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# **Red 3BS dye extraction in liquid surfactant membrane using** continuous extractive reactor process

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Abstract. Nowadays, the use of liquid membrane process in treating dye waste solution is more preferable compared to the batch mode as it provides higher recovery performance and huge treated volume. The aim of this paper is to determine the extraction efficiency of using continuous liquid surfactant membrane (CLSM) process for extraction of reactive dye Red 3BS in order to ensure that LSM process more applicable and commercialize in industries. Liquid membrane process comprises of three liquid phases which are external (dye aqueous solution), organic liquid membrane and internal aqueous phase was developed. Liquid membrane and internal aqueous phase were emulsified and dispersed into the wastewater solution (feed/dye aqueous phase) to be treated. The organic liquid membranes contain Aliquat 336, D2EHPA, Sorbitan Monooleote (Span 80) and kerosene as an extractant, synergist extractant surfactant and diluents, were used respectively. The sodium hydroxide (NaOH) was used as stripping agent in internal aqueous phase for the recovery process. The main factors studied for continuous dye extraction process were agitation speed, retention lime and treatment ratio. The system shows stable performance within 15 min duration of continuous extraction time along the process. The results showed that almost 100 % of Reactive Red 3BS ions were successfully extracted at the optimum conditions of 250 rpm agitation speed, 1: 7 of treatment ratio and 5 min of retention time. Therefore, as a conclusion, CLSM can be proposed as a very promising method for extraction of Red 3BS.

#### **1. Introduction**

Nowadays the textile industries have gained great concern of using dyes in production of consumer products. Severe pollution problems worldwide created by textile industries are dyecontaining waste solutions. Annual production of textile industries estimated was 10,000 various types of textile dyes with 7.105 metric tonnes are worldwide available commercially [1]. Toxicity of

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reactive dyes as a main substance in textile industries that comprise a lot of hazardous chemical compounds for instance carcinogenic and mutagenic organic compounds, toxic, halogenated organics and benzidine structure exhibiting colours in water body and had become a very vital environmental problem. Basically, for every kilogram of textile product in the textile processing including bath residues from preparation, dyeing, washing, soaking, finishing and slashing, it has been chronicled that almost 125 to 150 litre of water is utilized [2]. The environmental concern with dye is blockage in sunlight penetration into water and seriously influence on the ecosystem. Besides, high exposure time of organisms under dyes pollution can cause acute and chronic effects. It is proved that are generally resistant to oxidizers, temperature and light and dyes contained complex aromatic molecular structure [3]. Currently, various techniques to treatment the waste solution have been done in order to resolve this serious problem. The examples of the techniques are photodegradation [4], adsorption [5], chemical coagulation/flocculation technology [6] and oxidation process [7]. However, these methods were unprofitable due to too expensive to be used wisely and only extract on selective dye only [8].

Over the last few decades, liquid surfactant membranes (LSM) has become an attractive alternative for the treatment of azo-dyes from industrial effluents and has been known as one of the effective extraction process [9]. This is due to its outstanding features for example low energy consumption, high recovery, high selectivity, non-equilibrium mass transfer, and extraction and recovery occur at one simultaneous unit operation [10][11]. Nevertheless, utmost of the traditional liquid membranes are functioning in batch mode operation that needs a larger tank volume with longer time of operation than that parallel continuous process, and as a consequence this methods become fewer favourable to large- scale systems. Henceforth, continuous liquid membranes have to be developed to overwhelm the limits of the batch liquid membrane.

Several examples of continuous liquid membrane established in current centuries are the continuous LSM worked with different equipment setups. For example, Lee and Kim, (2011) compared the performance of a continuous LSM with a parallel batch process and concluded that the efficiencies of both systems were compatible [14]. Bhowal *et al.*, (2012) modified the traditional spray column by introducing a rotating spray column in which emulsions droplets were dispersed under the action of centrifugal force in lieu of terrestrial gravity [12]. They used the rotating spray column to separate Cr (VI) ions from aqueous solutions by LSM and discovered that the transport rate of Cr (VI) ions in the rotating spray column. Besides spray columns. mechanically-agitated columns have also been used in LSM processes. For example, Oldshue-Rushton column was applied in the separation of penicillin G [13] and acetic acid [14] from aqueous solutions by LSM and rotating disc contactor was used in the separation of different metal ions.

Previously, the extraction of Red 3BS in batch LSM process has been successfully extracted [9][15]. However CLSM has not been studied in the industries owing to certain industrial predicaments place in the batch liquid membrane owing to swelling of liquid surfactant membrane (LSM) [16]. In the present work, the studied was carried out further for the continuous LSM to determine the extraction efficiency and in order to ensure that LSM process more applicable and commercialize in industries. The influence of treatment ratio, agitation speed and retention time towards Red 3BS extraction behaviour were investigated, and the enrichment is discussed.

# 2. Methodology

# 2.1 Materials

The carrier, D2EHPA, Di-(2 thylhexyl) phosphoric acid and Aliquat 336, Tricaprylylmethylammonium chloride was procured from Sigma Aldrich. Salicyclic acid (SA)

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as a co-carrier was procured from Merch. The surfactant, Span 80 was supplied by Fluka Chemica. Sodium hydroxide (NaOH) was acquired from Merck and was used as stripping agent. On the other hand, kerosene as diluents was obtained from Sigma Aldrich. 1-octanol was used as modifier and supplied by Merck. Remazol Red 3BS in a powdered form as the targeted solute was supplied by one of the textile company in Terengganu. Simulated waste solution of Red 3BS was set by dissolving certain quantity of the dye in purified water. Table 1 present the maximum wavelength, molecular size, molecular weight and chemical structure of Red 3BS. All reagents used were of guaranteed reagent grade.

Anionic dye	Chemical structure	Maximum wavelength
		(λmax)
Red 3BS (Molecular Weight = 1136.32 g/mol)	NaO <sub>3</sub> S SO <sub>3</sub> Na H N N N H NaO <sub>3</sub> S SO <sub>3</sub> Na H N N N H NaO <sub>3</sub> S SO <sub>3</sub> Na SO <sub>3</sub> Na NaO <sub>3</sub> SOH <sub>2</sub> CH <sub>2</sub> CO <sub>2</sub> S	511

Table 1	<b>Properties</b>	of Remazol	Red 3BS	dye
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Continuous stirred tank reactors (CSTR) was used in this study. The schematic diagram of the continuous-flow mixer settler apparatus, single-stage, used in this work was presents in Figure 1. The tank dimension is about 12 cm in diameter and 15 cm height. Maximum capacity for the extraction vesel is 1.5 L with 4 baffled. There are two peristaltic pumps (Cole-Parmer, Masterflex Models) equipped with quick-load heads used to pump the emulsions and feed solutions through two inlet lines mounted on the upper wall of the vessel. The pump speed variation was control within 1 % using digital drives. Each time before running the experiment, the pumps were calibrated. Along the experiment, an outlet port mounted at the extractor bottom was controlled for the liquid level to be constant at level of 1000 mL. In the meantime, an outlet port mounted on the separator bottom wall was used to take the samples.



Figure 1. Schematic diagram of continuous LSM extraction process [17]

<sup>2.2</sup> Preparation of Water in Oil (W/O) Emulsions

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The primary W/O emulsions was prepared by mixing equal volume (50 mL) of stripping liquid solution (NaOH) with organic membrane solution (kerosene with carrier, 1-octanol and Span 80) at 12000 rpm Homogenizer (Heidolph Silent Crusher-M) for 3 minutes and the milky white stable emulsions was attained.

### 2.3 Red 3BS extraction in CLSM

A continuously supplied of primary emulsions into the emulsions vessel with series of 100 mL W/O emulsions of optimized condition [15] was prepared using 120 mL beaker. Before each step of experiment, the emulsions need to be freshly prepared. After that, the prepared emulsions and 50 ppm reactive dye solution (dye aqueous phase) were mixing continuously in the extractor using IICA RW 20 Digital Dual Range Mixers digital mixer system at agitation speed of 350 rpm for 5 minutes retention time. To control the flow rate of feed phase and emulsions solution, pump controller 1 and pump controller 2 were adjusted respectively into the extraction vessel to maintain the treatment ratio.

Every five minutes of extraction process, the sample was taken out along the process. Sample taken from the extraction process undergoes phase separation using separating funnel within 10 minutes. The aqueous phase is the treated dye solution. This aqueous phase need to be filtered out and analyzed using UV-spectrophotometer Jenway model to determine the extraction efficiency of dye. Steady state was indicated as the dye concentrations did not change over a period of two or three samples. After achieving the steady state concentration, the pumps and mixer were stopped to separate the emulsions and the aqueous phase. The separation of emulsions was conducted in a separating funnel within 15 minutes, where the three-phase dispersion was settled into the emulsions and the dye aqueous phase. The treated dye solution (dye aqueous phase) at the bottom of the separating funnel was taken for concentration measurement. Then, the internal of the emulsion liquid membrane was separated to recover the dye ions. Lastly, the emulsions was undergo demulsification using high voltage electrostatic coalesce to recover dye in the stripping phase and determine its enrichment. The equal techniques were recurrent for others variable ranges as shown in Table 2. The variables were tested by method one-factor-at-a-time (OFAT).

Table 2. Variable ranges in CLSIVI process		
Variable	Range	
Agitation speed (rpm)	150,250,350,450	
Retention time (min)	3, 5, 7.9	
Treatment ratio	1: 3, 1: 5, 1: 7, 1: 9	

Table 2. Variable ranges in CLSM process

#### 2.4 Calculation and Determination

The calculation for recovery or enrichment, extraction or removal and breakage percentage for Red 3bs ions during the SLM process was determined by Equation 1, 2 and 3 respectively:

$$Enrichment = \frac{Cinternal}{Ci}$$
(1)

Extraction (%) 
$$= \frac{Co-Ci}{Co} X \, 100$$
 (2)

Breakage (%) = 
$$\frac{Vf - Vi}{Vi} X \ 100$$
 (3)

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Where initial concentration of dye represent as Co and concentration at time t represent as  $C_t$  whereas after the demulsification process, the final dye ion concentrations in the stripping phase represents as  $C_{internal}$ , the initial volume of emulsions before extraction *represent as*  $V_i$  and the final volume of emulsions after extraction represent as  $V_f$ .

# 3. Result and Discussions

#### 3.1 Transport mechanisms for Red 3BS dye in CLSM process

Figure 2 shows the transport mechanism of the extraction and recovery for Red 3BS dye by CLSM techniques where it is referred to the liquid-liquid extraction process.



 $Dye-NR_4(HR)_4$  complex

Figure 2. Transport mechanism for dye ion transfer across liquid membrane

The selected extractant in this mechanism, D2EHPA and Aliquat 336 reacts chemically with the dye ion in diluent and form complex at the membrane-external interface as illustrated in Equation 4:

$$Dye_{(aq)} + 2(RH)_{2(org)} + R_4 N^+ Cl^-_{(org)} \iff Dye - R_4 N(HR)_{4(org)} + HCl$$
(4)

Where, the D2EHPA and Aliquat 336 in the organic membrane phase represent as  $(RH)_2$  and  $R_4N^+C\Gamma$ , respectively.

Then, the dye- $R_4N(HR)_4$  complexes was transported by the synergist carrier through the membrane phase towards internal-membrane interphase and stripping process will take place. The dye complex reacted with the stripping agent (NaOH) at the membrane-internal interphase to release dye ion into the internal aqueous phase and concentrated as exhibited in Equation 5. The regenerated extractants diffuse to the feed - membranes interphase through the membrane and the transport process is repeated to carry other ions and complete the extraction and stripping process:

$$Dye-R_4N(HR)_{4(org)} + 2H^+_{(org)} + 2Na^+OH^-(aq)$$

$$\longleftrightarrow Na_2^+Dye^-_{(aq)} + NR_4(HR)_{4(org)} + 2H_2O$$
(5)

Where, NaOH is sodium hydroxide in aqueous phase.

# 3.2 Effect of agitation speed

Figure 3 depicted influences of agitator speeds toward removal performance of Red 3BS are studied in the range of 150 to 450 rpm. The result displays that at 1 Min extraction time almost 100 % of dyes were extracted at 350 and 450 rpm agitation speed while around 80 % were observed for 150 & 250 rpm. In short extraction time, high agitation speed (350 rpm & 450)

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rpm) can create excessive shear force to deform emulsions globules into smaller size, which is conducive to diffusion of dye into emulsions globule. Consequently, by increasing the speed of agitation, the mass transfer area increase and the rate of mass transfer is also increased. This result confirmed with finding of Othman et al., (2011) which revealed the some trend [9]. Further increase the extraction time to 15 min, the extraction increase and become plateau indicating the stability of CLSM process within the period of time. This showed that CLSM process is stable and seemly suit for being used over the wide range of agitator speed. Since the process is stable and seemly suit for being used over the wide range of agitator speed, the stability along the extraction time was taken into account in chasing the most favorable agitation speed. The result shows that at 250 rpm and 450 rpm, the extraction percentage gradually increase within 15 min of the extraction time. However, lower agitation speed was chosen. This is due to increasing the speed of agitator will rise the number of smaller globules, however, by increasing the agitation speed to exceed the higher limit which contributes to the energy causes emulsions rupture or breakage [18). This principle also has been confirmed by the research study of Lee and Chan, (1990) that showed as the agitation speed exceed 400 rpm, the concentration of dye aqueous phase falls to minimum and then bounces back and increases [19]. This phenomenon concluded that there are two competing processes involves in the transport of the dye, which are leakage of the internal solution due to breaking of some emulsions globules and the diffusion of the solute through the emulsions membranes into the internal aqueous phase. Therefore, high agitation speed is not preferable. Based on the result obtained, the best value of agitation speed is found to be 250 rpm because of high extraction efficiency (99%) and low energy consumption which is more economically desirable.



**Figure 3**. The influence of agitator speed on dye removal performance ( Data conditions: [Aliquot] = 0.1 M; [D2EHPA] = 0.08 M; [SA] = 0.1 M; [NaOH] = 0.1 M; [Span80] = 3 % w/v; [1-octanol] = 5 % w/v; TR = 1: 3; retention time =  $5 \min$  and T =  $\pm 26 \degree$ C )

# 3.3 Effect of retention time

Retention time is the time period for which the emulsions phase (W/O) is in contact with the dye aqueous phase to form three phase (W/O/W) emulsions process. The retention time on the separation of Red 3BS dye was varied in the range of 3 to 9 minutes as shown in Figure 4.



Figure 4. Effect of retention time on extraction efficiency (Experimental conditions: [Aliquot] = 0.1 M; [D2EHPA] = 0.08 M; agitation speed = 250 rpm; TR = 1: 3 [Span 80] = 3 % w/v; [NaOH] = 0.1 M; and [1-octanol] = 5 % w/v; [SA] = 0.1 M; T =  $\pm 26 \degree$ C)

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It is observed that after 3 min contact time, the solute extraction performance become plateau for all range of retention time indicating minimum time required to reach steady state in a continuous extraction column. It is evident that minimum retention time needed for the process is 3 min. This is due to contact time of the emulsions phase and dye aqueous phase is sufficient for the dye extraction to occur thereby extraction efficiency was good. This finding is in agreement with study of Lee and Kim, 2011 who found that a sufficient retention time gives a better dispersion of LSM into the external phase [14].

In stripping process, it becomes important to extract back the extracted species from membrane phase and allow recycling of the organic phase without loss of efficiency. Table 3 depicted the influence of retention time on dye enrichment. The result shows that 5 times enrichment was achieved with 3 minutes retention time. This could be due to a fact that short contact time is insufficient for dye ion to diffuse through membrane internal interphase for stripping process. As a result, more time is required for recovery. Further increment of retention time to 5 minutes, the dye enrichment was improved to 9 times. This is due to longer contact time between emulsions and dye aqueous phases. This impressive increase in the dye enrichment can be attributed that sufficient time given opportunities for more dye ion transported over the membrane into the stripping phase for recovery. Consequently, enhance the solute-carrier complex formation in the membrane and accelerates the stripping efficiency [20]. Generally, fluid circulated by the impeller (rotational speed) must be swept into the entire vessel in a reasonable time (retention time) in order to enhance the extraction in the continuous extractor [17].

Retention Time (min)	Enrichment
3	5
5	9
7	6
9	3

 Table 3. Effect of retention time on enrichment

However, increase the retention time over 5 minutes to 7 and 9 minutes reduced the dye enrichment to 6 times and 3 times, respectively. Increase of retention time creates instability to the emulsions. At this point, extending the contact time between emulsions and dye aqueous phase means more entrainment of aqueous to stripping aqueous phase of emulsions were promoted and lead to the emulsions swelling or breakage. Consequently, it will reduce the recovery efficiency. Study of Kulkarni and Mahajani, (2002) obtained similar result where longer retention time lead to more transport of water into the internal aqueous phase causing to swelling of membrane [11]. Therefore, 5 minutes is the optimum retention time as it retains the maximum capability of extract dye from the feed solution and recovery efficiency.

# 3.4 Effect of treatment ratio

Figure 5 depicted dye removal performance influenced by the volume ratio of emulsions to dye aqueous phase on. By adding the feed liquid phase quantity in the LSM, the percentage of extraction was consistent, almost 100 % of dyes were extracted within 15 min of extraction time indicating the CLSM process was stable and has led to the increasing of mass transport of solutes at the external into the membrane phase. This finding indicates that same amount of emulsions con provide good extraction performance up to 1:9 treatment ratio.

The enrichment and swelling effect of four samples with varies treatment ratio under the operation condition are exhibited in Table 4. At treatment ratio 1: 3, the dye enrichment was 9 times. With decreases in treatment ratio, the emulsions volume is larger in the system. This results to the greater size of emulsions globules formation. In this case, the possibility of

membrane instability was taken into account as high amount of emulsions enhances the globules interactions, leading to coalescence of globules. As a result, the globules form coalesced easily and emulsions will simply break during the extraction process. Eventually, this phenomenon will cause membrane to rupture, thus causing the stripping phase to leak into the feed aqueous phase. In addition, the more amounts for the emulsions was being used to extract dye ion at the lower treatment ratio means. To obtain optimum recovery through respect to the dye aqueous phase, from the process point views, this is undesired treatment ratio [21].



**Figure 5**. Effect of treatment ratio on extraction efficiency (Experimental conditions: retention time = 5 min, agitation speed = 250 rpm; [Span 80] = 3 % w/v; [1-octanol] = 5 % w/v: [NaOH] = 0.1 M; [SA] = 0.1 M; [D2EHPA] = 0.08 M; [Aliquat]=0.1 M; and T = ± 26 °C )

Consequently, it was found that treatment ratio of 1: 5 and 1: 7 reached high magnitude of enrichment which was 12 and 11 times, respectively. This can be attributed that at high treatment ratio, the amount of dye exist in dye aqueous phase is higher. Thus, more dye ion has been stripped. Therefore, the dye enrichment was increased. According to Kumar et al, (2018), with a higher treatment ratio less emulsions is used for extraction which is desired from the point view of maximum enrichment of metal with respect to the feed phase [22]. At the same time, the process was stable where no breakage or swelling was observed. The possible explanation for this was at a greater treatment ratio, effect of the osmotic pressure was reduced hence produces smaller size of globules. Consequently, the membrane stability increases.

As the treatment ratio was increase from 1:7 to 1:9, the enrichment was decrease from 11 to 6 times, respectively. This is attributed to the rupture of emulsions globule where further increase treatment ratio leads towards an increasing osmotic pressure gradient between the emulsions and feed aqueous phases. This finding was further support by Sulaiman et al., (2014) which stated that with higher treatment ratio, emulsions breakage happened [23]. Besides, the emulsions droplets may not contain enough internal reagents to react with the dye ion transported through the liquid membrane. Accordingly, the extracted dye ions accumulate in the membrane phase and carrier does not diffuse back to react with the dye in the dye aqueous phase. Therefore, the dye enrichment dropped. In order to make sure good dispersion of emulsions, treatment ratio of 1: 7 was considered as an optimal condition due to its stable performance of extraction and enrichment. Furthermore, application of lower volume of emulsions is economical feasible.

 Table 4. Influence of treatment ratio on emulsions breakage and enrichment

Treatment ratio	Enrichment	Breakage (%)
1:3	9	4

1:5	12	-
1:7	11	-
1:9	6	-

#### 4. Conclusion

This study concludes that the optimum condition of CLSM was obtained at 250 rpm agitation speed. 5 minutes of retention time and treatment ratio of 1: 7. At these favourable conditions, the remarkable Red 3BS extraction percentage achieving almost 100 % and concentrated in the stripping liquid phase where its enrichment of 11 times. This has demonstrated the high performance of the CLSM process. Eventually, this study shows the promising method of CLSM and contribution to the knowledge of continuous-process of reactive dye waste solution treatment by liquid emulsions membrane for further works.

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