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TECHNICAL NOTE

Separation of Some Dyes from Aqueous Solutions by Flotation

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ABSTRACT

Separation of the dyes brilliant green, neutral red, eriochrome black T, and eosin from aqueous solutions by flotation was studied using oleic acid surfactant. Nearly 100% of the investigated dyes could be floated under the optimum conditions. The effect of pH on the flotation efficiency was studied in particular. At pH ≤ 2, 7.5, ≤ 5, and ≤ 6, the maximum flotation was achieved for brilliant green, neutral red, eriochrome black T, and eosin, respectively. The effects of oleic acid and dye concentrations, some ions, and temperature on the floatability were examined. Moreover, the selective separation of some dyes was attempted.

INTRODUCTION

The foam separation of soluble and suspended matter in water has drawn increasing attention in connection with industrial wastewater (1, 2), sewage treatment (1, 2), the removal of dyes (1–8) and trace amounts of radioactive substances from solutions (1), and the concentration of rare elements from seawater (2, 9). The technique of ion flotation involves the removal of surface-inactive ions from aqueous solutions with the aid of bubbles and can be divided into several processes which differ in detail, such as foam flotation (on foam fractionation), precipitate flotation, and adsorbing-colloid flotation (1). Foam flotation consists of the flotation of ions in a solution by adsorption on ascending bubbles. In precipitate flotation, ions in the solution are precipitated by the addition of proper substances or by controlling the pH, and the precipitates formed are floated by adding a surfactant. Adsorbing-colloid flotation consists of the adsorption of ions from a solution on adsorbent particles like bentonite or a synthetic ion-exchanger; the particles are subsequently floated by the addition of a suitable surfactant.

Kobayashi et al. (8) studied in detail the removal of the dyes scarlet red, congo red, and crystal violet by the method of adsorbing-colloid flotation, using bentonite as an adsorbent and benzylhexadecyldimethylammonium chloride as a surfactant. The concentration of the dyes were determined colorimetrically. However, the measurements were hindered by the presence of bentonite particles which also reduced the light intensity by scattering.

Therefore, to compensate for this effect, our present work was undertaken to float the dyes brilliant green (BG), neutral red (NR), eriochrome black T (EBT), and eosin (ES) using oleic acid (HOL) as the surfactant without bentonite as an adsorbent. Moreover, the method does not use nitrogen as a source of bubbles, but obtains bubbles by simple manual shaking.

EXPERIMENTAL

Apparatus

The cell used for the flotation measurements was the same as has been described in previous papers (10–12). A Griffin Model 40 Colourimeter was used for dye determinations. The pH measurements were carried out with a Hanna Instruments 8519 digital pH meter.

Reagents

The oleic acid (HOL) stock solution (6.63×10^{-2} mol·L^{-1}) was prepared from oleic acid, food grade (sp. gr. 0.895) in kerosene. Aqueous solutions (1.0×10^{-4} mol·L^{-1}) of the dyes brilliant green (BG), $C_{27}H_{34}N_2O_4S$, neutral red (NR), $C_{15}H_{17}ClN_4$, eriochrome black T (EBT), $C_{20}H_{12}N_3NaO_7S$, and eosin (ES), $C_{20}H_6Br_4Na_2O_5$, were prepared by dissolving the calculated amount of the dye in 1 liter of double distilled water. All other reagents used were of analytical grade.

Procedure

The sample solution of flotation was prepared by introducing 1 mL of the stock solution of the dye and HOL into the flotation cell, adjusting the pH to the desired value with HCl or NaOH, and diluting the solution to 10 mL with distilled water. The flotation cell was shaken upside down

20 times by hand. After the cell had been kept standing for 10 minutes to complete the flotation, the underlying liquid was taken out for the determination of the dye concentration.

The light absorbances were measured for the dyes, and calibration curves were constructed at their maximal wavelengths (600 nm for BG and 520 nm for NR, EBT, and ES). The flotation efficiency of a dye is expressed by

$$F = [(C_i - C_f)/C_i] \times 100\%$$

where C_i and C_f are the initial and final concentrations of the dye, respectively.

To study the effect of temperature on flotation efficiency, the dye solution and the surfactant HOL were either heated or cooled to the same temperature. The dye solution and HOL were quickly poured at time zero into the flotation cell jacketed with 1-cm thick fiberglass insulation. The flotation procedure mentioned above was repeated.

Unless otherwise specified, the measurements were carried out at room temperature, about 25°C.

RESULTS AND DISCUSSION

Effect of HOL Concentration

Figure 1 shows the effect of surfactant concentration, HOL, on the floatability of a constant concentration (1.0×10^{-5} mol·L^{-1}) of the studied dyes, BG, NR, EBT, and ES, at pH 7. As may be seen, the flotation efficiency (*F%*) of the dyes BG, NR, and EBT increases with increasing HOL concentration. At higher concentrations of HOL, the flotation slightly decreases, which may be due to adhesion of the scum to the walls of the flotation cell and to the excessive foaming which were observed experimentally. With respect to ES, the flotation efficiency is not affected by increasing the HOL concentration. The experimental data show that the floatability of ES is about 2% at pH 7 (Figs. 1 and 2).

Effect of Dyes Concentrations

Figure 2 shows the flotation efficiency of the dyes BG, NR, EBT, and ES as a function of their concentrations using HOL (15.9×10^{-3} mol·L^{-1}) at pH 7. As may be seen, at concentration of dyes $\leq 2 \times 10^{-5}$ mol·L^{-1}, BG, NR, and EBT have a flotation efficiency of 100%. Above such concentrations the floatability of BG is more than that of NR and EBT. This may be attributed to the fact that both BG and NR have a positive quaternary ammonium group which combines with oleate ions obtained from

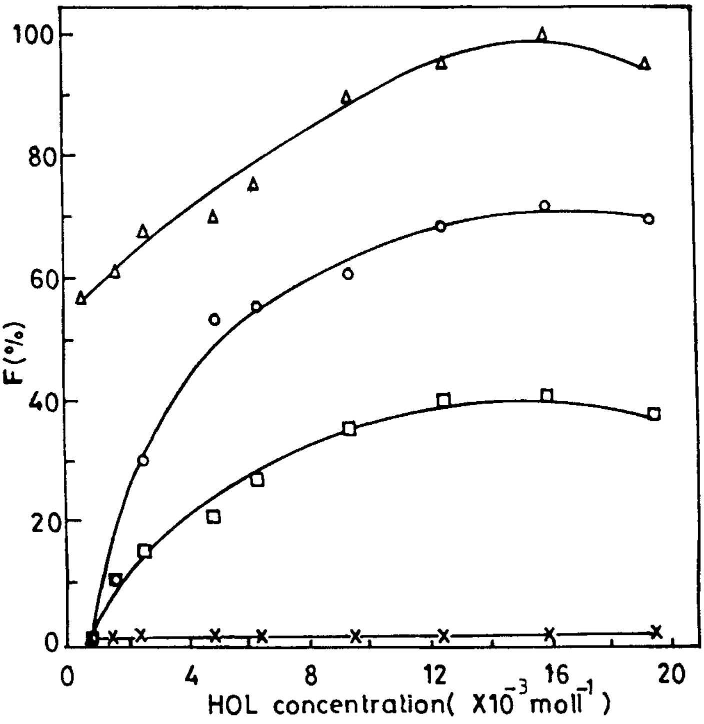

FIG. 1 Effect of HOL concentration on the floatability of 1 × 10^{-5} mol·L^{-1} of the dyes: (△) BG, (○) NR, (□) EBT, and (×) ES at pH 7.

the dissociation of HOL at pH ≥ 5.2 (13). EBT may combine by hydrogen bonding, through its OH functional groups, with oleate ions and be floated. ES, a disodium salt, may be present at pH ≥ 7 in the anion form uncapable of flotation with oleate ions.

Effect of pH

The effect of pH on the floatability of the dyes (1 × 10^{-5} mol·L^{-1}) with HOL is shown in Fig. 3. It can be seen that NR begins to float at pH 6 and reachs its maximum at pH 7.5. BG has a maximum flotation efficiency at pH ≤ 2, perhaps due to physical adsorption of HOL on the dye, and forming a hydrophobic associate which is floated by air bubbles. The flotation of BG decreases to zero at pH 5 and again increases to its maximum (90%) at pH 7.5. This may due to the hydrolysis of BG at pH 7 and higher, giving a positive dye which combines with oleate ions and

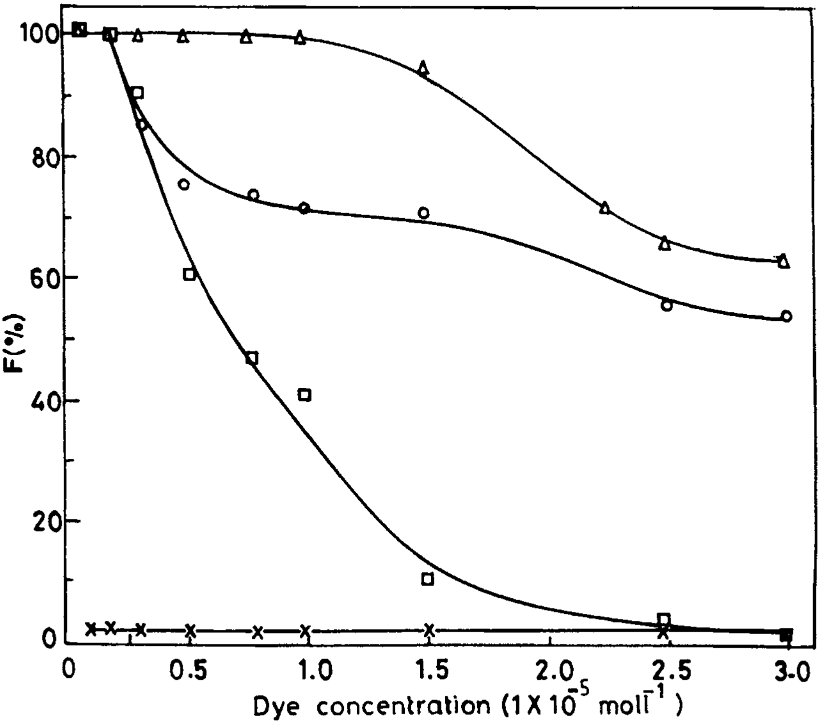

FIG. 2 Effect of dye concentration on their floatability at pH 7 and HOL, 15.9 × 10^{-3} mol·L^{-1}: (△) BG, (○) NR, (□) EBT, and (×) ES.

is floated. EBT has its maximum flotation at pH ≤ 5. This may be attributed to the combination of oleic acid (HOL) and EBT by hydrogen bonding through the OH functional groups of EBT. This was confirmed by the fact that vigorous shaking during flotation delayed the time of flotation. ES (a disodium salt) has its maximum flotation efficiency at pH ≤ 6. This may due to the fact that the disodium salt has an acid form (in acidic medium) which combines with HOL through hydrogen bonding and is floated.

Effect of Temperature

A change of temperature during flotation of up to 85°C has no effect on the floatability of the studied dyes, but it does decrease the time of flotation. This may be due to the fact that temperature promotes aggrega-

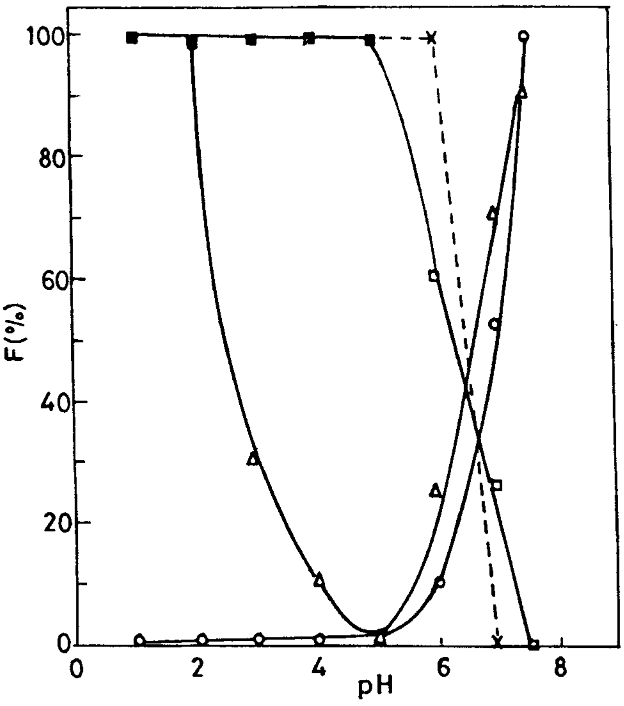

FIG. 3 Effect of pH on the floatability of the dyes (1×10^{-5} mol·L^{-1}) with HOL (6.63×10^{-3} mol·L^{-1}): (△) BG, (○) NR, (□) EBT, and (×) ES.

tion of the particles, partly due to an increase in the velocity gradient with temperature (14).

Effect of Some Foreign Ions

The addition of sodium and potassium cations to the flotation medium has no effect on the flotation efficiency of the investigated dyes at the optimum conditions. Moreover, sulfate, nitrate, phosphate, chloride, bromide, and iodide anions do not affect the floatability of the dyes.

Selective Separation of Dyes

The selective separation of dyes from each other was attempted by controlling the pH in the following combinations: 1) NR + BG; 2) NR + EBT; 3) NR + ES; and 4) NR + BG + ES. In the first three combinations, NR is separated selectively from BG, EBT, and ES by flotation at pH 2. In the last combination, NR is separated selectively from ES and BG by

TABLE 1
The Optimum Conditions of Dye Flotation

Dye	BG	NR	EBT	ES
Dye concentration $(\mathrm{mol \cdot L^{-1}})$	1×10^{-5}	1×10^{-5}	1×10^{-5}	1×10^{-5}
HOL concentration	6.63×10^{-3}	6.63×10^{-3}	6.63×10^{-3}	6.63×10^{-3}
Flotation (%)	100	100	100	100
pH	≤2	7.5	≤5	≤6

flotation at pH 2. By increasing the pH of the scum to 5 in the last combination, flotation results in complete separation of ES from BG.

The optimum conditions for flotation of the examined dyes is shown in Table 1. It is noteworthy that 6.63×10^{-3} M HOL is the suitable concentration for the separation of the studied dyes. Such a concentration is lower than the critical micelle concentration (cmc) of HOL, $\sim 10^{-2}$ M (15).

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