C25 is in the solution. However, a smaller decrease in absorbance intensity for NPE and a slight increase in intensity for REO 7.5 are observed. The data for the batochromic shift in the spectra (**Table 1**) allow to assume that soluble complexes of the dye and AAE MSC or AAE C25 are formed, and, as a result, a significant loss in intensity at $\lambda_{max}$ ca. 585 and 630 nm occurs.

When a surfactant containing a tertiary amine group, e.g. AAE C25 or ADAE T25 is used, changes in the UV-vis spectra are observed (**Table 1**), suggesting a relatively strong, but uneven extent of interaction of both these surfactants with the dye in the temperature region of 30° to 60 °C . Surprisingly, the results of the
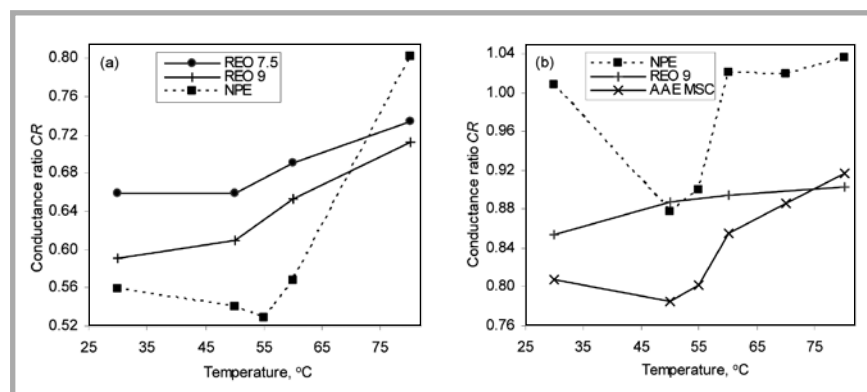
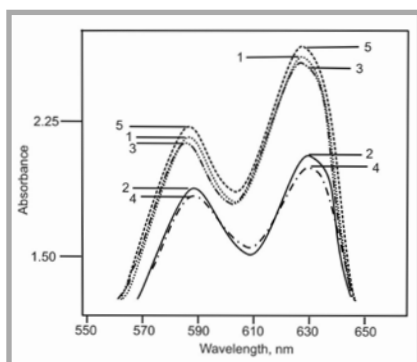**Figure 4.** *Influence of temperature on the conductance ratio (CR) for AB80 solutions containing alkoxylated alkohol and NPE, $c_d = 1.7$ mmol dm$^{-3}$.*

**Figure 5.** *Absorption spectrum of AB80 aqueous solution in the presence of nonionic surfactants (c$_d$ = 0.18 mmol) at pH 4.2 and 30 °C: no surfactant (1), AAE MSC (2), NPE (3), AAE C25 (4), REO 7.5 (5).*

conductivity measurements (**Table 2**) show that AAE C25 in an aqueous dye solution does not interact with the dye as strongly as ADAE T25.

Comparison of the spectral data (**Table 1**) with that of the conductivity measurements presented in **Table 2** allows to assume that attractive electrostatic interaction is a major driving force for AAE S22

and ADAE T25, while AAE MSC and AAE C25 contribute to the hydrophobic interaction with the dye. The changes that occur in the UV-vis spectra of the dye upon addition of REO 7.5 or NPE were less pronounced, which indicates a lower interaction between the dye and surfactant. A similar behaviour in two nonionic surfactants, namely, polyoxyethylene nonyphenolether (NPE-12) and polyoxyethylene stearylamine in direct dyes solutions was observed by S.Pegiadou-Koemtzopoulou et al. [13]. The authors measured surface tension as well as critical micellar concentration and observed the difference in the absorbance of dyes in the presence of both surfactants. According to S.Pegiadou-Koemtzopoulou et al. [13], this observation confirmed the formation of hydrophobic complexes in NPE-12 and hydrophilic complexes in stearylamine with dyes in aqueous solutions.

The data obtained indicate that a temperature of ca. 50 °C is a point above which an important change in the origin of acid anthraquinone dye/surfactant interaction occurs and results in a decrease in interaction.

The spectral data as well as the results of the conductance ratio (**Figures 3** and **4**) obtained in this study support the conclusion that the extent of dye interaction with REO 7.5 or NPE is lower compared with that of AAE S22 or ADAE T25.

**Dyeing**

The results of dyeing with AB80 in the presence of nonionic surfactants are presented in **Tables 2** and **3**. Comparison of the data obtained from dyeing with conductance ratio values *CR* for distinct dye/surfactant systems shows an obvious relationship between the values of *CR* representing the extent of dye/surfactant interaction and the characteristics of dyeing. The results also show that cocamine ethoxylate (AAE C25), similar to the linear fatty alcohol ethoxylate REO 7.5, manifests a relatively weak interaction with the dye in terms of conductivity measurements (**Table 2**) and does not reduce dye exhaustion from the dyebath as significantly as other AAE.

Thus, the results presented above suggest that the presence of the tertiary amino group in the nonionic surfactant molecule, which is able to accumulate a positive charge in acid media, is not the only factor determining the behaviour of surfactants in an aqueous dye solution. It can be assumed that the role of hydrophobic interaction between the dye and surfactant in an aqueous solution is significant and should be seriously considered during the selection of a surfactant as an auxiliary for wool dyeing.

The relative efficiency of ethoxylated alkylamines when applied in low temperature dyeing of wool fibre falls into the following range of activity reduction: AAE C25, AAE MSC, AAE S22 and ADAE T25 (**Tables 2** and **3**). Ethoxylates of fatty alcohols and nonylphenol ethoxylate fall into the following range of CR reduction, respectively: REO 7.5, REO 9, NPE.

The results presented in **Table 3** show that ethoxylated alkyl diamine *Ethoduomeen T/13* N,N'N'-Tris(2-hydroxyethyl)-N-tallow1,3-diaminopropane (ADAE T13), which contains two amine groups and a few EO units, is there fore relaively hydrophobic (dispersible/insoluble in water) and its inclined to interact with the dye due to hydrophobic interaction, which causes the aggregation of the dye. A high level of dye aggregation in the dye solution results in non-level sedimentation

**Table 1.** *Absorbance values changes and batochromic shift at λ$_{max}$ ca. 626 nm for AB/surfactant solutions.*

| Surfactant | Absorbance | λ$_{max}$ shift, nm | Loss in intensity of dye **, % |
|---|---|---|---|
| 30 °C temperature | | | |
| Without surfactant | 2.6591 | – | – |
| NPE | 2.5906 | 0.75 | 2.57 |
| REO 7.5 | 2.6182 | 1.00 | 1.54 |
| AAE MSC | 2.1244 | 3.25 | 20.11 |
| AAE C25 | 2.0210 | 4.25 | 24.00 |
| AAE S22 | 1.9570 | 4.0 | 26.41 |
| ADAE T25 | 1.7630 | 5.0 | 33.71 |
| 60 °C temperature | | | |
| Without surfactant | 2.5744 | – | – |
| NPE | 2.5233 | 0.25 | 1.98 |
| REO 7.5 | 2.5696 | 0.25 | 0.18 |
| AAE MSC | 2.1202 | 2.75 | 17.64 |
| AAE C25 | 2.1022 | 2.75 | 18.34 |
| AAE S22 | 1.8934 | 5.00 | 23.49 |
| ADAE T25 | 1.6967 | 5.50 | 31.45 |

* absolute deviation – 0.25 nm
** comparing with dye only solution

**Table 2.** *Influence of conductance ratio of AB80/surfactant solutions on dyeing characteristics at 60 °C.*

| Surfactant | *CR* | Dye exhaustion, % | Colour strength *K/S* |
|---|---|---|---|
| AAE C25 | 0.60 | 83.9 | 10.7 |
| AAE MSC | 0.55 | 56.3 | 7.15 |
| AAE S22 | 0.40 | 50.4 | 6.25 |
| ADAE T25 | 0.41 | 40.9 | 4.15 |
| REO 7.5 | 0.69 | 96.2 | 14.1 |
| REO 9 | 0.65 | 98.55 | 14.25 |
| NPE | 0.57 | 98.95 | 13.6 |
| Without surfactant | – | 79.7 | 9.25 |

**Table 3.** *Influence of alkylamine ethoxylates (AAE) on dyeing rate with AB80 at 85 °C.*

| Surfactant | Dye exhaustion, % | Colour strength K/S |
|---|---|---|
| AAE MSC | 80.5 | 14.5 |
| AAE S17 | 43.1 | 6.6 |
| AAE S22 | 68.1 | 12.6 |
| AAE C25 | 99.7 | 19.1 |
| ADAE T25 | 63.4 | 11.5 |
| ADAE T13 | 43.8 | 3.6 |
| Without surfactant | 99.8 | 17.4 |

of dye on the surface of fibre. Therefore, lower visible light absorption and a lower value of *K/S* could be obtained compared with that for dyeing in the presence of AAE S17. The latter surfactant is more hydrophilic compared with ADAE T13 and contains only one amine group, and thus the interaction between the surfactant and the dye was lower; therefore the adsorption of dye on the surface of the fiber could be more uniform.

Significantly, the range of surfactants examined with respect to dye/surfactant interaction in terms of conductivity measurements (inversely proportional to the conductance ratio *CR*) is in correlation with the range of surfactant activity with respect to colour strength and dye exhaustion from the dyebath stated above. In other words, the range of the examined nonionic surfactant efficiency in wool fibre dyeing with anthraquinone AB80 is dependent on *CR* values for the corresponding variation in the dye/surfactant solution.

The results presented in **Table 3** show that when dyeing was proceeded at 85 °C in the presence of AAE C25, the dye exhaustion was already complete and the extent of the exhaustion was similar to that for REO 7.5 **(Table 2)** when dyeing at 60 °C temperature.

AAE S22 containing 12 EO gives a higher dye exhaustion fom the dyebath and provides a higher colour strength *K/S* of dyed fabric compared with that of AEE S17 containing 7 EO **(Table 3)**. This is a demonstration of the effect of ethoxylated alkyl amines on dye solubilisation as a means of producing dye exhaustion in the dyebath. Thus the value of dye exhaustion for AAE S17 presented in **Table 3** supports the conclusion that alkylamine ethoxylates are inclined to form complexes (a lower conductance ratio *CR*, and a relatively strong interaction with anionic dye, **Figure 3**), and that the amount of EO

in the surfactant is very important factor as it affects dye exhaustion in the dyebath.

At 60 °C the dye exhaustion **(Table 2)** as well as the colour strength *K/S* of the dyed specimens was enhanced when dyeing was performed in the presence of AAE C25, while the other AAE type surfactants used in this study significantly reduced the extent of the dye exhaustion and colour strength. These data allow to assume that a lower dye surfactant interaction, evaluated with the use of conductivity and UV-vis absorption measurements of dyeing liquor, causes higher dye exhaustion as well as strength of colour of the dyed wool fabric.

The results in **Table 2** show that the two surfactants – REO- 7.5 and REO- 9, based on alkoxyethoxylates produced and supplied by different enterprises, are similar with respect to the effect on dye exhaustion from the dyebath and to the colour strength *K/S* of dyed fabric in the presence of these surfactants . In connection with the statement made above, the results presented in **Figure 4.a** allow to conclude that the alkoxyethoxylated surfactant used in this study behaves in the dyebath in a similar way to the dye in terms of the degree of interaction and the dependency of temperature on this interaction.

The results of dyeing at 85 °C showed that dye exhaustion was almost complete in the presence of fatty alcohol ethoxylates as well as in the absence of auxiliaries. However, there was a significant difference in the values of the colour strength of the dyed fabric specimens measured using a Datacolor spectrophotometer.

## ■ Conclusions

Anthraquinone acid dye/nonionic surfactant systems were studied by measuring the specific conductance and UV-vis absorption spectrometry of aqueous solutions, and the extent of interaction between the dye and surfactant was estimated. The results reveal that the extent of dye/surfactant interaction is significant when the mollar ratio is ca. 1:1, and the concentration of alkoxyethoxylates used is lower than the critical micellar concentration. The alkylaminethoxylates used in this study mostly manifest a higher ability to form complexes with the dye compared with the alkoxyethoxylates. With an increase in temperature, significant changes in the extent of dye/surfactant interaction occur in terms of the conductance ratio measurement .

The results of the investigation highlight the importance of both the hydrophobicity and weak protonisation of surfactants in the interaction of a surfactant with anionic dye, as well as the influence of the extent of interaction on the rate of wool fibre dyeing. Enhanced dyeing at 60 °C can be achieved with nonionic alkoxy-ethoxylates as well as with polyoxyethylated nonylphenol, rather than with weak, cationic and more hydrophilic alkylaminoethoxylates.

The range of surfactants examined with respect to dye/surfactant interaction in terms of conductivity measurements is in correlation with the range of surfactant activity with respect to dye exhaustion from the dyebath and colour strength of the dyed fabric.

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