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Removal of Red 3BS Dye from Wastewater using Emulsion Liquid Membrane Process

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Abstract: Emulsion Liquid Membrane (ELM) is one of the process that has very high potential for industrial wastewater treatment of dyes. In this research, Red 3BS dye was extracted from simulated wastewater using Tridodecylamine as the carrier agent, sodium hydroxide as the stripping agent, kerosene as the diluent and span 80 was used as emulsifier. The important parameters affecting the Emulsion Liquid Membrane (ELM) extraction process including external phase concentration, treat ratio (emulsion/ external phase), concentration of carrier and agitation speed were investigated. The results show that the Red 3BS dye can be extracted almost 70% from simulated wastewater. The highest Red 3BS dye extraction was obtained by using 0.07 M Tridodecylamine, 5% (w/v) Span 80, 250 rpm agitation speed, 0.5 M thiourea in 1.0 M sodium hydroxide as the stripping agent, 1:3 treat ratio and kerosene as the diluent. As a conclusion, the Emulsion Liquid Membrane has a high potential in the extraction of dye from wastewater.

Key words: Waste treatment, emulsion liquid membrane, dye

INTRODUCTION

Textile production is one of the main industrial businesses in most parts of the world. It provides huge advantages to economic values as well as social aspect. The world production of textile is reported to reach 30 million tons annually (Ahmad *et al*., 2006) due to the well development of infrastructure. Batik making is one of the textile industries. In Malaysia, Batik is not just a fashion trend but it's also known as the art legacy. The development of this industry leads to the high demand of dye production. Therefore, the unsystematic handling of dyes will cause to water pollution.

Dye was undegradable substances and practically quite expensive in the international market even in the Malaysia due to its demand in various fields. Due to these problems, dye wastewaters have to be treated and recovered the dyes. Even though, previously, there are several methods are used to manage this problem such as adsorption on various sorbent (Demirbas, 2009), chemicals decomposition (oxidation) (Razaee *et al*., 2008), ion exchange method (Cotrino *et al*., 2007) and biological treatment (Koyuncu and Afsar, 1996). These methods were unprofitable due to high maintenance and only extract on the selective dye.

Usually, reactive dyes are employed in the textile and paper due to high stability, excellent colourfastness, bright colour and ease application industry. The wastewater that contains dye has to be treated due to its impact to the human and surrounding. Moreover, dyes waste is being listed by Environment Protection Agency (EPA) hazardous waste K 181 due to their carcinogenicity and toxicity (Harbhajan, 2006).

Red 3BS is an anionic reactive dye where sodium ion (Na^+) positive charge attached at dye compound contain benzidine and aromatic structure make it hazardous in batik industry effluents. Besides, it also has been proving a high potential carcinogenic amine (Harbhajan, 2006) which can bring harm to living thing if the wastewater not treated.

One of the alternative ways to remove and recover dyes from waste water was using liquid membrane emulsion (ELM). This method was the easy way for the removal chemicals (pollutants) by transported them into desired phase. ELM also reduces the cost of expensive extractant since low concentration was used. Beside, ELM has a very significant in recovery process and operation done in a shorter time (Masu *et al*., 2005). This method proved has several advantages over others physicochemical due its ability to concentrate the pollutant 10-100 times (Shen *et al*., 1996). The major problem associated with ELM is membrane stability. This problem can be solved by choosing the suitable parameters during the process.

This study will present the investigation on the effect of carrier concentration, agitation speed, external phase concentration and treat ratio on the extraction of dye using ELM process. Each parameter will be varies to get the optimum parameter for extraction of dye Red 3BS.

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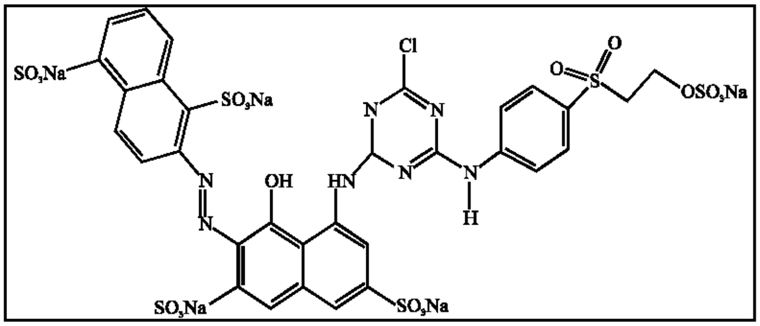

Fig. 1: Chemical structure of Red 3BS

MATERIALS AND METHODS

Materials: The dye used was Red 3BS that obtained from Batik Industry in Kuala Terengganu. The chemical structure of this dye was shown in the Fig. 1. All reagents were of analytical grade. Dye solution was prepared by dissolving requisite amount of the dye in distilled water. TDA (Tridodecylamine) and NaOH (sodium hydroxide) was used respectively as a carrier and stripping agent was both obtained from Merck KGaA. Kerosene from Acros Organics was used as the diluent. Meanwhile, Thiourea was used to increase the efficiency of the stripping process was supply from GCE laboratory chemical. While, Span 80 (sorbitan monooleate) product from Fluka Chemica Company was used as surfactant.

Methodology: The emulsion was prepared by emulsifying aqueous solutions (stripping phase) with formulated organic phase. Equal volume (5 mL) organic and aqueous solutions were stirred continuously at 12000 rpm using homogenizer Heidolph Silent Crusher M model for 5 min to obtain a white stable emulsion liquid membrane. The emulsion must have freshly prepared each time before the permeation experiment to avoid any destruction occurred.

The prepared emulsion was then dispersed into the agitated vessel having the external solution (simulated dye solution) with treat ratio of 1 to 3. The mixture was magnetically stirred at 250 rpm by Teflon coated magnetic bar for 10 min. Then, the samples are quickly poured into a separation funnel and left for phase separation. The aqueous phase was filtered in order to remove entrainment and this aqueous phase was analyzed by a spectrophotometer, model Cole Parmer 1100RS.

The result from the spectrophotometer will be converted from absorbance value to find the final concentration of the solution by using standard curve for Red 3BS. Therefore the percentage of extraction can be calculated using the equation below:

$$\text{Extraction (\%)} = \frac{C_0 - C_i}{C_0} \times 100\% \quad (1)$$

where, C_0 is initial concentration of dye and C_i is concentration at time t.

RESULTS AND DISCUSSION

Effect of carrier concentration: The effect of carrier concentration is shown in Fig. 2. The result shows that the extraction increase as the concentration of TDA increases. Meanwhile the emulsion breakages are 10% for all concentration range.

Increasing the concentration of the TDA (Tridodecylamine) from 0.01 M to 0.07 M, the extraction percentage of Red 3bs increased up to 70%. At this condition, high carrier will be found in the membrane and then increase the viscosity of membrane phase, which lead to the small globules of emulsion. Small globule results in the larger surface area. Therefore, mass transfers between external phases to internal phase also increase (Das *et al*., 2008).

Meanwhile further increase the carrier concentration (more than 0.07 M), the extraction percentage was decreased. It could be observed that increasing the amount of carrier has two effects: the viscosity of membrane phase, which limits the extraction rate, decreases by increasing the carrier concentration and hence the carrier acts as thinner for the membrane phase and at the same time, increasing the carrier concentration over a certain limit decreases the stability of the emulsion (Othman *et al*., 2006).

Effect of agitation speed: The effect of agitation speed on the extraction of Red 3BS dye from the simulated wastewater is shown in Fig. 3. The agitation speed was varies from 250 to 450 rpm. It is evident from the figure that agitation at 250 rpm has the highest extraction percentage. When the agitation speed was lower or higher

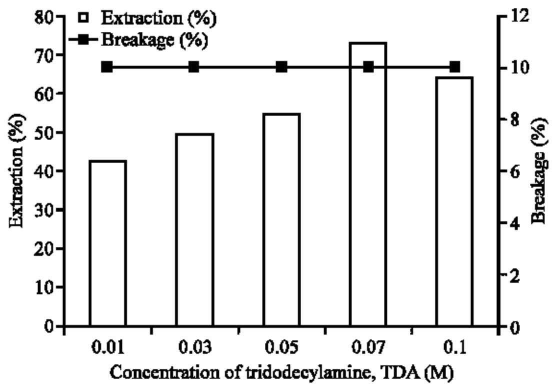

Fig. 2: Percentage of extraction and breakage at various Tridodecylamine (TDA) concentrations. (Experimental conditions: Span 80: 5%, NaOH: 1.0 M, Thiourea: 0.5 M, agitation speed: 250 rpm, contact time: 10 min, Emulsifying speed: 12000 rpm, Initial concentration of dye: 50 ppm, TR: 1:3)

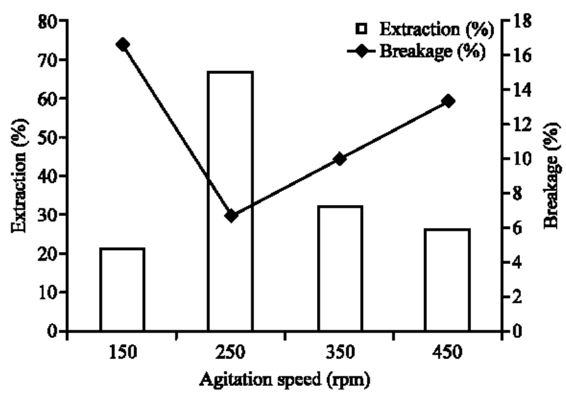

Fig. 3: Percentage of extraction and breakage at the several of agitation speed. (Experiment conditions: TDA: 0.07 M, Span 80: 5%, NaOH: 1 M, Thiourea: 0.5 M, contact time: 10 min, Emulsifying speed: 12000 rpm, Initial concentration of dye: 50 ppm, TR: 1:3)

than 250 rpm the result shows the extraction percentages were low. Meanwhile the emulsion breakages are high at these conditions.

Basically, in the separation process, with the increase of agitation speed, the diameter of each emulsion droplet becomes smaller due to more intense shear. Thus, the overall mass transfer surface area of the emulsion droplets increases (Othman *et al.*, 2006). Therefore, increasing of the speed to 250 rpm, the size of the emulsion will dispersed at surface of the external phase. Then, emulsion globules deceased and the interfacial area available for the

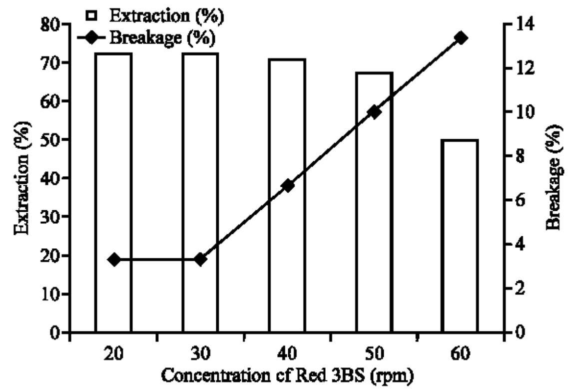

Fig. 4: Percentage of extraction and breakage at the various initial concentrations of Red 3BS. (Experiment condition: TDA: 0.07 M, Span 80: 5%, NaOH: 1 M, Thiourea: 0.5 M, contact time: 10 min, Agitation speed: 250 rpm, Emulsifying speed: 12000 rpm, Initial concentration of dye: 50 ppm, TR: 1:3)

mass transfer increased. This is due to the shears force which acts on the emulsion globules increases and this make the globule become smaller (Djenouhat *et al.*, 2008).

Increased further the agitation speed to 450 rpm resulted on decreasing in the percentage of extraction. At this stage, some droplets are getting broken because of high shear force after reaching larger size. At the same time the swelling also increasing. There exists a trade-off between these two effects. The swollen droplets can break on their own or due to the induced shear force (Masu *et al.*, 2005). On the other hand, the higher rate of shear and subsequent higher rate of breakage of globules at the higher agitation speed values, the higher rate of breakage allows more leakage of the solute into the feed phase from the internal phase (Kaghazchi *et al.*, 2003).

For lower agitation speed, the extraction efficiency low because of the formation of the larger emulsion globules involving a decrease of the area of mass transfer. On the other hand, the break up emulsion is more pronounced and leakage of the internal phase occurs. This can be explained by the increasing of osmotic swelling of the membrane (Djenouhat *et al.*, 2008).

Effect of external phase concentration: The effect of initial concentration of external phase on the extraction of Red 3BS dye from the simulated wastewater is shown in Fig. 4. The external phase concentration was varies from 20 ppm to 60 ppm. The result shows that the extraction percentage was decrease due to the increasing in concentration of external phase. Meanwhile the emulsion breakages are increase for the increasing of external phase concentration.

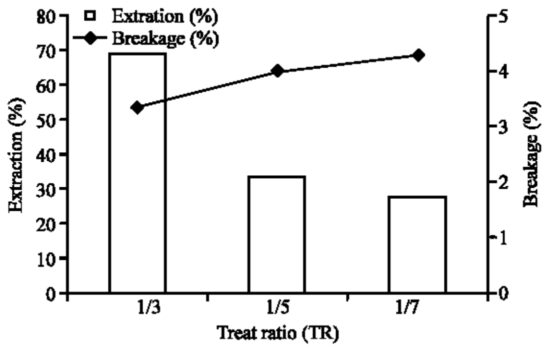

Fig. 5: Percentage of extraction and breakage at the different of treat ratio (TR). (Experiment conditions: TDA: 0.07 M, Span 80: 5%, NaOH: 1 M, Thiourea: 0.5 M, contact time: 10 min, Agitation speed: 250 rpm, Emulsifying speed: 12000 rpm, Initial concentration of dye: 50 ppm)

The removal percentage was decrease with the increasing of the external phase concentration. This is due to the saturation of the internal droplet in the peripheral region of the emulsion is attained more rapidly for high concentration in the external phase (Venkatesan and Meera, 2009). Therefore, when the solute concentration high, the complexes dye extractant will diffuse through the membrane phase to the more inner region of the globules to release dye in the internal phase (Djenouhat *et al*., 2008). The figure also shows when increase the external phase concentration will increase the swelling effect (Djenouhat *et al*., 2008). The phenomenon of swelling take place when the water transport from external phase to the internal phase is mainly driven by the difference in osmotic pressure between the external and the internal phases (Wan and Zhang, 2002).

Effect of treat ratio: The effect of treat ratio on the extraction of Red 3BS dye from the simulated wastewater is shown in Fig. 5. The treat ratio was varies from 1:3 to 1:7. The result shows that the extraction increase as the treat ratio increases. Meanwhile the emulsion breakages are increase with the decreasing of treat ratio. The treat ratio is defined as the ratio of emulsion phase volume to the aqueous feed phase volume. It plays an important role in determining the effectiveness of the ELM. The treat ratio was varied by changing the amount of feed phase and keeping the volume of the emulsion constant.

Figure 5 also shows that with an increase in the treat ratio, the volume of emulsion as a whole increased. Therefore, the surface area for mass transfer increased owing to the formation of a larger number of emulsion globules. As a result, a higher degree of extraction was obtained (Masu *et al*., 2005). Beside, the swelling phenomenon become notable, fast and accompanied by an embrittlement that will lead to more significant coalescence of the internal droplets (Djenouhat *et al*., 2008). This behaviour involves an increase of the emulsion breakage. Beside, at lower treat ratio lead to the low of percentage extraction. It is due to the amount of droplet that did not contain enough internal reagents to react with a solute transported through the liquid membrane. As the result, extraction of dye Red 3BS accumulated in the membrane phase and causes no carrier diffuse back to react with dye in external phase. Furthermore, the influence of the volume ratio on the stability was not significant, because the breakage decrease very slightly with an increasing of treat ratio (Masu *et al*., 2005).

CONCLUSION

It was demonstrated that the ELM technique was very promising in extracting Red 3BS dye from simulated wastewater. In order to achieve the objective several parameters has been studied such as concentration of carrier, agitation speed, initial external phase concentration (sources phase) and treat ratio. It was found that TDA and Sodium hydroxide provide very attractive results as carrier and stripping agent to Red 3BS dye. On the other hand, kerosene and Span 80 was chosen as an organic media for liquid membrane and surfactant respectively. Therefore, the most suitable Tridocecylamine concentration (carrier agent) is 0.07 M in kerosene, 250 rpm agitation speed, 50 ppm external phase concentration and treat ratio 1:3 under others condition which is surfactant 5 % w/v, concentration of stripping agent 1 M, emulsifying speed and time 12000 rpm and 5 min, respectively which provided high extraction percentage of Red 3BS from simulated wastewater using Emulsion Liquid Membrane.

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