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Comparative Study of Liquid –Liquid Extraction and Bulk Liquid Membrane for Rhodamine B

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Abstract:- In the liquid liquid extraction (LLE) the extraction of rhodamine B (RB) from aqueous solutions by using 0.8M phenol in xylene was studied. The influencing parameters such as the effect of pH of the aqueous phase, the effect of extractant concentration, the effect of diluents and the stripping reagents has been investigated. The maximum extraction efficiency was obtained at pH 1±0.1 and the maximum stripping efficiency of dye from loaded organic dye was obtained using 10M acetic acid concentration. In the liquid membranes (LM), the above optimum conditions were applied and carried out further experiments. The extraction and stripping process were carried out in single step it is called pertraction process. The influence of pH of the aqueous donor phase, phenol concentration in the membrane phase, the effect of stripping reagents in the received phase rate of stirring speed, transport mechanism has been investigated.

Key words: **Efficiency, liquid membrane, Organic phase, Rhodamine B, Stripping.**

I. INTRODUCTION

Dyes are widely used textile industries for coloring fabrics and it is highly visible material. When release of dye into the environment is associated with the incomplete exhaustion of dyes onto textile fiber from an aqueous dyeing process and the need to reduce the amount of residual dye in textile has thus become a major concern in recent years. Once the dye has contaminated into the water its removal by conventional wastewater treatment method is particularly difficult because many of the dyes are stable to light and oxidizing agents and are resistant to aerobic biooxidation [1]. There are several methods have been investigated for color removal from industrial effluents to decrease their impact on the environment. Various methods have been used to remove dyes from aqueous solution, the mostly used methods such as photochemical degradation, adsorption, electropenton's flocculation, chemical oxidation, hypochlorite, adsorption, ozone treatment [2]-[7]. The advantages and disadvantages of some methods of dye removal from wastewater are given in Table 1.

Table I Advantages and Disadvantages of Some Physical and Chemical Methods

Physical and chemical methods	Advantages	Disadvantages
Fenton's reagent	Effective decolourisation	Sludge generation
Ozonation		Short half life
Photochemical	No change in effluent volume	Formation of by-products
NaOCl	No sludge generation	Release aromatic amines
Cucurbituril	Initiate azo-bond cleavage	High cost
Electrochemical	Good sorption capacity of dyes	High cost of electricity
Activated carbon	Non-hazardous end products	Very expensive
Peat	Highly effective for various dyes	Surface area is low
Silica gel	Good adsorbent	Side reactions in effluent
Membrane filtration	Effective for basic dyes	Concentrated sludge production
Ion exchange	Removes all dyes	Not effective for all dyes
	No adsorbent loss	

In the literature study, Adsorptive removal of RB by activated carbon prepared from perthenium biomass through sulfuric acid treatment was also reported [8]. The decolourization and degradation of RB in aqueous solution by UV/H_2O_2 treatment was studied [9]. Use of vegetable oil in supported liquid membrane for the transport of RB was also studied [10]. Determination of RB in soft drink, wastewater and lipstick samples after solid phase extraction was studied [11]. Removal of the hazardous dyes RB from wastewater photo catalytic and adsorption treatments by using TiO_2 has been investigated [12]. Liquid liquid extraction has been useful technique of removal of textile anionic and cationic dyes from industrial waste water [13]-[18]. Liquid membrane (LM) systems are being studied extensively by researchers in such fields as analytical, inorganic and organic chemistry, chemical engineering, biotechnology and biomedical engineering and wastewater treatment etc. According to configuration definition, can be divided into three types of membranes such as bulk liquid, supported (or) immobilized liquid and emulsion liquid membrane. Further, technological problems with the stability of emulsion liquid membrane and supported liquid membrane gave led researchers in recent years to look for alternative bulk liquid membrane or bulk water immiscible liquid membrane. BLM

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separation combines the solvent extraction and stripping processes re-extraction in a single step Compared with conventional separation processes, such as LLE, membrane techniques are characterized by the technical simplicity and high efficiency in separating (or) enriching material from gaseous or liquid mixtures. Also, the main advantage of liquid membrane is the amount of organic solvent and carrier (extractant) is remarkably reduced. BLM is one of the simplest designs for performing liquid membrane processes, which are often used to investigate the complexation and transport properties of synthetic and natural ionophores with salts [19], [20]. Liquid membranes have been successfully used to treat aqueous solutions contaminated with heavy metal ions such as copper, zinc, cadmium, nickel, mercury, lead, silver, palladium and gold [21]-[24], amino acids and many studies have been carried out using liquid membranes for separation and concentration of amino acids to human food animal feed additivities and in the pharmaceutical field. The solvents, carriers and phase modifiers are invariably diluted in the organic diluents such as kerosene, chloroform, dichloromethane, n-dodecane, isododecane, n- heptanes, n- hexane etc. [25]-[27]. In the present study, the removal and recovery of RB from wastewater using phenol in xylene was studied. In LLE, the affecting parameters was also included; the effect of pH, the effect of extractant concentration and the effect of stripping reagents and concentration. In BLM, kinetic study, effect of stirring speed, interference study and effect of initial feed phase concentration has been investigated.

II. EXPERIMENTAL

A. Materials and Methods

Phenol (99%, Merck), hexane (95%, Merck), toluene (99.5%, Merck), xylene (99.9%, Merck), benzene (99%, Merck), hydrochloric acid (35%, Merck), sodium hydroxide (97%, Qualigens), Rhodamine B (99.9%, S D Fine), salicylic acid (99.8%, Merck), benzoic acid (99.5%, RFCL), oxalic acid (99%, Merck), acetic acid (99.6%, Merck) these chemicals were used without further purification. The absorbance of dye sample was determined using UV visible spectrophotometer (Elico SL 159). The pH adjustments of source phase (dye solutions) using Elico LI 120. Systronics Electrophoresis 606 was used to find out whether the dye is cationic or anionic. The three phases were stirred with mechanical stirrer using REMI lab stirrer.

B. Procedure

LLE: solvent extraction experiments were carried out at 30 ± 0.5˚ C. The pH of the dye solution was adjusted by using base like 0.02N and 1N sodium hydroxide solution. A feed phase containing dye (100mg/L, V=25mL) and the organic phase (V=25mL) were introduced in a separating funnel. The two phases are immiscible with each other. The two phases were mixed gently for known time and then left to separate. The raffinate was collected for measurement of the remaining dyes in calculated as per the following equations.

$$E = 1- [Dye]_{aq}/[Dye]_{aq0} \times 100 \quad \text{- - - - - - - - - (1)}$$

Where $[dye]_{aq}$ = dye concentration in the aqueous phase (mg/L), $[dye]_{aq0}$ = initial dye concentration in the aqueous phase, E = percentage of extracted dye.

In the stripping, the loaded organic dye (V=25mL) and the stripping solution, acetic acid (V=25mL) were added together into a separating funnel and shaken at 100rpm. The aqueous strippant was taken for measurements of absorbance. From this value, the percentage of stripped dye was calculated by the following equation.

$$R = [Feed]_s/[feed]_{aq0} \times 100 \quad \text{- - - - - - - - - (2)}$$

Where R = percentage of stripped dye, $[feed]_s$ = concentration of stripped feed phase and $[Feed]_{aq0}$ = initial concentration of feed phase.

BLM: H type BLM contains three phases, two of the phases are aqueous phases namely feed (donor) and strip {receiving (or) acceptor} phase and the third phase is membrane phase shown in fig1. The aqueous phase containing dye solution (100mg/L, V=300mL) and receiving phase was filled in acetic acid (10M, V=300mL). Equal volumes (300mL) of aqueous solutions for the feed and stripping phase were placed into the two compartments. These two layers were separated by the organic solvent such as xylene which acts as the liquid membrane (LM) phase. After the phases were stirred well with mechanical stirrer and then it was conformed that in the absence of phenol in the membrane phase, transfer of dye across the membrane does not takes place.

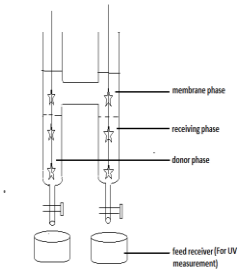

Fig.1. 'H' Type Bulk Liquid Membrane

III. RESULTS AND DISCUSSION

A. Liquid extraction experiments

1. Effect of pH of feed phase

The pH of the initial solution plays an important role in extraction process due to salts and other additive also affected the pH of dye containing wastewater. Extraction of RB from the aqueous solution was studied using 0.8 M phenol with the pH range from 9 to 13 ± 0.1 of different dye concentrations. The pH was adjusted using 1N and 0.02N NaOH. The extraction efficiency of the dye increases with increasing pH of the feed phase. The maximum extraction efficiency of dye obtained were as follows: 99.0% for 100mg/L, 95.6% for 200 mg/L, 93.2%

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for 300 mg/L and 90.8% for 400 mg/L was found at pH 12 ± 0.1 shown in fig 2. For further studies, it was decided to maintain the extraction at pH 12 ± 0.1. Hence the suggested mechanism was given below.

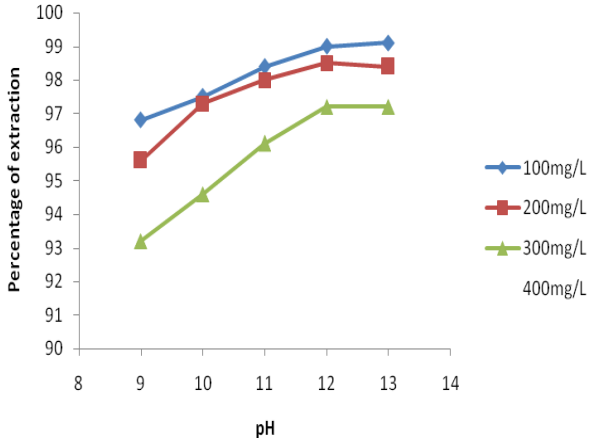


Fig.2. Effect of pH on the extraction of Rhodamine B (Experimental conditions: volume of feed phase = 25 mL, volume of organic phase = 25 mL, extractant concentration 0.8 M and equilibrium time = 8 min)

2. Effect of extractant concentration in the organic phase

Extraction experiment was first carried out in the absence of extractant. It was found that there was no extraction of RB from feed to organic phase. So, it requires some kind of anionic carrier (extractant) since RB is a cationic dye. In this study phenol acts as a phenoxide anionic carrier. Hence phenol has been selected as a carrier. The effect of phenol concentration on the extraction of RB was studied with varying extractant concentration from 0.2 M to 0.9 M. The experimental data for the percentage of extraction of dye increased from 98.0 – 99.0% with increase in extractant concentration from 0.2 to 0.9 M at pH 12 ± 0.1 was shown in fig 3. This confirms that 0.8 M phenol is effectively extracting RB from aqueous solution. So 0.8 M phenol was used for further experiments.

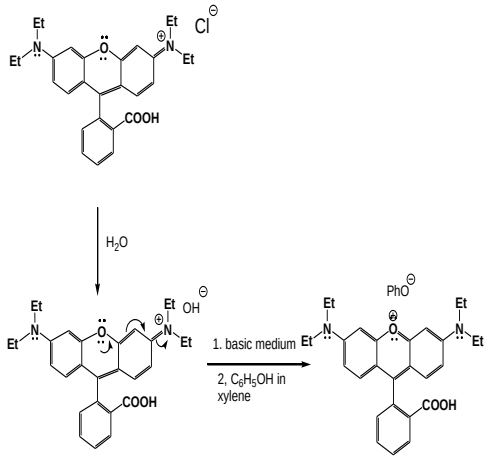


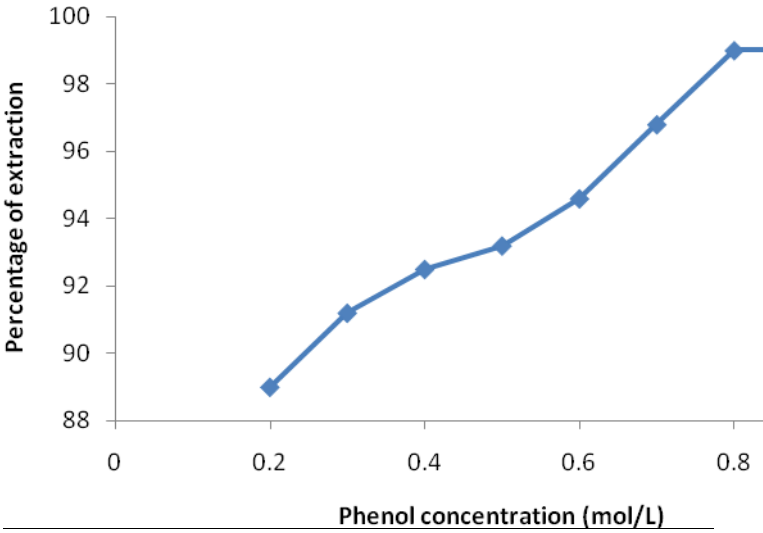
Fig.3. Effect of phenol concentration (Experimental conditions: volume of feed phase = 25 mL, volume of organic phase = 25 mL, pH 12.0 ± 0.1 and equilibrium time = 8 min).

3. Effect of diluents

Phenol is sparingly soluble in water and organic solvents which is colourless solution. When used organic solvents which is immiscible with water. The RB was extracted with organic solvents that contain several common organic compounds and immiscible with water. Hexane were used from the group of aliphatic hydrocarbon, toluene, benzene and xylene were used from the group of aromatic hydrocarbons. Among them aromatic hydrocarbons extracted the dye very effectively at pH = 12 ± 0.1 using 0.8 M phenol and the results were presented in table 2. So xylene was selected for the extraction of RB for further studies, due to it is less toxicity compared to benzene and toluene. More than 99.0% of the dye was extracted from aqueous solution using xylene as a solvent.

Table II Effect of Diluent

Diluents	Percentage of extraction			
	100mg/L	200mg/L	300mg/L	400mg/L
Benzene	96.0	94.5	90.0	87.5
Toluene	98.5	95.0	93.8	92.0
Xylene	99.0	98.5	97.2	94.6
Hexane	90.0	85.0	76.0	60.0



4. Effect of stripping reagent concentration

Stripping means the back extract of dye from loaded organic phase to aqueous phase. The RB was extracted with organic solvents that contain several common organic compounds and immiscible with water. Various aromatic and aliphatic stripping reagents such as salicylic acid, benzoic acid, acetic acid, and oxalic acid were used in this experiment. Among them acetic acid was found to be strip very well from loaded organic phase to aqueous phase. Acetic acid is a weak aliphatic acid compared with others and maximum amount of dye was

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stripped from loaded organic phase using it. Further study was carried out acetic acid as a stripping agent. The results were presented in Table 3. It shows that the stripping efficiency increased with increasing concentration of acetic acid from 6 M to 11 M. Maximum stripping efficiency was obtained at 10 M acetic acid concentration. This might be due to H^+ ion increased with increasing acetic acid concentration, H^+ ion interact with loaded organic dye and the dye was successfully stripped.

Table III Effect of Stripping Agent Concentration

Acetic acid concentration (mol/L)	Percentage of extraction
7	96.5
8	97.0
9	97.6
10	98.0
11	97.7

B. Bulk liquid membrane experiments

1. Effect of carrier concentration in the membrane phase

In the transport process, carrier plays an important role, which act as a phase transfer catalyst. In a blank experiment was performed without carrier (only solvent) filled in the membrane phase there was no detectable movement of dye through the liquid membrane was found in this process. Therefore it reveals that, the transport of dye was carried out when carrier was added with the solvent in the membrane phase. The experiments were performed by varying amount of the carrier in the range from 0.2M to 1.0M. The results were shown in fig 4. It shows that the transport of dye was follows: 0.2M for 35%, 0.4M for 60%, 0.6M for 75%, 0.8M for 86% and 1M for 87% were obtained. It means the transport of dye was increases with increasing carrier concentration in the membrane phase. It might be because the formation of dye-phenoxide ion pair complex was high in the presence of 0.8M phenol and therefore the extraction of dye from source phase to membrane phase was successfully achieved.

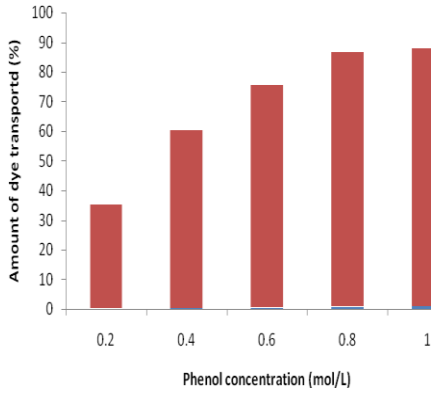

Fig.4. Effect of phenol concentration on the feed phase Experimental conditions: donor phase: 300ml for RB, receiving phase=300ml for acetic acid, membrane phase=750ml for various phenol concentration, pH=12 ± 0.1 and transport time 150 min.

2. Effect of pH of the donor phase

The results were presented in table 3. It reveals that the amount of dye transported was least at pH 7 ± 0.1 which started pH up to a value of pH 12 ± 0.1 beyond no change. At pH 7 ± 0.1, the formation phenoxide occurs less, resulting in the membrane of the solubility of phenol in the dye was less therefore the lower amount of dye was transported. With increasing pH (>7), the interaction between the hydroxyl ions and the dye for phenol also increases. At near pH 12 ± 0.1, it was seen that the complexation is more favored and the transport rate increases. Hence the pH of the feed phase was adjusted to 12 ± 0.1for all further experiments.

Table IV Effect of pH on the donor phase

pH	Extracted (%)	Stripped (%)
7	46	25
8	63	44
9	75	55
10	81	62
11	90	70
12	96	86

3. Effect of time

The experiment was performed for investigation of the effect of time shows that the dye extracted from feed phase into the organic phase as well as those released in the receiving phase increase with time, a quantitative transport of dye takes place beyond 150 min. Fig. 5 shows that when increases the time; the concentration of dye was gradually increased in donor phase. It indicates that the dye was extracted from donor to membrane phase. The dye concentration was increased up to maximum (29%) in membrane phase after the dye was decreases it means that the dye was successfully stripped from membrane to receiving phase. The maximum dye (96%) transported at 150 min. Hence the time 150 min was recommended for further experiments.

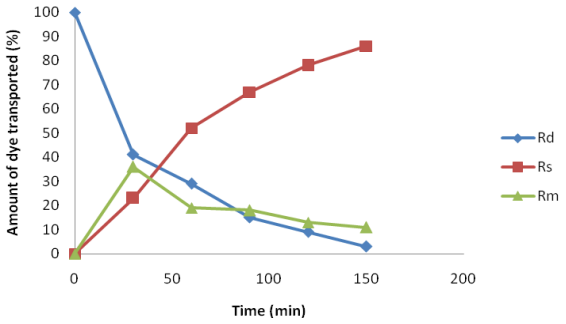

Fig.5. Effect of time on dye transport. Experimental conditions: feed phase=300ml for 100mg/L RB, receiving phase=300ml for 10M acetic acid, membrane phase=750ml for 0.8M phenol, pH 12 ± 0.1 and transport time=150min.

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4. Mechanism of transport of dye

Based on the results obtained in the present study, a coupled mechanism is proposed for the transport of cationic dye across the bulk liquid membrane. At one of the compartment such as the donor side, dye-phenoxide ion pair complex was formed at the feed membrane interface; it dissolves completely in the membrane phase and gets distributed throughout the organic solvent. The carrier anion receives proton from the acetic acid in receiving phase and then diffuses back into the organic membrane as neutral carrier [28]. At another compartment receiving phase, the dye-phenoxide ion pair complex was reacts with acetic acid in the membrane receiving interface and then dye was diffuses into the receiving phase. The proposed mechanism was shown in fig 6.

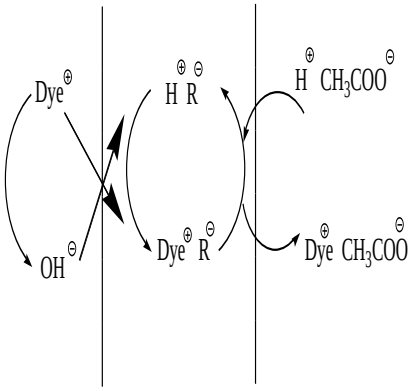

Fig.6. Schematic Representation for the Proposed Transport Mechanism

5. Comparison of LLE and BLM for transport of RB dye

The transport of dye through BLM and LLE maintaining the same content of feed and receiving aqueous phases were compared. In LLE, which had two steps like extraction and stripping carried out whereas in BLM carried out extraction and stripping simultaneously. The values of percentage of transport of dye through BLM and LLE the important affecting parameters were compared and the results were presented in Table V. It reveals that in LLE the dye was maximum extracted in a short time whereas in BLM the maximum transport of dye was occurred at 150min. Because LM processes are slow process more or less extraction and stripping occurs simultaneously.

Table V Comparison of LLE and BLM

Parameters	Liquid liquid extraction	Bulk liquid membrane
pH	12 ± 0.1	12 ± 0.1
Extractant Concentration	0.8M phenol	0.8M phenol
Diluents	Xylene	Xylene
Stripping reagent	10M Acetic acid	10M Acetic acid
Time	5min for extraction and 8min for stripping	Transport time150min
Maximum % of extracted dye	99.5% of dye extracted	97% of dye extracted

IV. CONCLUSION

In the present study demonstrates, using of phenol as an excellent carrier for the efficient transport of RB through BLM. The transport of dye was maximum (86%) obtained using 0.8M phenol concentration and pH 12 ± 0.1 in the donor phase, 10M acetic acid in the receiving phase, stirring speed 200rpm and transport time sat 150min. This study demonstrates that the usefulness of the LM technique for making it possible to combine extraction and stripping operations in a single step process. It may be applied for recovery of dyes from textile effluent in textile industry.

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