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Removal of anionic dye from aqueous solution using a cationic carrier

Govindaraju Muthuraman* and Mohamedjakkaria Mohamed Ibrahim

Abstract

Background: The transport of Cibacron Red FN-R (Sigma-Aldrich, MO, USA) anionic dye through a methylene chloride (Merck, India) bulk liquid membrane containing a tetrabutylammonium bromide (TBAB; Merck, India) as an excellent carrier was studied. The extracted dye simultaneously stripped into aqueous stripping solution.

Results: The basic fundamental parameters were determined. These include pH of the feed, nitric acid concentration in the strip solution, TBAB concentration in the membrane phase, initial dye concentration in the feed phase and rate of stirring. The transport efficiency of the dye increased with increasing carrier concentration from $6.06 \pm 0.1 \times 10^{-6}$ to $3.85 \pm 0.1 \times 10^{-4}$ M. The transport study was highly selective for anionic dye, and the efficiency of transport was not affected by the presence of electrolytes such as sodium chloride and sodium sulphates in feed solution.

Conclusions: The percentage of dye transported across the liquid membrane after 70 min was $95 \pm 0.1\%$. When the optimum parameters were used for real textile dyeing effluent for recovery of dyes, the results were found to be satisfactory.

Keywords: Transport, Anionic dye, Tetrabutylammonium bromide, Bulk liquid membrane, Recovery, Efficiency

Background

Among different pollutants of aquatic ecosystem, dyes are of a large and important group of chemicals. They are widely used in industries such as textile, paper, rubber, plastic, cosmetic etc., to colour the products. These dyes are invariably left as industrial waste and consequently discharged mostly to surface water resources. Dyes, even in low concentration, are visually detected and can affect the aquatic life and food web. These coloured compounds are not only aesthetically displeasing but also inhibiting sunlight into the stream, thus affecting the photosynthetic reaction [1]. The removal of colour synthetic organic dyestuff from waste effluents becomes environmentally important. It is rather difficult to treat dye effluents because of their synthetic origins and their mainly aromatic structures, which are biologically non-degradable [2]. Therefore, the decolorization of wastewaters containing these dyes prior to discharge is mandatory by environmental regulations in most countries [3].

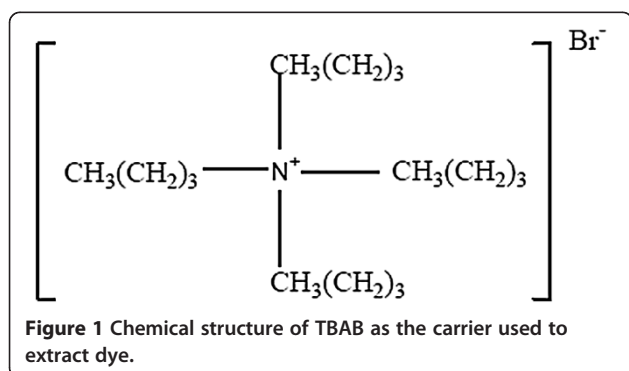
There are several methods for dye removal such as biological treatment [4-6] coagulation/flocculation [4,7], chemical oxidation and photocatalytic processes [8,9] ozone treatment [10], membrane processes [11-14], adsorption [15-20] and liquid-liquid extraction [21-25]. Among these, liquid membrane is an alternative method over conventional treatment methods for industrial wastewater. This method is low cost, simple, with high efficiency and energy saving in comparison to other processes. In the recent years, a remarkable increase of the application of liquid membranes in separation processes has been observed. These membranes include bulk liquid membrane (BLM), emulsion liquid membrane and supported liquid membrane [26,27]. Transport of textile anionic dyes using cationic carrier by bulk liquid membrane has been studied and found that tetrabutylammonium bromide (TBAB) was an excellent carrier for selective and efficient transport of dyes. At a stirring speed of 250 rpm, 97% dye has been transported from feed to strip within 70 min. The percentage recovery of the dye was not found to decrease in the presence of anions (chloride and sulphates) in the feed solution [28].

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Urutiaga et al. [29] investigated the effect of phenolic concentration on the partition coefficient in Cyanex 923 kerosene/water system (Cytex Industries, Inc., Woodland Park, USA) at six different concentrations. They reported that the equilibrium concentration of phenol had a strong effect on the partition coefficient for phenol between trialkylphosphine oxides (Cyanex 923) in kerosene and water. Ma et al. [30] reported the transport of amino acid through di-(2-ethylhexyl) phosphoric acid (D₂EHPA) as carrier in kerosene. The liquid membrane system exhibited excellent transport selectivity for amino acids containing a side chain of high lipophilicity constant, like L-phenylamine, L-tryptophan, L-leucine and L-isoleucine. As a target amino acid with bulky aromatic side chain of L-phenylamine, the influence of its concentration and the concentration of D₂EHPA on the interfacial tension and the effect of temperature on its transport through the BLM were investigated. The pseudo-first-order apparent rate constants of the interfacial transport of L-phenylalanine for the extraction reaction and stripping reaction were estimated.

A kinetics of L-isoleucine transport through a liquid membrane containing D₂EHPA in kerosene was also studied [31]. The influence of pH in the aqueous feed solution, D₂EHPA concentration in the organic phase, the stripping solution composition and H₂SO₄ concentration in the stripping solution were investigated, and the effects of stirring speed and temperature on the transport of L-isoleucine through a BLM were studied. The pseudo-first-order apparent rate constants (k_1 , k_{-1} , k_2) and the apparent activation energy values of the interfacial transport of extraction, back extraction and stripping have been determined.

In the present work, transport of Cibacron Red FN-R through a methylene chloride bulk liquid membrane containing a TBAB as a carrier (Figure 1) was studied. Different experimental conditions, such as the effect of TBAB concentration in the membrane, the effect of stripping agent, the effect of pH in feed phase, the rate of stirring and the effect of time were also investigated.



Methods

Liquid-liquid extraction

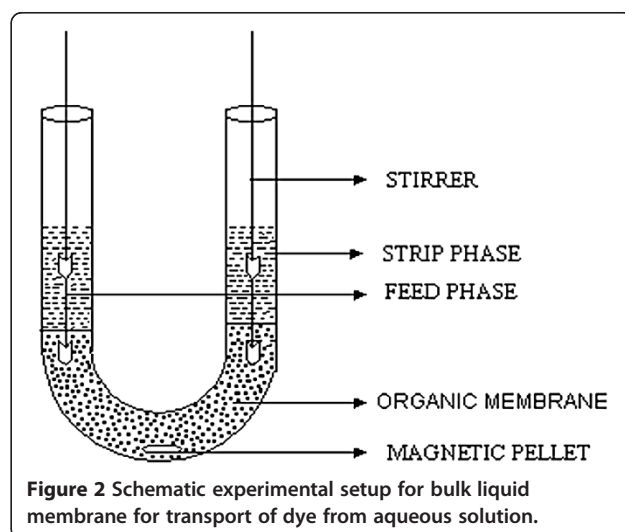
Solvent extraction experiments were undertaken at $27 \pm 0.5^\circ\text{C}$. Diluted H₂SO₄ and NaOH were used to adjust the pH of the aqueous solutions. A feed phase containing dye (100 mg L^{-1}) and organic phase were introduced in a separating funnel. The phases were mixed gently for known time and then left to separate. The raffinate was collected for the measurement of the remaining dyes in the solution. The distribution ratio and the percentage of extraction were calculated.

Bulk liquid membrane

The BLM apparatus (U-type) used for the study is shown in Figure 2. The inner dimension of the transport cell was 70 mm diameter \times 250 mm depth. Experiments were carried out at ambient temperature ($27 \pm 0.5^\circ\text{C}$), and the aqueous source (feed) phase (100 mL) dye and strip phase (100 mL) nitric acid were taken in the BLM apparatus. The dichloromethane solution (50 mL) contained TBAB. The organic layer was stirred by Teflon coated magnetic bar (2.0 cm) (DuPont, Haryana, India), and both aqueous layers (feed and strip) were stirred using the stirrer. In transport, experiment samples were taken out from the feed and strip phases, and the dye concentration was measured using spectrophotometer ($\lambda_{\text{max}} = 542.5 \text{ nm}$) with good reproducibility.

Kinetic procedure

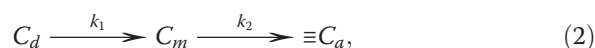
The variation of the dye concentration with time was directly determined on both donor (feed) (C_d in M) and acceptor (strip) (C_a in M) phases using spectrophotometer at regular intervals for 70 min. The corresponding change of the dye concentration in the membrane phase was determined from the material balance between the



phases. For practical reasons, dimensionless reduced concentration of dyes in the donor (R_d), membrane (R_m) and acceptor (R_a) were used [32]:

$$R_d = C_d/C_{do}, \quad R_m = C_m/C_{do}, \quad R_a = C_a/C_{do}, \quad (1)$$

where C_{do} is the initial dye concentration in the donor phase, while C_d , C_m and C_a represent the dye concentrations in donor, membrane and acceptor phases, respectively. From this expression, the material balance can be established as $R_d + R_m + R_a = 1$. When R_d , R_m and R_a values are inspected, the results suggest that the dye transport obeys the kinetics of the two consecutive irreversible first-order reactions according to the kinetics scheme [33]:



where k_1 and k_2 are pseudo first-order apparent rate constants of the extraction and the back extraction, respectively. The changes of carrier concentration and other factors affect the dye concentration, donor, membrane and acceptor phases. As a result of this effect, k_1 and k_2 values were changed. The kinetic scheme, Equation (2), for consecutive irreversible reactions can be described by the following rate equations [34]:

$$dR_d/dt = k_1 R_d \equiv J_d \quad (3)$$

$$dR/dt = -kR - kR \quad (4)$$

$$dR_a/dt = k_2 R_m \equiv J_a, \quad (5)$$

where J stands for flux. When $k_1 \neq k_2$, the integrating Equations (3) to (5) give:

$$R_d = \exp(-k_1 t) \quad (6)$$

$$R_m = k_1/k_2 - k_1 [\exp(-k_1 t) - \exp(-k_2 t)] \quad (7)$$

$$R_a = 1 - 1/k_2 - k_1 [k_2 \exp(-k_1 t) - k_1 \exp(-k_2 t)]. \quad (8)$$

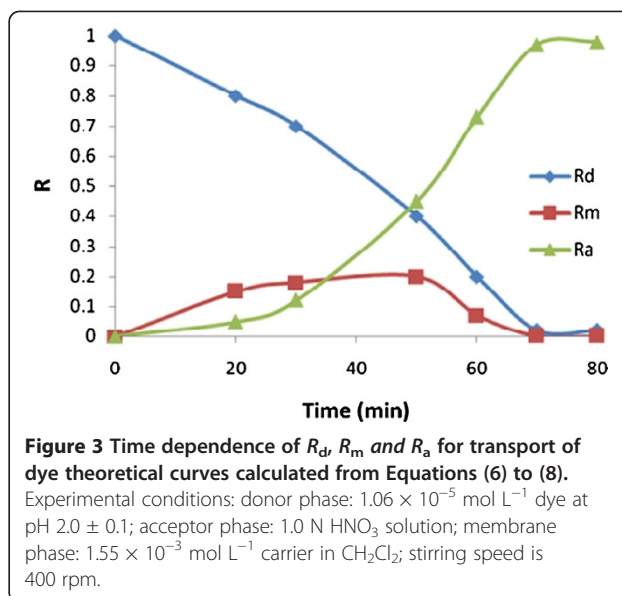
It can be seen that R_d versus t yields a decreasing mono-exponential curve, whereas the time variation of both R_m and R_a was bi-exponential. The actual numerical analysis was carried out by non-linear regression. The variation of R_d , R_m and R_a with time through the liquid membrane is shown in Figure 3.

The maximum value of R_m (when $dR_m/dt = 0$) and the corresponding t_{max} can be written as follows:

$$R_m^{max} = (k_1/k_2)^{-k_2/(k_1-k_2)} \quad (9)$$

$$t_{max} = \ln(k_1/k_2)/(1/k_1 - k_2) \quad (10)$$

By considering the first-order time differentiation of Equations (6) to (8) at $t = t_{max}$, one obtains the following equations:



$$[dR_d/dt]_{max} = k_1 (k_1/k_2)^{-k_1/(k_1-k_2)} \equiv J_d^{max} \quad (11)$$

$$[dR_m/dt]_{max} = 0 \quad (12)$$

$$[dR_a/dt] = k_2 (k_1/k_2)^{-k_2/(k_1-k_2)} \equiv J_a^{max}. \quad (13)$$

The system is at steady state, so the dye concentration in the membrane ($t = t_{max}$) does not vary with time (Equation (12)).

Results and discussion

Liquid-liquid extraction experiments

Effect of TBAB concentration in the organic phase and pH of the feed phase

The effect of TBAB concentration on the percentage extraction of the dye was investigated in the range of 6.06×10^{-6} to 3.10×10^{-3} mol L⁻¹ of TBAB concentration (Table 1). The concentration of the dye used in the part of the study was 100 mg L⁻¹. The extraction efficiency of the dye increases at lower pH, then decreases at higher pH. There was a significant increase in the percentage of extraction when the concentration of TBAB was between 4.85×10^{-5} and 1.55×10^{-3} mol L⁻¹. At 1.55×10^{-3} mol L⁻¹ TBAB concentration, the percentage of the extraction was 95.1%. Beyond this concentration, the percentage of the extraction increased very slowly. Sarkar and Dhadke [35] also found that increasing the carrier concentration had no considerable effect on the transport of the bismuth. Hence, the subsequent extraction studies were conducted using 1.55×10^{-3} mol L⁻¹ of extractant.

Table 1 Effect of carrier concentration and pH of cibacron FN-R

Sample number	TBAB concentration (mol L ⁻¹)	pH	Percentage of dye extraction
1	6.06 × 10 ⁻⁶	2.0	65.1
2	6.06 × 10 ⁻⁶	4.0	70.1
3	6.06 × 10 ⁻⁶	6.0	73.1
4	6.06 × 10 ⁻⁶	10.0	63.1
5	6.06 × 10 ⁻⁶	12.0	50.1
6	4.85 × 10 ⁻⁵	2.0	85.1
7	4.85 × 10 ⁻⁵	4.0	85.1
8	4.85 × 10 ⁻⁵	6.0	75.1
9	4.85 × 10 ⁻⁵	10.0	66.1
10	4.85 × 10 ⁻⁵	12.0	52.1
11	1.55 × 10 ⁻³	2.0	95.1
12	1.55 × 10 ⁻³	4.0	95.1
13	1.55 × 10 ⁻³	6.0	94.1
14	1.55 × 10 ⁻³	10.0	60.1
15	1.55 × 10 ⁻³	12.0	45.1
16	3.10 × 10 ⁻³	2.0	99.1
17	3.10 × 10 ⁻³	4.0	99.1
18	3.10 × 10 ⁻³	6.0	98.1
19	3.10 × 10 ⁻³	10.0	60.1
20	3.10 × 10 ⁻³	12.0	45.1

Effect of stripping solution

In any extraction process, it is very imperative to back extract the extracted dye from the organic phase and to allow recycling of the organic phase without loss of efficiency. Various inorganic acids such as HCl, HNO₃ and H₂SO₄ were used as stripping agents in this study. Nitric acid stripped the dye very well from the organic phase (Table 2) compared to the other two inorganic acids. Hence, 1N HNO₃ was found to be suitable for stripping the extracted dye. It was found that the maximum amount of dye (96.2%) was stripped within 10 min. Further increase in time did not improve the stripping efficiency.

Effect of stripping reagent concentration

The effect of HNO₃ concentration on the stripping efficiency was studied for the range of 0.1 to 1N, and the

Table 2 Effect of stripping agent concentration

Sample number	1N acid	Percentage of dye stripped		
		1.06 × 10 ⁻⁵ mol L ⁻¹	2.12 × 10 ⁻⁴ mol L ⁻¹	5.30 × 10 ⁻⁴ mol L ⁻¹
1	HCl	90.5	89.2	87.6
1	H ₂ SO ₄	92.1	92.0	90.2
3	HNO ₃	96.2	95.2	94.5

result of the percentage stripping is shown in Figure 4. The percentage of stripping was 96% with 1N HNO₃ and 88.97% with 0.6 N HNO₃. The result shows that the higher the concentration of HNO₃, the higher will be the percentage of stripping. This was due to the higher amount of H⁺ ion responsible in carrying the anionic dye complex from the organic phase to the aqueous phase. Thus, HNO₃ of 1N concentration was chosen for subsequent studies.

Bulk liquid membrane experiments

Effect of stirring speed

The transport experiments were performed at six different stirring speeds, 50, 100, 200, 300, 400 and 500 rpm. It indicates that the stirring speed affects the transport rates of dye through the liquid membrane. Figure 5 shows the flux values at different stirring speeds, and the flux increases almost linearly with the increase of stirring speed below 400 rpm. It also reveals that diffusion was the rate-limiting step in the feed-membrane transport of dye from the feed solution to the membrane phase. The flux increases at lower stirring speeds (50 to 300 rpm), and then is nearly constant at higher stirring speeds (400 to 500 rpm). Yang and Fane [36] also found similar effect at higher stirring speeds. A 'blank' experiment was performed in which the membrane contained no carrier. No detectable transport of dye through the liquid membrane was found, suggesting that the transport of dye through the liquid membrane was fulfilled by the carrier.

Effect of temperature

Figure 6 presents the percentage of the dye stripped from the feed phase to the acceptor/receiving phase (RP) as a function of temperature. It is obvious that the

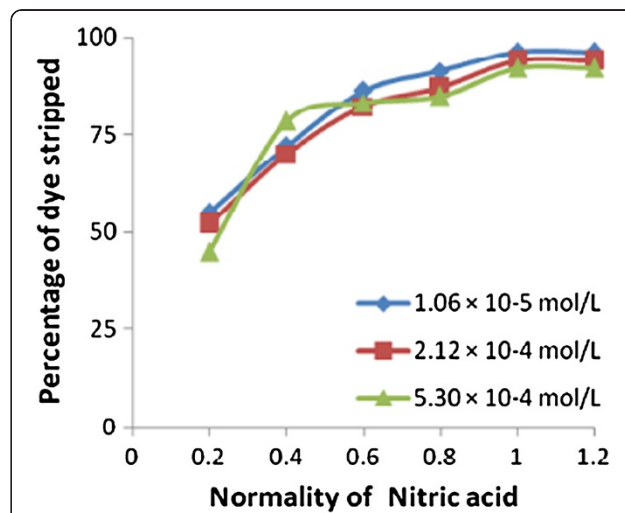


Figure 4 Effect of stripping reagent concentration. Experimental conditions: feed: different concentrations of dye solution; strip: different normality of HNO₃.

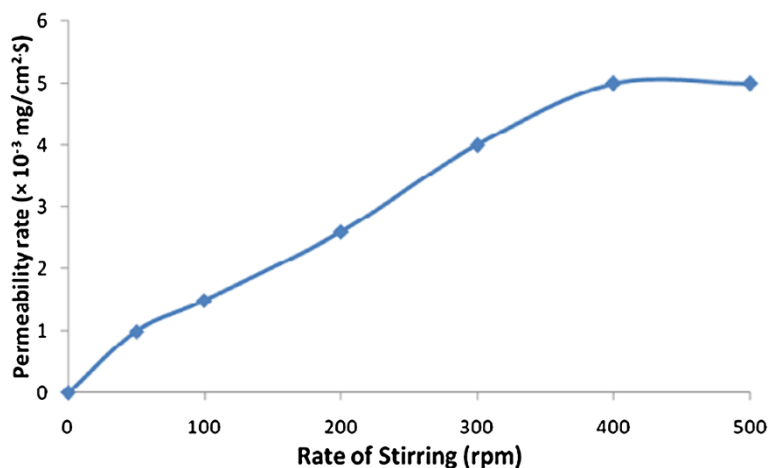


Figure 5 Effect of stirring. Experimental conditions: donor phase $1.06 \times 10^{-5} \text{ mol L}^{-1}$ dye at $\text{pH } 2.0 \pm 0.1$; acceptor phase: 1.0 N HNO_3 solution; membrane phase: $1.55 \times 10^{-3} \text{ mol L}^{-1}$ carrier in CH_2Cl_2 ; transport time is 70 min.

efficiency of the uptake from the source phase (feed) as well as the release of dye into the receiving (strip) solution enhances with temperature in the range of 10°C to 30°C . The transport was stripping-controlled, so the effects may not be pronounced especially at higher temperature. The results can be interpreted by considering several effects. Among them is the influence of the temperature on the complexation reaction with other ions by the extractant carrier at the donor/membrane interface and on the decomplexation of the dye at the second interface (i.e. membrane/acceptor phase). The effect of temperature on the diffusion of the transported species through liquid membrane, due to the temperature dependency of the membrane viscosity, is another effect of temperature on the transport process. However, the dependency of the distribution of the carrier on temperature should also be taken into account in describing the experimental results.

Effect of time

The time dependence of dye transport through the liquid membrane under the optimum experimental condition was studied. Figure 7 shows a rapid rise in dye concentration in the receiving phase as well as a sharp decrease in the dye concentration in the source phase (SP) during the first 70 min of transport. After 70 min, 95% of the dye was transported into the RP, 3% remained in SP, and about 2% of the dye remained in the organic phase. After 80 min, no significant transport of the dye was noticed. Hence, the time 70 min was recommended.

UV analysis

The UV absorption spectrum of Cibacron Red FN-R in aqueous and organic solutions exhibits an absorption maximum at 585 and 590 nm. The absorption maximum did not change in the stripped dye. This means that

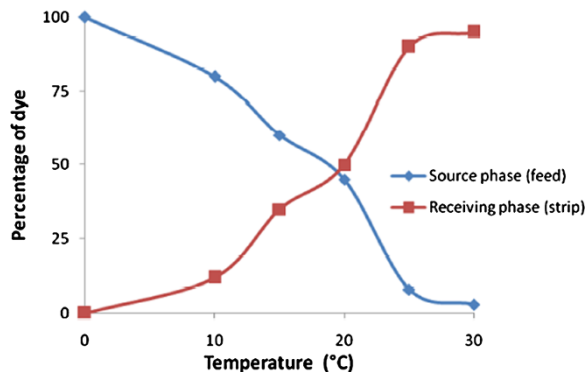


Figure 6 Effect of temperature. Experimental conditions: donor phase $1.06 \times 10^{-5} \text{ mol L}^{-1}$ dye at $\text{pH } 2.0 \pm 0.1$; acceptor phase: 1.0 N HNO_3 solution; membrane phase: $1.55 \times 10^{-3} \text{ mol L}^{-1}$ carrier in CH_2Cl_2 ; transport time is 70 min.

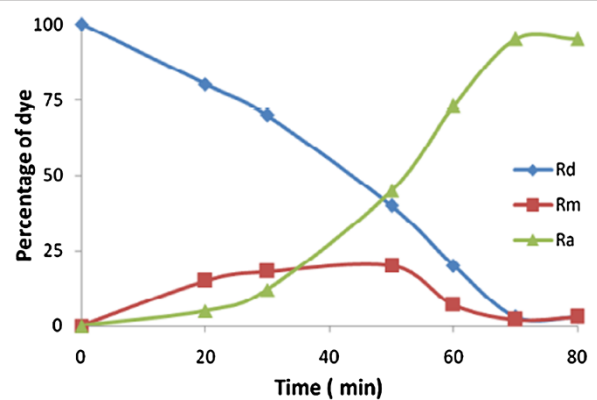


Figure 7 Effect of time. Experimental conditions: donor phase $1.06 \times 10^{-5} \text{ mol L}^{-1}$ dye at $\text{pH } 2.0 \pm 0.1$; acceptor phase 1.0 N HNO_3 solution; membrane phase $1.55 \times 10^{-3} \text{ mol L}^{-1}$ carrier in CH_2Cl_2 .

Table 3 Effect of kinetic parameters for transport of dye at different carrier concentration

Sample number	Concentration (mol L ⁻¹)	k ₁ (min ⁻¹)	k ₂ (min ⁻¹)	t _{max} (min)	R _m ^{max} (mL)	J _d ^{max} (min ⁻¹)	J _a ^{max} (min ⁻¹)
1	6.06 × 10 ⁻⁶	0.82 × 10 ³	0.81 × 10 ³	1,852.11	0.32	-0.31 × 10 ³	0.30 × 10 ³
2	4.85 × 10 ⁻⁵	1.64 × 10 ³	1.52 × 10 ³	502.40	0.35	-0.51 × 10 ³	0.56 × 10 ³
3	3.85 × 10 ⁻⁴	3.21 × 10 ³	2.91 × 10 ³	208.10	0.37	-1.1 × 10 ³	1.1 × 10 ³
4	1.55 × 10 ⁻³	3.7 × 10 ³	3.4 × 10 ³	157.54	0.38	-1.30 × 10 ³	1.20 × 10 ³

none of the functional groups were changed at the time of stripping. An enhancement in the absorbance with bathochromic spectral shift was observed in the organic layer. This might be due to the influence of methylene chloride in the organic layer. This spectral study shows that the recovered dye can be reused.

Kinetic parameters

The kinetic parameters for transport of dye are presented in Table 3. As it was seen, the transport rate, k₁, k₂ and R_m^{max} increase, and t_{max} decreases with the increase in carrier concentration. The k₂ values increase with the increase in carrier concentration in the organic phase from 6.06 × 10⁻⁶, 4.85 × 10⁻⁵ and 3.85 × 10⁻⁴ mol L⁻¹. From the results, 3.85 × 10⁻⁴ mol L⁻¹ carrier concentration was employed as the optimum carrier concentration.

Application of the developed BLM for textile wastewater

The wastewater from a local textile industry was selected for testing the applicability of the developed bulk liquid membrane system, which was alkaline in nature (pH 12.5 ± 0.1). Under optimised condition (feed phase 100 mL of 1.06 × 10⁻⁵ mol L⁻¹, 50 mL of 1.55 × 10⁻³ mol L⁻¹ carrier, acceptor phase 100 mL of 1.0 N HNO₃, equilibration time = 70 min), the textile dye waste was extracted, and the extracted dye was stripped into nitric acid. It was noticed that the extraction and stripping were not affected by the various types of salts present in the textile wastewater.

Experimental

Reagents and instruments

The following inorganic salts, acids and organic solvents were used in the experiments without further purification: NaCl (99.8%), NaOH (>96.0%), tetrabutylammonium bromide (99.5%), Cibacron Red (99.8%), HNO₃ (65%) and dichloromethane (99.5%). The chemicals were obtained from Merck Sharp & Dohme Corp. (Whitehouse Station, USA) and Thermo Fisher Scientific, Inc. (Waltham, USA) suppliers. A UV-visible spectrophotometer (Spekol 1200, Analytical Jena, Germany) was used to measure the absorbance of the dye and its concentration; the pH of the aqueous solution was measured by a pH metre (WTW Wissenschaftlich-Technische Werkstätten

GmbH, Weilheim, Germany). For agitation of solution, stirrer was used (IKD-KS 50, Germany). Deionized water was used for preparing all the aqueous solutions.

Conclusion

Cibacron Red FN-R was efficiently transported by means of a liquid membrane containing TBAB-CH₂Cl₂. The efficiency of the method depends on the important parameters such as the pH of the feed, dye concentration, carrier concentration, stirring speed and temperature. Flux increases almost linearly with the increase of stirring speed. After 70 min, there was no significant transport of dye noticed. In optimised conditions, the transport efficiency of Cibacron Red FN-R was after 70 min with 95.1 ± 1.1%. The proposed method was applied to real textile effluent and found that the transport efficiency after 70 min was 95%.

Competing interest

The authors declare that they have no competing interests.

Authors' contributions

GM carried out the bulk liquid membrane studies, and MMI participated in the liquid-liquid extraction study. All authors read and approved the final manuscript.

Authors' information

Authors are working in the Department of Chemistry, Presidency College, Chennai, India. Currently, they are doing research in the field of turbidity removal from drinking water using natural coagulants.

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