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### Journal of Hazardous Materials

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# Recovery of synthetic dye from simulated wastewater using emulsion liquid membrane process containing tri-dodecyl amine as a mobile carrier

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#### ARTICLE INFO

Article history:
Received 20 April 2011
Received in revised form
26 September 2011
Accepted 4 October 2011
Available online 8 October 2011

Keywords: Emulsion liquid membrane Tridodecylamine Reactive dye Electrical field Recovery

#### ABSTRACT

The extraction of Red 3BS reactive dye from aqueous solution was studied using emulsion liquid membrane (ELM). ELM is one of the processes that have very high potential in treating industrial wastewater consisting of dyes. In this research, Red 3BS reactive dye was extracted from simulated wastewater using tridodecylamine (TDA) as the carrier agent, salicyclic acid (SA) to protonate TDA, sodium chloride as the stripping agent, kerosene as the diluent and SPAN 80 as emulsifier. Experimental parameters investigated were salicyclic acid concentration, extraction time, SPAN 80 concentration, sodium chloride concentration, TDA concentration, agitation speed, homogenizer speed, emulsifying time and treat ratio. The results show almost 100% of Red 3BS was removed and stripped in the receiving phase at the optimum condition in this ELM system. High voltage coalesce was applied to break the emulsion hence, enables recovery of Red 3BS in the receiving phase.

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#### 1. Introduction

Increasing pollutants released to the environment have alerted both public and government since the discharged effluent could destroy the nature we live in. One of the major issues contributing to this crisis is water pollution. The growth in industry and the modification of manufacturing processes may have resulted in an increase in volume, composition and complexity of wastewater discharged to the environment [1]. The discharged wastewater can contain heavy metals, reactive dyes, organic pollutants and suspended solids. Reactive dye is a substance that contains a lot of hazardous chemical compounds such as benzidine structure, halogenated organics, toxic, carcinogenic and mutagenic organic compounds [2,3] exhibiting colours in water body and had become a very important environmental issue. Practically, it has been recorded that almost 125–1501 of water is used for every kilogram of textile product in the textile processing including bath residues from preparation, dyeing, washing, soaking, finishing and slashing [4].

The selection of separation method strongly focuses on the nature of the dye, operation treatment cost and composition of waste product. In some cases, the use of primary technique may not be sufficient to achieve complete decolourization, therefore dye removal strategies could consist of a combination of

different techniques [5]. The treatment for removal of dye has been developed since the last two decades with the progress in adsorption separation technique to remove basic violet 10 and acid red on sulphonated coal and Ganoderma Lucidum [6]. Meanwhile, a simulated reactive dye was experimented by Irena et al. [7] using nanofiltration membrane. Combination system to treat synthetic dve wastewater also has been proposed by Panswad and Luangdilok [8] using both anaerobic and aerobic simultaneously. Electrochemical oxidation of dyestuffs wastewater was studied by Jia and Yang [9] and Pelegrini et al. [10]. Removal and recovery of dye using ion exchange method was proposed by Mona and Yehia [11] whereas Snider and Porter [12] introduced ozonation treatment based on the composition of textile dyeing wastewater. Photocatalysis process was investigated [13] though Fenton method was also reported for degradation of textile dyes [14]. Coagulation-flocculation method was addressed as an alternative to the more conventional processes such as adsorption [15]. Liquid-liquid extraction (LLE) also has significant potential as an effective method for treatment of removal dyes [16-20]. However, membrane technology such as ELM, bulk liquid membrane (BLM) and supported liquid membrane (SLM) could be a greatly capable method for both removal and recovery of dye stuffs [21–23].

Among the techniques, ELM could be competitive when the targeted species is present at low concentrations in the aqueous solution. The novelty of ELM also is very promising and practicable for purification and recovery of targeted solute [24]. ELM separation process constitutes an emerging technology with a wide variety of applications, such as the removal, recovery, and purification of

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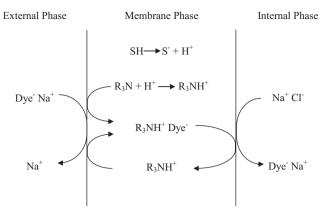


Fig. 1. Facilitated transport mechanism for reactive dyes transfer across a liquid membrane

many organic and inorganic compounds from dilute solutions of industrial interest [25]. However, stability of the membrane is the major problem associated to this technique. The challenges in a membrane system are due to swelling and breakage effect that could initiate an unstable emulsion resulting in a decrease in the degree of concentration of solute attained in the internal phase. This problem can be solved by choosing suitable parameters through an investigation on the effect of different factors on membrane stability.

The objective of this ELM separation technique is to separate solutes into receiving phase through a very thin layer of liquid membrane phase. The recovery of solute using ELM is very significant since the permeable barrier in emulsion is very selective, thus only targeted solute is allowed to pass through the barrier, whereas the rejected components stay as a raffinate in the solution and then discharged [26]. This method is proven to have several advantages over other physicochemical methods due to its ability to concentrate the pollutant up to 10–100 times [27].

To the best of our knowledge, no work has been conducted on the recovery of reactive dyes from aqueous phase by ELM. Therefore, in this study experimental works have been carried out to remove and further recover anionic reactive dyes, Remazol Red 3BS (R3BS), from simulated wastewater by ELM with the objective to determine the efficiency of the removal and recovery of the dye. The ELM technique was carried out with tridodecylamine (TDA) as a carrier agent with the presence of salicyclic acid (SA). The process parameters investigated include carrier concentration, stripping agent concentration, SA concentration, surfactant concentration, extraction time, agitation speed, homogenizer speed, emulsifying time and treat ratio. The focus is to optimize the process in order to give the optimum condition for the best recovery of reactive dyes from aqueous solution.

Mechanism of carrier facilitated transport extraction and stripping process of reactive dye by TDA is shown in Fig. 1. Mass transfer is assisted by a carrier present in the membrane phase as well as ion concentration gradient between the two sides of the membrane phase. This mechanism is for the recovery and enrichment of dye ions in receiving phase.

#### 2. Experimental

#### 2.1. Chemicals and reagents

Kerosene as diluents was obtained from Acros Organic, TDA as carrier from Merck Schuchardt OHG Germany, Salicyclic acid (SA) from Fisher Chemical, sodium chloride (NaCl) and sodium salicate (normally use in batik industry) from Merck Schuchardt OHG Germany and SPAN 80 from Fluka. R3BS reactive dye was obtained

from Nozi Batik Industry from Kuala Terengganu, Malaysia. The chemical structures of R3BS dye is shown in Table 1. The operation equipments are agitator IKA C-MAG, homogenizer Heidolph Silent Crusher M, UV/VIS Spectrophotometer model Jenway 6305 and high voltage coalescer.

#### 2.2. Experimental procedures

A primary emulsion was prepared by emulsifying an equal volume of aqueous solution (stripping phase) with formulated organic phase (diluents, surfactant and carrier) using homogenizer. Stability test of the emulsion was conducted on different concentration of surfactant, homogenizer speed, emulsifying time and agitation speed. The optimum ranges were applied in extraction study. Reactive dye solution was prepared by dissolving desired amount of dye concentration with distilled water. An equal volume of 5 ml portions of organic solution and an aqueous strip solution was stirred continuously at 12,000 rpm using motor driven homogenizer for 5 min to attain a stable primary emulsion system. The emulsion must be freshly prepared each time before the experiments. Then, the emulsion was dispersed into the agitated vessel of 50 ppm reactive dye solution (external phase) with appropriate treat ratio.

The double emulsion of water in oil in water (W/O/W) was mixed using magnetic stirrer at 250 rpm in a conical flask to allow the extraction and stripping process to occur. The three-phase dispersion was stirred for 10 min. After that, the samples were quickly introduced into a separation funnel and left for phase separation in half an hour. Two layers were formed in the separating funnel; upper layer is W/O emulsion, while bottom layer is aqueous treated phase. Then, the water in oil (W/O) emulsion was separated from the aqueous phase. For recovery purpose, this emulsion was demulsified using a high voltage coalescer to obtain the receiving phase/internal phase. The external and internal aqueous phase were analysed to determine the percentage of extraction and stripping recovery of reactive dye. The same procedures were repeated for different condition and formulations.

#### 2.3. Determination and calculation

The quantitative evaluation of reactive dyes was conducted using a UV–vis spectrophotometer at a suitable maximum wavelength. The concentration of dye ion is determined spectrophotometrically by absorbent of light through the dye solution. The percentage of removal efficiency, *R* is calculated using the following equation:

$$R = \frac{[\mathrm{Dye}]_{\mathrm{i}} - [\mathrm{Dye}]_{\mathrm{f}}}{[\mathrm{Dye}]_{\mathrm{i}}} \times 100\% \tag{1}$$

where  $[Dye]_i$  is the initial dye concentration (ppm) and  $[Dye]_f$  is the final dye concentration (ppm).

#### 3. Results and discussions

#### 3.1. Reactive dye removal and stripping efficiency

The experimental results for reactive dyes removal and stripping efficiency are shown in Table 2. The experiments were carried out in duplicates and the investigation and optimization was carried out one factor at a time and the best condition in each investigation was used in the conservative experiments that follow.

#### 3.1.1. Salicyclic acid concentration

Fig. 2 shows the effect of SA concentration on the percentage of extraction and stripping of reactive dye. The results show that 0.001 M SA is adequate to enhance the extraction process which is almost 100% of dye was extracted. This is due to the hydrogen

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