# Removal of safranin from aqueous solution through liquid emulsion membrane

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**Abstract.** One of the real issues of the recent years is water contamination because of harmful synthetic dyes. Liquid Membranes (LM) resemble a promising alternative to the current separation processes, demonstrating various points of interest as far as effectiveness, selectivity, and operational expenses. The improvement of various Liquid Membranes designs has been a matter of examination by few researchers, particularly for the expulsion of dyes from aqueous solutions. The choice of organic surfactants plays an essential role in the efficiency of the dye removal. In LM design, the most significant step towards productivity is the decision of the surfactant type and its concentration. Liquid emulsion membrane (LEM) was used to remove safranin from aqueous solutions in which the emulsion was made with the help of D2EHPA as carrier, kerosene was used as a diluent and Span 80 (Sorbiton monooleate) was used as an emulsifying agent or surfactant. Various sorts of internal stages were utilized, to be specific sulphuric acid and sodium hydroxide. The impact of parameters influencing extraction efficiency such as pH of feed solution, concentrations of surfactant and emulsifying agent in membrane phase, volume ratio of internal phase to membrane phase, internal phase concentration, agitation speed and time of extraction were analyzed.

**Keywords:** liquid membrane, synthetic dyes, surfactant, safranin, D2EHPA

## 1. Introduction

Groundwater Disposal of large volumes of wastewater containing lethal organic dyes is a common problem of many industries around the globe. Adequate and inexpensive treatment process is therefore essential before the discharge of industrial effluents in water bodies. Incessant research efforts were being made either to treat such effluents for facilitating trouble-free disposal, or to recover the profitable chemicals and reuse the process water (Das *et al.* 2008). Organic substances are key effluents from various industries like textile, pharmaceuticals, pesticides & paper and pulp industries, dyes manufacturing, synthetic chemicals (Sumalatha B. *et al.* 2016). A variety of chemicals such as wetting agents, dyes, surfactants, fixing agents and other additives are used in several processes like bleaching and dyeing (Alamddine and El Jamal, 2009). It has been estimated that more than 700,000 tonnes of dyes are used out of which 15-20% are left in the effluent during the dyeing process (Robinson *et al.* 2001). Contamination of water resources with dyes is not desirable because of their aesthetically displeasing nature. The presence of residual colour, high levels of electrolytes, toxic effluents (S, vat dyes, enzymes, $CH_3COOH$, soaps, nitrates, and Cr compounds), heavy metals (Cu, Ar, Pb, Cd, Hg, Ni, and Co), mordants, dyeing auxiliaries severely affect the aquatic life. Carcinogenic by products are formed by reaction of disinfectants with dye fixing agents and non-bio degradable dyeing chemicals (Bhatt and Rani, 2013).

In spite of its drawback LM offers several advantages, such as, (a) large interfacial area for mass transfer, especially at the inner oil-water interface, due to the minute size of the aqueous phase droplets (b) high dispersion rate of the organic dye ions through the membrane (c) simultaneous and concurrent performance of extraction (at the outer interface) and stripping (at the inner interface) in the same system, and (d) potential of treating a variety of compounds in industrial settling at a better higher speed.

In the three-phase extraction technique solute molecules were extracted from a continuously flowing aqueous sample through an organic liquid phase into another usually temporally static, aqueous phase (Vu *et al.* 2003, Ren *et al.* 2009, Singh *et al.* 2010, Mondal *et al.* 2011). Liquid membrane (LM) is a relatively fresh and prospective separation system consisting of a liquid film through which selective mass transfers of gases, ions, or molecules occur via permeation and transport processes. Owing to its advantages over the solid membranes and solvent extraction (Boyadzhiev, 1990).

Das et.al.,(Das *et al.* 2008) studied the extraction of methylene blue (MB) and crystal violet (CV) using ELM. They made an ELM by using n-heptane as solvent, NaOH as the internal phase and dye solution as the feed or the external phase. Emulsion was stabilized using surfactant span 80. They found maximum extraction of 99% MB and 95% CV for single system and 97% MB and 90% CV for binary system.

The group of authors demonstrated possibilities for extraction and recovery of anionic dye yellow- 99 using ELM consisting of aliquat-336 as extractant, span 80 surfactant and cyclohexane as solvent. On the optimized parameters 99.98% of extraction has been found (Bahloul *et al.* 2016).

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Some of the authors have performed the experiments to remove the textile dyes using eatable oils in case of Supported liquid membrane and polypropylene as supported Teflon membrane. Finally, they concluded that in 5 hr maximum extraction was 97%. Muthuraman and Teng (2009) found 100% extraction of rhodamine B (RB) by using Palm oil as the solvent in supported liquid membrane at 300 rpm after 5 h with feed phase of 11 pH.

Hajarabeevia and Mohammed (2009) demonstrated cationic dyes facilitated transport through a SLM where D2EHPA acts as the carrier. They used coconut oil as the solvent to extract MB and RB. Using feed phase at a pH of 4, 50% D2EPHA carrier concentration and 100ml acetic acid as strip phase the SLM set up gives an extraction of 94.2% for MV and 90% for RB when run for 7 hours and at 600 rpm.

Liquid emulsion membrane (LEM) technique has been successfully applied by many researchers for the recovery of various synthetic dyes as well as heavy metal ions (Patnaik, 1995, Dâ¢as and Hamdaoui, 2010, Othman *et al.* 2011). However, best of my literature review I have not been found any literature about application of LEM technique for the removal of safranin from aqueous solution.

## 2. Reagents and procedure

### 2.1 Reagents

In this separation study safranin ($C_{20}H_{19}ClN_4$) was used as synthetic dyes from Sigma-Aldrich. The other name of safranin is basic red 2, cotton red. Di-(2-ethylhexyl) phosphoric acid (D2EHPA, $C_{16}H_{35}O_4P$) and kerosene was used as carrier and solvent or diluent. Span 80 (sorbitan monooleate, $C_{24}H_{44}O_6$) non-ionic surfactant used for stabilizing the emulsion, were obtained from SDS fine chemicals. Analytical grade sulphuric acid ($H_2SO_4$) for the preparation of internal phase; sodium hydroxide (NaOH), and hydrochloric acid (HCL) were purchase from the Sigma Aldrich. To analyze the removal efficiency of the ELM, 10-50 ppm solution of the dye were prepared by dissolving safranin in bidistilled water.

### 2.2 Experimental procedure and setup

#### 2.2.1 Feed phase preparation
A feed solution (50ppm) of safranin dye has been prepared by dissolving a known quantity of dye in de-ionized water. To analyze the pH effect on the extraction the feed dye solution pH has been adjusted from 1 to 10 by adding 2 N HCl or NaOH.

#### 2.2.2 Emulsion phase preparation
To prepare the 50 ml emulsion phase; 3% (v/v) Di-(2-ethylhexyl) phosphoric acid (D2EHPA) as a carrier, 3% (v/v) span 80 as surfactant and rest 94 % kerosene were taken. After agitation for 10 minutes at 4500 rpm to form a homogeneous emulsion phase, internal phase of known molarity of H2SO4 was added slowly in different ratios (ranging from 0.75 to 1) to the prepared membrane phase.

To get a stable emulsion phase agitator was used approximately at 5000rpm for 10 min.

#### 2.2.3 Extraction
The double emulsion of water in oil (w/o/w) was mixed in a conical flask to allow the extraction and stripping process. The phase was stirred for 5 minutes for varying speed of 300-900 rpm. After that the sample was quickly introduced into a separating funnel and left for phase separation for about 15 minutes. The two layers were formed in the separating funnel; upper layer is W/O emulsion, while bottom layer is aqueous treated phase. The bottom aqueous phase was examined after every 5 minutes for the concentration of dye using spectrophotometer at 518nm.

## 3. Results and discussion

To examine the effect on extraction of the safranin form the aqueous solution some parameters are varied viz the volume ratio of stripping phase to membrane phase, concentration of internal phase, concentration of carrier and surfactant, pH of feed phase and agitation speed. To achieve maximum extraction stability of the membrane is the main concern. The stability of the membrane is depends on the droplet size and emulsifier affinity for both organic and aqueous phase. Emulsifiers should be appropriate because, they play an important role in both emulsion formation and extraction.

### 3.1 Effect of the volume fraction of stripping phase

As we change the volume ratio of stripping phase to membrane phase the stability of emulsion is affected which leads to changes in extraction rates. As observed from Fig.1, on increasing the volume ratio of stripping to membrane phase from 0.75 to 2 by maintaining membrane volume constant, the percentage extraction of safranin changes from 94.30% to 96.09% with maximum extraction being 97.38% at ratio 1:1.

From the Fig.1, it was observed that the stability of emulsion was secured with increase in the phase ratio. The increment in the internal or stripping phase volume fraction shifted the internal drop size distribution towards larger sizes and increased the emulsion viscosity. The increase of droplet size further decreased the interfacial contact area between the emulsion and continuous phase and thereby decreased the extraction efficiency. Additionally, for higher volume ratios, the volume of organic phase was not sufficient for enclosing the entire internal aqueous phase (Juang and Lin, 2004) and the distribution of the W/O emulsion was more complicated.

In order to obtain a consistent dispersion of the internal phase droplets in the membrane phase and a good dissemination of the W/O emulsion in external phase, the volume ratio of internal phase to membrane phase of 1:1 was selected as the best ratio. A similar result was obtained in the extraction of congo red (CR), an anionic diazo dye, from aqueous solutions by emulsion liquid membrane (ELM) using span 80 as surfactant and kerosene, hexane and heptane as diluents (Dâ¢as and Hamdaoui, 2010).
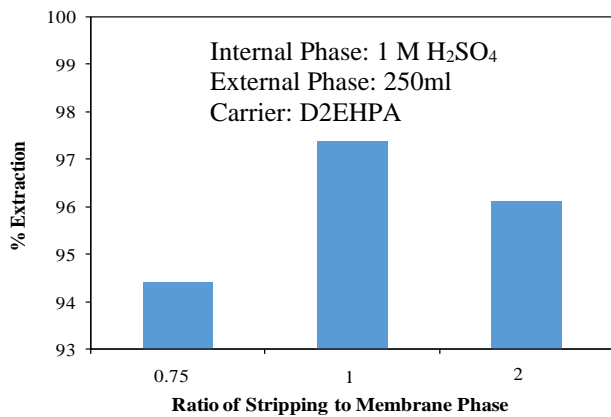
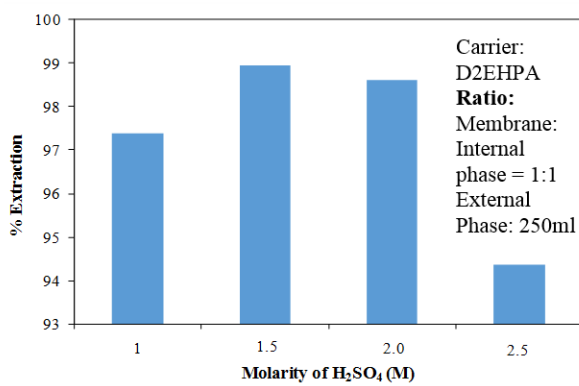Fig. 1 Effect of ratio of internal to membrane phase on percentage Extraction



Fig. 2 Effect of Molarity of $H_2SO_4$ on percentage Extraction

### 3.2 Effect of concentration of internal phase

Most of the authors has also been used $H_2SO_4$ as stripping agent in his study (Muthuraman and Palanivelu, 2006, Muthuraman and Teng, 2009). The effect of (stripping) internal phase concentration on emulsion stability has been shown in Fig.2. It shows that at lower concentration of $H_2SO_4$ as stripping agent, the extraction percentage and stability is low. The possible explanation of this result could be a complex formulation between the extractant and dye in the external phase during the extraction process. Moreover, the complex shifted through the membrane into the internal phase. While passing through the internal phase, the dye part of this complex will be stripped away by the stripping agent. At low concentration of the stripping agent, only a limited amount of dye will be stripped because all stripping agents will be completely consumed (Patnaik, 1995). However, a small concentration of the stripping agent does not significantly affect the stability of the emulsion. This is because as long as there was enough volume of stripping agent introduced to the carrier, it was sufficient to produce a good structure of emulsion. The experimental study has been carried using various concentration of $H_2SO_4$ as shown in Fig 2. It has been observed that 1.5 M $H_2SO_4$ gives better extraction. Therefore, in further experiments 1.5 M $H_2SO_4$ has been used.

Increasing the concentration of the stripping agent to 1.5 M will increase the percentage of extraction to 98.93%. However, further increasing the concentration beyond 1.5 M causes decrease in percentage extraction due to membrane instability at higher concentration beyond 1.5M $H_2SO_4$. Therefore, it can be suggested that at high concentration of internal phase membrane loss its stability.

### 3.3 Effect of carrier concentration

The conception of carrier facilitated transport is such that the ions of diffusing dye are carried through the membrane phase from external-membrane interface to internal-membrane interface by subsuming a carrier compound (Othman *et al.* 2011). Figure 3 shows the effect of carrier concentration in the organic phase on the extraction and stripping of safranin. The changes in concentration of D2EHPA from 0.09 M to 0.25 M presents a significant increase in the permeation rate of safranin dye ion up to 0.15 M through external-membrane interface followed by gradual decrease up to 0.25 M. From these result it could be suggested that 0.15 M concentration of D2EHPA is optimum. Therefore, in our case in order to get maximum extraction at .15 M D2EHPA 1.5 ml) which shows the 3.0 % of total membrane phase.

Higher D2EHPA concentration accelerates the migration of the dye ions towards the oil phase interface encouraging exceptional extraction process. However, the enhancement is less prominent when D2EHPA is increased beyond 0.15 M as it established lowered rate of extraction, recovery and stripping. This might be because more amount of D2EHPA would result in an pronounced boost in the membrane thickness hence combating the dye ion to infuse through external-membrane interface to perform extraction process and permeate through membrane-internal interface to carry out stripping process (Hajarabeevi *et al.* 2009). Membrane viscosity and interfacial tension between membrane and external feed phase is found to increase dominated by the formation of larger globules thus decreasing the diffusivity of the dye-carrier complex in membrane phase (Srivastava *et al.* 2017). As depicted from the Fig. 3, the best concentration of D2EHPA to promote the optimum ELM parameter is 0.15 M giving 99.34% extraction of safranin.

### 3.4 Effect of surfactant concentration

Surfactant concentration is an important factor as it directly affects the stability and permanence of ELM, the swelling of emulsion and the rate of extraction (DÃ¢as and Hamdaoui, 2010). The effect of surfactant concentration on extraction rate and stripping of the dye was enquired and studied in the concentration range of 1-4 % (w/v) as shown in Fig.4. The fig. 4 represents the variation of percentage extraction from 99.34% to 98.63%. This indicates that the emulsion stability and the percentage extraction was strongly dependent on the concentration of surfactant. The percentage extraction increases to 99.90% by increasing the concentration of surfactant up to 2% (w/v) and gradually decreases afterwards. Because emulsions become fragile and breaks effortlessly because this amount is inadequate for neighbouring the entire internal aqueous phase.
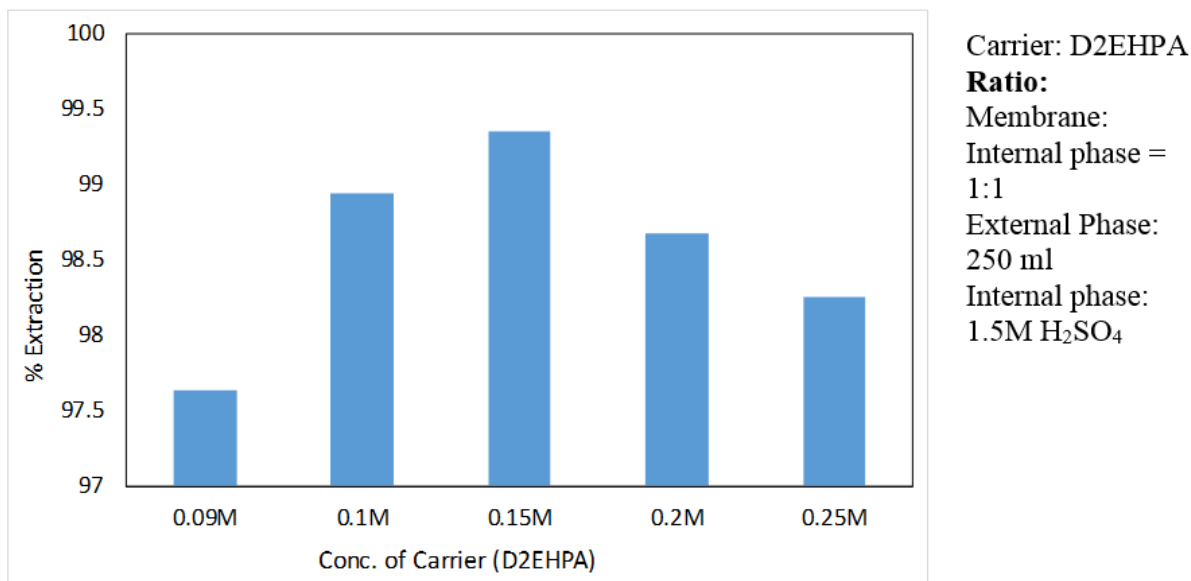
Fig. 3 Effect of Molarity of D2EHPA on percentage Extraction

Carrier: D2EHPA
**Ratio:**
Membrane:
Internal phase =
1:1
External Phase:
250 ml
Internal phase:
1.5M $H_2SO_4$

Further increase or decrease in pH of the feed phase
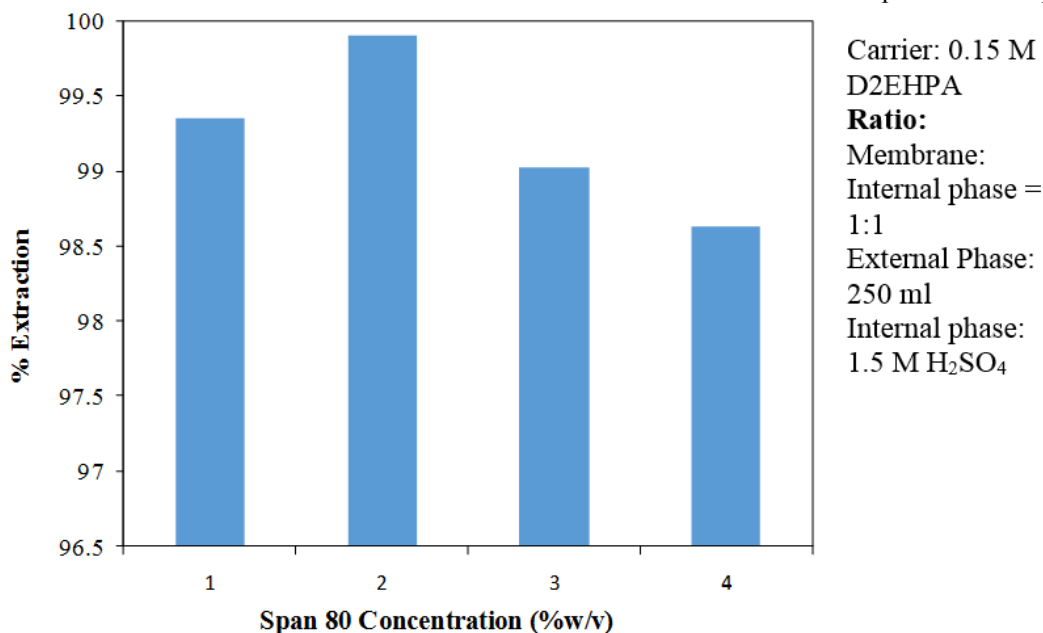


Fig. 4 Effect of Concentration of Span 80 on percentage Extraction

Carrier: 0.15 M
D2EHPA
**Ratio:**
Membrane:
Internal phase =
1:1
External Phase:
250 ml
Internal phase:
1.5 M $H_2SO_4$

Consequently, the concentration of surfactant in the membrane phase must be minimum but it should be enough to stabilize the emulsion (Bahloul *et al.* 2016).

### 3.5 Effect of pH on extraction

To study the effect of pH of feed or external phase on the extraction of dye from the aqueous solution, it was varied between from 1 to 10. The results of the variation of percentage extraction with pH of dye solution are depicted in Fig. 15. The carrier and surfactant concentration in the membrane phase were kept 0.15 M and 5% w/v respectively. It can be inferred from Fig. 5, that the maximum extraction was found to be 99.90% at pH 7.

lowered efficiency of extraction due to the decrease in fraction of dye molecules present in the cationic form in the feed solution (Hajarabeevi *et al.* 2009)

### 3.6 Effect of agitation speed

The agitation speed plays a major role in the rate of extraction of safranin through emulsion liquid membrane process. In the mixing vessel, the emulsion is dispersed generally into a large excess of the external phase (Othman *et al.* 2013). The effects of mixing speed on percentage extraction are shown in Fig. 6. The results of series of experiments varying the agitation speed from 300 to 900 rpm shows that the emulsion stability and percentage extraction increases from 99.43% to 99.90% on increasing
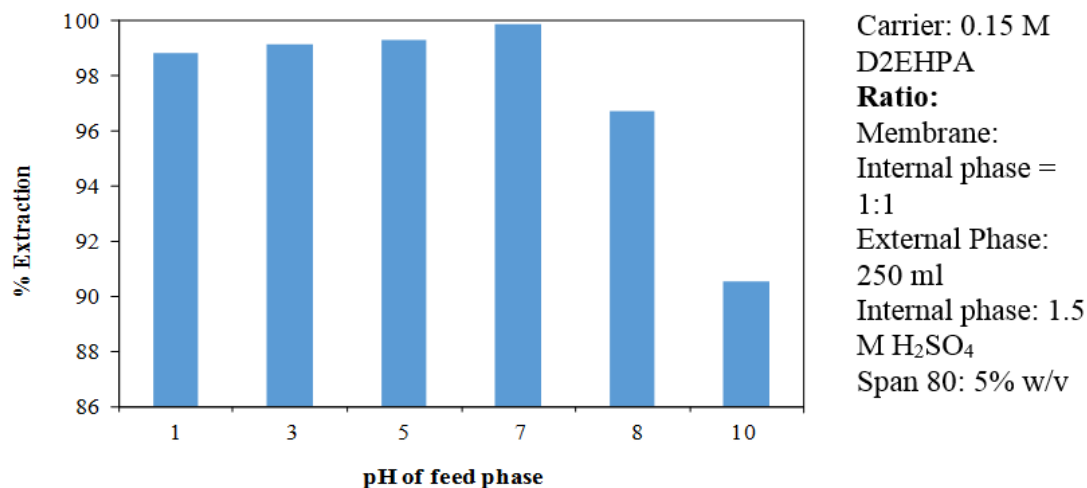
Carrier: 0.15 M
D2EHPA
**Ratio:**
Membrane:
Internal phase =
1:1
External Phase:
250 ml
Internal phase: 1.5
M H$_2$SO$_4$
Span 80: 5% w/v

Fig. 5 Effect of pH of feed phase on percentage Extraction



Carrier: 0.15 M
D2EHPA
**Ratio:**
Membrane: Internal
phase = 1:1
External Phase: 250
ml
Internal phase: 1.5 M
H$_2$SO$_4$
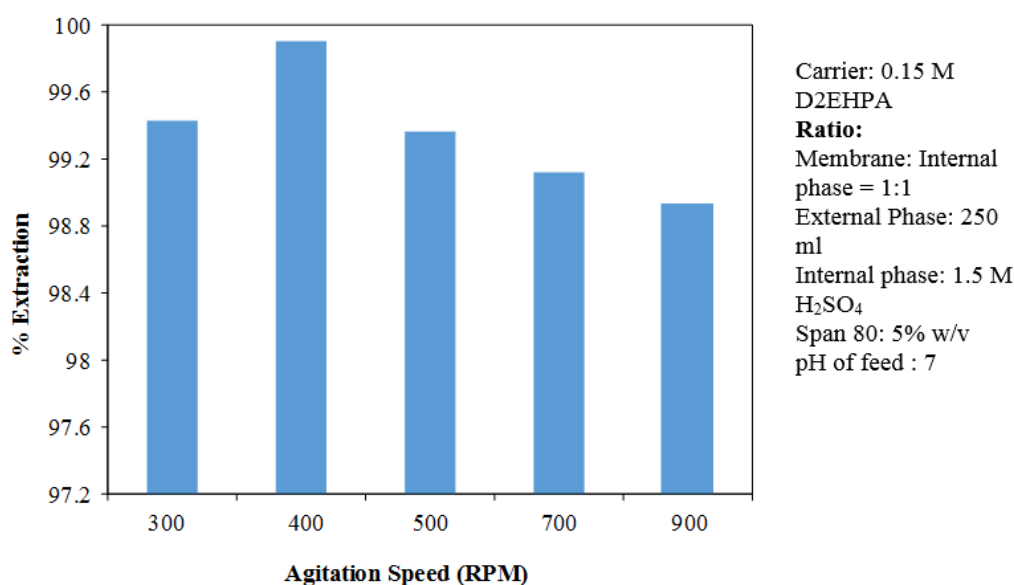Span 80: 5% w/v
pH of feed : 7

Fig. 6 Effect of agitation speed on percentage Extraction

the agitation speed from 300 to 400 rpm and gradually decreases to 98.93% thereafter. Raising the agitation speed and the time for preparing the W/O emulsion is beneficial to membrane stability due to formation of smaller internal droplets, but raising the speed for dispersing the W/O emulsion in the external phase results in an increase in membrane breakage. Increase in the level of agitation and mixing would possibly amplify the mass transfer rates and interfacial area. But, up to some level of mixing speed, the breakage the emulsion droplets it is also likely due to the shear generated, thereby lowering the overall extraction performance. This specifies that the aqueous boundary thickness might shrink and diminish continuously with increase in stirring speed and that the boundary layer is least at 400 rpm. Further decrease in dye extraction from 400 rpm could be possibly due to the high turbulence caused by shear forces at elevated speeds, resulting in the displacement of carrier molecules from the membrane pore. The consequence of hydrodynamic instability due to swelling is a very intricate phenomenon. At a microscopic level, the emulsion tendency to break is because of the

shear attaining a larger size and simultaneously swelling is also performed. There exists a trade-off between these two effects. Either the swollen particles can breakdown on their own or the breakage is induced by shear (Djenouhat *et al.* 2008). Increasing the agitation speed above a critical value not only decreases the extraction rate, but also affects the stability of the emulsion and makes the emulsion inconsistent. At higher agitation rate, hydrodynamic shear and swelling work together causing decrease in percentage extraction.

## 4. Transport mechanism

In liquid emulsion membranes technique the separation of dyes is occurs according to the film model of mass transfer theory. The complete separation process is divided into the following steps:
- Diffusion through the boundary layer in the feed solution,
- Sorption on the feed solution/liquid membrane

interface,

- Diffusion through the boundary layer on the feed side,

- Transport in the membrane, e.g. convective transport in the mixing zone,

- Diffusion through the boundary layer on the receiving side,

- Desorption on the membrane/receiving solution interface,

- Diffusion through the boundary layer in the receiving solution.

## 5. Conclusions

It is demonstrated that the ELM technique is very promising in extracting safranin from aqueous solution. In order to achieve the objective several parameters have been studied such as ratio of stripping to membrane phase, concentration of $H_2SO_4$, concentration of D2EHPA, concentration of SPAN 80, pH of dye solution and agitation speed in ELM system. The most suitable parameter conditions are at 1:1 volume ratio of internal aqueous phase to membrane phase, 1.5 M sulphuric acid, 0.15 M concentration of D2EHPA, 5% (w/v) concentration of SPAN 80, neutral pH of feed solution and 400 rpm agitation speed. At this optimum condition almost 99.90% of the dye was extracted. Investigation of the optimum condition of ELM system is necessary in order to realize the intention of ELM in removal of dye. ELM technique is thus a promising alternative to conventional methods and should increase awareness of the potential for recovery of dyes.

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