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Liquid—Liquid Extraction of Basic Yellow 28, Basic Blue 41, and Basic Red 46 Dyes from Aqueous Solutions with Reverse Micelles

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ABSTRACT: The removal of 2-[[(4-methoxyphenyl)methylhydrazono]methyl]-1,3,3-trimethyl-3*H*-indolium methyl sulfate (Basic Yellow 28), 2-[*N*-ethyl-4-[(6-methoxy-3-methyl-1,3-benzothiazol-3-ium-2-yl)diazenyl]anilino]ethanol methyl sulfate (Basic Blue 41), and *N*-benzyl-4-[(1,4-dimethyl-1,5-dihydro-1,2,4-triazol-1-ium-5-yl)diazenyl]-*N*-methylaniline bromide (Basic Red 46) cationic textile dyes from model wastewater by liquid—liquid extraction with reverse micelles was performed with isopentanol as a solvent and sodium dodecylsulfate as an anionic surfactant. The dye concentration in both aqueous and organic phase was determined by visible spectrophotometry analysis. The influence of dye and surfactant initial concentrations on the solute partition coefficient and percentage of removal were studied. The partition coefficient and percentage of removal increase with the decrease in initial dye concentration and with the increase in surfactant concentration.

INTRODUCTION

Many fields of up-to-date technology, including the textile industry, use synthetic dyes to color their products.¹ Dyes can be classified according to its chemical structure or the method used to fix it into textile fiber.² In textile industries, the removal of textile dyes from wastewater has been a subject of great interest in the past decades and has been brought to discussion because of new environmental laws, restriction on water availability, and charge for its use. On the other hand, this subject is of major concern; once the discharge of wastewater in the environment may be hazardous for human health and the ecosystem, for example, azo dyes are reduced to potentially hazardous aromatic amines under anaerobic conditions.^{3,4}

Because the conventional biological treatments are less effective for wastewater containing azo dyes,⁵ in open literature, various techniques have been proposed to remove dyes from water, such as oxidation,^{6,7} adsorption,^{8–11} flocculation-precipitation,¹² membrane separation,¹³ electro-coagulation,^{14,15} photocatalysis,^{16,17} ultrasound^{18,19} and liquid—liquid extraction.^{20–24}

Liquid—liquid extraction of dyes from water using reverse micelles was reported by Pandit and Basu.²⁰ The authors studied the removal of methyl orange (anionic dye) with the cationic surfactant hexadecytrimethyl ammonium bromide (HTAB) and methylene blue (cationic dye) with anionic surfactant sodium dodecylbenzene sulfonate (SDBS) from water, using isopentanol as an extractant. Reverse micelles occur when a surfactant molecule is dissolved in organic solvent in concentrations superior to the critical micellar concentration, and its hydrophilic groups are sequestered inside the micelle while hydrophobic ones remain outside.

In the present work, the removal of 2-[[(4-methoxyphenyl)methylhydrazono]methyl]-1,3,3-trimethyl-3H-indolium methyl sulfate (Basic Yellow 28, BY28), 2-[*N*-ethyl-4-[(6-methoxy-3-methyl-1,3benzothiazol-3-ium-2-yl)diazenyl]anilino]ethanol methyl sulfate (Basic Blue 41, BB41), and *N*-benzyl-4-[(1,4-dimethyl-1,5-dihydro-1,2,4-triazol-1-ium-5-yl)diazenyl]-*N*-methylaniline bromide (Basic Red 46, BR46) cationic dyes from model wastewater using isopentanol as solvent and sodium dodecylsulfate (SDS) as anionic surfactant was studied. The effect of initial dye concentration and surfactant concentration on the solute partition coefficient and percentage of removal were determined with liquid—liquid equilibrium (LLE) experiments, which is necessary for the design of a liquid—liquid extraction process.

EXPERIMENTAL SECTION

Materials. The cationic dyes Basic Yellow 28, Basic Blue 41, and Basic Red 46 were supplied by Ciba Industry; sodium dodecylsulfate (purity 0.90 in mass fraction, critical micellar concentration = 0.0139 mg·L⁻¹)²⁵ was purchased from Synth, and isopentanol (purity > 0.985 in mass fraction) was purchased from Vetec. Figure 1 shows the chemical structure of dyes, and Table 1 shows the physical properties of dyes and chemicals used in this work.

Procedure. Initially, the visible spectra of Basic Yellow 28, Basic Blue 41, and Basic Red 46 cationic dyes were obtained for the determination of maximum absorbance wavelengths (λ) using a Varian Cary Win 50 UV spectrophotometer, accurate to \pm 0.0001, as shown in Figure 2. The λ values are shown in Table 1 and were used for (a) the construction of absorbance (A) versus dye concentration calibration curves for water-rich and isopentanol-rich phases and (b) the analysis of equilibrium phases. All experiments were made at T = 298.2 K and atmospheric pressure (\approx 95 kPa).

In liquid—liquid equilibrium experiments, the method used here is similar to that used by Pandit and Basu.^{20,21} Stock solutions of known composition (mg·L⁻¹) of dye in water and SDS in isopentanol were prepared in a Shimadzu AX200 analytical balance, accurate to \pm 0.0001 g. According to the scheme of experimental procedure shown in Figure 3, known masses of each stock solution

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(a) 2-[[(4-methoxyphenyl)methylhydrazono]methyl]-1,3,3-trimethyl-3H-indolium methyl sulfate



(b) 2-[N-ethyl-4-[(6-methoxy-3-methyl-1,3-benzothiazol-3-ium-2-yl)diazenyl]anilino]ethanol methyl sulfate



(c) N-benzyl-4-[(1,4-dimethyl-1,5-dihydro-1,2,4-triazol-1-ium-5-yl)diazenyl]-Nmethylaniline bromide



Table 1. Physical Properties of Chemicals Used in This Work

	molecular mass	maximum absorbance wavelength ($\lambda/{ m nm}$)		
compound	$g \cdot mol^{-1}$	literature	experimental	
isopentanol	88.17			
SDS	288.38		327.0	
Basic Yellow 28	433.52	437.0 ²⁶	438.7	
Basic Blue 41	482.57	609.0 ²⁷	610.0	
Basic Red 46	403.32	529.0 ²⁶	530.4	



Figure 2. Absorbance (A) versus wavelength (λ) visible spectra for Basic Yellow 28 (solid line), Basic Blue 41 (dotted line), and Basic Red 46 (dashed line).

were added to an equilibrium cell connected to a Microquímica MQDTC99-20 thermostatic bath (precision of \pm 0.1 K). The fourcomponent heterogeneous mixture was vigorously agitated with a Fisatom 752A magnetic stirrer for 30 min to ensure intimate contact between phases. Then, the system was left to settle for 12 h to achieve liquid—liquid equilibrium, with the formation of two



Figure 3. Scheme of the experimental procedure.

Table 2. Absorbance (A) versus Dye Concentration in Water and Isopentanol

Basic Yellow 28		Basic I	Basic Blue 41		Basic Red 46	
$mg \cdot L^{-1}$	Α	$mg \cdot L^{-1}$	Α	$mg \cdot L^{-1}$	Α	
Water						
87.0	0.7045	96.8	0.5493	32.3	0.4075	
43.5	0.3531	48.4	0.2837	25.8	0.3317	
34.8	0.2845	38.7	0.2341	19.4	0.2430	
26.1	0.2192	29.0	0.1820	12.9	0.1691	
17.4	0.1519	9.7	0.0569	6.5	0.0885	
4.7	0.0491	5.2	0.0362	3.2	0.0579	
3.5	0.0487	3.9	0.0267			
Isopentanol						
107.2	0.5350	119.3	0.1789	79.6	0.7738	
53.6	0.2811	59.7	0.0954	39.8	0.3848	
32.2	0.1679	47.7	0.0748	31.8	0.3117	
21.4	0.1045	35.8	0.0595	23.9	0.2302	
10.7	0.0557	23.9	0.0339	15.9	0.1340	
		11.9	0.0131	8.0	0.0637	

clear liquid phases: the colored solvent phase containing the dye encapsulated in the reverse micelles and the limpid aqueous phase. Preliminary tests showed that these times are enough to attain equilibrium. In each equilibrium phase, samples were collected using microsyringes (Hamilton, volumes (25 to 500) μ L). Organic phase samples were diluted in isopentanol, and aqueous phase samples were diluted in water before absorbance measurements. Dye concentrations in each phase were determined using previously obtained calibration curves.

Experimental data sets were divided according to the influence on dye extraction performance by:

- (i) Dye concentration: analyses of the effect of initial dye concentration (before LLE) on extraction data with constant initial SDS concentration in isopentanol.
- (ii) SDS concentration: analyses of the effect of initial SDS concentration (before LLE) on extraction data with constant initial dye concentrations in water.

In all experiments the isopentanol-to-water mass ratio used was 0.41. The dye extraction performance was determined with the partition coefficient (K) and the percentage of removal (E),





Figure 4. Calibration curves of absorbance versus concentration of dye in (a) water and (b) isopentanol. ■, BY28; ●, BB41; ▲, BR46.

given by eqs 1 and 2.

$$K = \frac{c_{\rm dye}^{\rm org}}{c_{\rm dye}^{\rm dq}} \tag{1}$$

$$E = \frac{c_{\rm dye}^{\rm feed} - c_{\rm dye}^{\rm aq}}{c_{\rm dye}^{\rm feed}} \cdot 100 \tag{2}$$

where c_{dye}^{feed} is the initial dye concentration in water; c_{dye}^{aq} is the final dye concentration in the aqueous phase after LLE; and c_{dye}^{org} is the dye concentration in the organic phase after LLE.

RESULTS AND DISCUSSION

Calibration Curves. Table 2 shows absorbance measurements of known aqueous (dye + water) and organic (dye + alcohol) mixtures. By applying linear regression on these data, the calibration curves, eqs 3 to 8, were obtained and used to determine dye concentration in each phase after equilibrium.

•	0.					
	before LLE	after	LLE			
	\mathcal{C}_{dye}^{feed}	c_{dye}^{aq}	$c_{\rm dye}^{\rm org}$			
data set	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	Κ	Е	
Basic Yellow 28						
1	401.08	3.35	540.50	163.22	99.16	
	551.41	5.52	661.72	120.30	99.00	
	701.68	7.11	713.59	100.37	98.99	
		Basic Blue 4	41			
2	401.08	1.72	2899.54	1685.15	99.57	
	521.35	3.18	4647.68	1462.15	99.39	
	671.63	5.04	5732.86	1137.38	99.25	
3	401.08	85.10	2029.36	23.85	78.78	
	551.41	300.80	2082.60	6.92	45.45	
	701.68	625.26	2221.25	3.55	10.89	
		Basic Red 4	6			
4	1060.83	219.52	1030.75	4.70	79.31	
	1631.07	417.15	1335.90	3.20	74.42	
	1900.95	605.58	1627.81	2.69	68.14	
5	551.41	186.06	266.48	1.43	66.26	
	701.68	250.69	296.44	1.18	64.27	

Table 3. Partition Coefficient (K) and Percentage of Removal

(E) Varying Dye Concentration



Figure 5. Effect of dye concentration on distribution coefficient for data sets 1 to 5. **I**, BY28, $c_{SDS} = 284.2 \text{ mg} \cdot \text{L}^{-1}$; **O**, BB41, $c_{SDS} = 284.2 \text{ mg} \cdot \text{L}^{-1}$; **O**, BB41, $c_{SDS} = 123.6 \text{ mg} \cdot \text{L}^{-1}$; **A**, BR46, $c_{SDS} = 617.7 \text{ mg} \cdot \text{L}^{-1}$; **A**, BR46, $c_{SDS} = 123.6 \text{ mg} \cdot \text{L}^{-1}$.

Equations 3 to 5 were used to calculate the dye concentration in unknown aqueous phases.

$$A_{\rm BY28}/{\rm L}\cdot{\rm mg}^{-1} = 34324 c_{\rm dye}^{\rm aq}$$
 $R^2 = 0.999$ (3)

$$A_{\rm BB41}/{\rm L} \cdot {\rm mg}^{-1} = 27297 c_{\rm dye}^{\rm aq}$$
 $R^2 = 0.998$ (4)

$$A_{\rm BR46}/{\rm L}\cdot{\rm mg}^{-1} = 39332 c_{\rm dye}^{\rm aq}$$
 $R^2 = 0.999$ (5)



Figure 6. Effect of initial dye concentration on the percentage of removal for data sets 1 to 5. **I**, BY28, $c_{SDS} = 284.2 \text{ mg} \cdot \text{L}^{-1}$; **O**, BB41, $c_{SDS} = 284.2 \text{ mg} \cdot \text{L}^{-1}$; **O**, BB41, $c_{SDS} = 123.6 \text{ mg} \cdot \text{L}^{-1}$; **A**, BR46, $c_{SDS} = 617.7 \text{ mg} \cdot \text{L}^{-1}$; **A**, BR46, $c_{SDS} = 123.6 \text{ mg} \cdot \text{L}^{-1}$.

Table 4. Partition Coefficient (K) and Percentage of Removal(E) Varying SDS Concentration

	befor	before LLE after LLE					
	$c_{\rm dye}^{\rm feed}$	$c_{ m SDS}$	$c_{\rm dye}^{\rm aq}$	$c_{\rm dye}^{ m org}$			
data set	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	K	Ε	
Basic Yellow 28							
6	100.3	654.70	1.55	195.95	126.31	98.45	
	100.3	1394.84	0.83	201.10	242.33	99.17	
	100.3	1764.50	0.53	250.77	470.15	99.47	
		Ba	sic Blue 41				
7	100.3	802.82	1.17	1845.14	1580.19	98.84	
	100.3	1024.91	1.00	1816.23	1812.29	99.00	
	100.3	1394.84	0.74	1770.26	2390.85	99.26	
	100.3	1838.39	0.54	1646.86	3049.00	99.46	
Basic Red 46							
8	309.5	2244.64	5.35	373.32	75.05	98.27	
	309.5	3424.57	3.68	372.40	101.79	98.81	
	309.5	4822.12	2.66	379.43	147.55	99.14	
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Equations 6 to 8 were used to calculate the dye concentration in unknown organic phases.

$$A_{\rm BY28}/{\rm L} \cdot {\rm mg}^{-1} = 26754 c_{\rm dye}^{\rm org}$$
 $R^2 = 0.998$ (6)

$$A_{\rm BB41}/\rm L \cdot mg^{-1} = 9111.3c_{\rm dye}^{\rm org}$$
 $R^2 = 0.994$ (7)

$$A_{\rm BR46}/{\rm L}\cdot{\rm mg}^{-1} = 39559 c_{\rm dye}^{\rm org}$$
 $R^2 = 0.999$ (8)

The correlation coefficients were all above 0.99. Figure 4 shows the calibration curves obtained for the three dyes in water and isopentanol.

Effect of Initial Dye Concentration. Here, the SDS concentration and solvent-to-water mass ratio were maintained constant, and the effect of initial dye concentration on liquid—liquid



Figure 7. SDS concentration effect on the partition coefficient for data sets 6 to 8. \blacksquare , BY28, $c_{dye}^{feed} = 100 \text{ mg} \cdot \text{L}^{-1}$; \blacklozenge , BB41, $c_{dye}^{feed} = 100 \text{ mg} \cdot \text{L}^{-1}$; \bigstar , BR46, $c_{dye}^{feed} = 310 \text{ mg} \cdot \text{L}^{-1}$.

extraction for discoloration of water was performed. An increase in the initial dye concentration simulates an increasing amount of contaminants in a real stream. The concentration of SDS in isopentanol was 284.2 mg·L⁻¹ for BY28 (data set 1), (284.2 and 123.6) mg·L⁻¹ for BB41 (data sets 2 and 3), and (617.7 and 123.6) mg·L⁻¹ for BR46 (data sets 4 and 5). Table 3 and Figures 5 and 6 show the measured composition, partition coefficient, and percentage of removal for data sets 1 to 5.

It is verified that the partition coefficients decrease with an increasing of initial dye concentration; the percentage of removal behaves in the same manner. These results can be explained by a reduction in the capacity of organic phase to solubilize the dye and remove it from water, that is, organic phase tends to become saturated of dye, and the dye concentration in the water phase is enhanced.

Comparing data sets 1 and 2, it is verified that BY28 presents partition coefficients 1 order of magnitude lower and a slightly lower percentage of removal from water than BB41. Comparing data sets 3 and 5, BB41 presents greater partition coefficients but a lower percentage of removal than BR46. An explanation for these phenomena may be related to the structure of the dyes and their interaction with micelles. However, it is beyond the scope of this work but serves for insight for future researches with complex solutions or molecular simulation, for example.

Effect of SDS Concentration. In this section, the dye concentration and solvent-to-water mass ratio were kept constant, and the effect of initial SDS concentration $(c_{SDS}/\text{mg} \cdot \text{L}^{-1})$ on liquid—liquid extraction of textile dyes was taken into account. The concentration of dye in water was 100.3 mg $\cdot \text{L}^{-1}$ for BY28 (data set 6), 100.3 mg $\cdot \text{L}^{-1}$ for BB41 (data set 7), and 309.5 mg $\cdot \text{L}^{-1}$ for BR46 (data set 8). Table 4 shows experimental results for data sets 6 to 8. Figures 7 and 8 show the partition coefficient and percentage of removal plotted against c_{SDS} , respectively.

It is verified that an increase in SDS concentration causes an increase in the partition coefficient for the three dyes. Considering the conditions of $c_{dye}^{feed} = 100.3 \text{ mg} \cdot \text{L}^{-1}$, BB41 presents values of *K* greater than 1500, while BY28 shows values between 120 and 500, approximately. Data for BR46 show *K* values between 75 and 150 but are treated separately because $c_{dye}^{feed} = 309.5 \text{ mg} \cdot \text{L}^{-1}$ was used rather than 100 mg $\cdot \text{L}^{-1}$.



Figure 8. SDS concentration effect on the percentage of dye removal for data sets 6 to 8. \blacksquare , BY28, $c_{dye}^{feed} = 100 \text{ mg} \cdot \text{L}^{-1}$; \blacklozenge , BB41, $c_{dye}^{feed} = 100 \text{ mg} \cdot \text{L}^{-1}$; \blacklozenge , BR46, $c_{dye}^{feed} = 310 \text{ mg} \cdot \text{L}^{-1}$.

The percentage of removal is also enhanced with an increase in SDS concentration for data sets 6 to 8. BB41 and BY28 present a similar profile, yielding a removal percentage > 98.0. BR46 shows similar results, but the profile is different from the other dyes, as explained above. The comprehension of these results for each dye involves further research on experimental and computational data, as cited in the section on the effect of initial dye concentration.

The pronounced increase of K with SDS concentration can be explained by an enhanced micelle formation due to higher SDS content in isopentanol, which increases the solubility of dyes in the organic phase during the mixing stage. This causes a great reduction of dye concentration in the aqueous phase, explaining the higher E values obtained.

CONCLUSIONS

The removal of three textile dyes, Basic Yellow 28, Basic Blue 41, and Basic Red 46, from water was performed by liquid—liquid extraction with reverse micelles, using sodium dodecylsulfate as surfactant and isopentanol as solvent. The partition coefficients and percentage of removal were obtained in nine different data sets.

For constant SDS concentration in isopentanol, it was verified that an increase in initial dye concentration in water, which simulates an increasing amount of dye contaminants in a real aqueous stream, causes a decrease in partition coefficient values for all dyes, independent of SDS concentration, which points out that the solute (dye) is easier to remove by extraction from diluted solutions. The percentage of removal decreases with the increasing of the initial dye concentration; this effect is more pronounced with BB41 with $c_{\text{SDS}} = 123.6 \text{ mg} \cdot \text{L}^{-1}$. Probably, a reduction in the capacity of organic phase to solubilize the dye and remove it from water occurs with higher feed dye concentrations.

In the study of influence of SDS concentration in dye removal, the values of K and E were quite augmented with the surfactant concentration and can be comprehended by an enhanced micelle formation due to higher SDS content in isopentanol. For the partition coefficients, BB41 presents greater values than BY28, but the removal percentage is similar for both dyes; BR46 presents the same profile, although with a different initial dye concentration. In these experiments, the percentage of removal of dye from aqueous phase achieved at least 98.0 in all cases. Isopentanol as a solvent and SDS as a surfactant are able to successfully remove these dyes from aqueous solutions. These systems were never studied before in literature and give insight for future researches in liquid—liquid equilibria in model and real systems of water + dye + surfactant + solvent.

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