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Carrier Assisted Emulsion Liquid Membrane Process for Recovery of Basic Dye from Wastewater using Continuous Extractor

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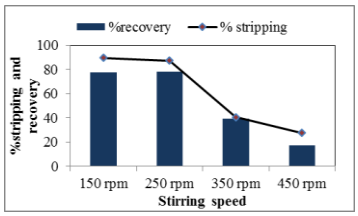
Graphical abstract



Abstract

Nowadays, water pollution has become major issue especially dye contaminated wastewaters from the textile industry. Dye causes serious environmental pollution and health problems. The removal of color from dye-contaminated wastewaters in the related industries becomes a major concern all over the world. In this research, several parameters of dye extraction and recovery in the continuous emulsion liquid membrane (ELM) process were investigated. This process consisted of three phases which are external (feed) phase, membrane phase and internal phase. The membrane phase was prepared by dissolving extractant bis(2-ethylhexyl)phosphoric acid (D2EHPA) and hydrophobic surfactant sorbitan monooleate (Span 80) in kerosene as diluents. The internal phase consisted of an aqueous solution of sulfuric acid (H_2SO_4). The important parameters governing the extraction process of dye such as stirring speed, initial dye concentration, Span 80 concentration and treat ratio (volume ratio of emulsion to external phase) were studied. The results showed that the optimum condition for 25ppm initial concentration of dye extraction are 250 rpm stirring speed, 5% (w/v) Span 80 and treat ratio 1:5. At this condition, the percentage of dye extraction, stripping and recovery were 98%, 82% and 81% respectively. Hence, continuous ELM technique is proven to be a very promising technique in industrial wastewater treatment and recovery of dye.

Keywords: Emulsion liquid membrane; continuous extractor; dye recovery; textile industry; wastewater

1.0 INTRODUCTION

Dye is the most obvious indicator of water pollution. Dye wastewater is a result of batch and continuous processes both in the dye manufacturing industries and in the dye manufacturing and dye consuming industries. Industries such as dyeing, printing, plastic, leather, paper and textile finishing are some of the sources for dye effluents. An indication of the scale of the problem is given by the fact that two percent of dyes that are produced are discharged directly in aqueous effluent [1]. There are many types of structural varieties for dyes such as, anionic which are direct, acid, and reactive dyes, cationic which are basic dyes, non-ionic which are disperse dyes, azo, diazo, anthroquinone based and metal complex dyes. Dyes such as acid, basic and direct are all water-soluble while disperse dyes have low solubility and colloidal dispersion properties. Basic dyes are commonly applied to polyester fibers, wool, silk, and acrylic fibers [2] due to the increasing development in production of synthetic fiber lately.

The presence of very small amounts of dyes in water which is less than 1 ppm for some dyes is highly visible and undesirable [3]. The dyes upset the biological activity in water bodies. They also pose a problem because they may be mutagenic and carcinogenic [4] and can cause severe damage to human beings, such as the dysfunction of kidney, reproductive system, liver, brain and central nervous system [5].

Therefore, industrial effluents containing dyes must be treated before discharged into the environment [6]. There are three main categories of treatment methods for removal of dyes which are chemical, physical and biological [7]. These processes include membrane filtration, photodegradation, adsorption, coagulation-flocculation, ion exchange, advanced oxidation, flotation, chemical reduction, ozonation, electrochemical and biological treatment. The technical and economic feasibility of each technique is determined by several factors which are dye types, wastewater composition, operation cost and generated waste products. The usage of one individual technique is not sufficient to achieve complete decolorization, therefore dye removal strategies consist of a combination of different techniques. Currently, the most widely used combination of techniques for dye removal from dye-containing industrial effluents is by physio-chemical means. Adsorption of dye stuffs on cotton fabric wastewater using semi-activated carbon has been proposed by Ozoh [8]. The problem regarding to this method is disposal approach of the spent activated carbon after removing of dye stuffs. Besides, removal and recovery

of dyes using ion exchange were investigated by many researchers such as Naim and Yehia [9] and Karcher *et al*. [10] on recovery of acid dyes from wastewater. Ahmad *et al*. [11] and Djenouhat *et al*. [12] reported that membrane separation process offered the method for increasing the removal of dyes. However, fouling membrane is the main problem for this technique. Solvent extraction or liquid-liquid extraction has been proposed by Panjit and Basu [13], Muthumaran and Palanivelu [14] and Muthumaran *et al*. [15] for both anionic and cationic dyes from wastewater. But it turns out with a lot of weaknesses.

However, ELM technique is an accessible and easy way for the removal of pollutants from wastewaters. It transports them into desired phases, where the pollutants can be concentrated by 10-100 times [16].The ELM separation technique has been regarded as membrane technology with considerable potential for a variety of applications. Many studies have been carried our using ELM for the recovery of metal ions, phenol, organic acids, cephalexin from dilute solution, aniline and bioactive materials [17]. This technique offers some advantages in comparison to common liquid–liquid extraction such as improvement of kinetics, selectivity of species to be removed and reduction of the necessary volume ratio of organic phase to aqueous feed solution. Furthermore, it is characterized by simplicity and high efficiency to dyes due to a large surface area within the emulsion globules and internal droplets for mass transfer [18]. This advanced extraction technique has a very good commercial potential.

In principal, the ELM process is a three phase dispersion system, with the primary emulsion consisting of organic and stripping phase being dispersed into the feed or effluent phase, which is the phase to be treated. For the separation of dyes even in very low concentration, the carrier is an organic soluble extractant, which selectively combines with the solutes to form a dye-carrier complex. This complex will permeate through the membrane from the outer to the inner interface. At the inner interface, the complex decomposes by the reversal of the equilibrium reaction and the dye ion is liberated into the internal phase and the regenerated carrier goes back into the membrane phase.

Demulsification is one of the critical steps in ELM to recover the membrane phase just after the extraction process. It is performed in order to separate the phases that make up the emulsion, the internal aqueous phase and the organic membrane phase. At the end of the process, the spent membrane phase can be reused and the receiving phase with enriched in the recovered solute can be recycled and recovered for solute. The recovery of membrane phase consists of organic phase, surfactant, carrier, valuable solute ion can be reuse [19].

This paper presented the continuous ELM extraction process of basic dye from liquid waste solution. Several parameters such as stirring speed, initial dye concentration, Span 80 concentration and treat ratio (volume ratio of emulsion to external phase) were studied.

■2.0 EXPERIMENTAL

2.1 Chemicals and Reagents

Methylene blue (minimum assay of 82%) was obtained from QRec, di(2-ethylhexyl) phosphoric acid (D2EPHA) (assay of 95%) was obtained from MERCK ,SPAN 80 (assay of 99%) was obtained from FLUKA, kerosene(assay of 78%) was obtained from FLUKA and sulfuric acid (95-97%) was obtained from QRec.

2.2 Experimental Procedures

The experimental scheme was classified into three parts. The first part was the formation of W/O emulsion. The second part was concerned with the investigation of the performance of the continuous ELM extraction on the removal of dye from the waste solution. The third part was demulsification.

Emulsion was prepared by emulsifying an aqueous solution of sulfuric acid (H2SO4) in an organic membrane phase which consists of the extractant bis(2-ethylhexyl)phosphoric acid (D2EHPA) and the hydrophobic surfactant sorbitan monooleate (Span 80) in kerosene as diluents by homogeniser at 12000rpm for about 10 minutes. Then, water in oil emulsion was dispersed into the dye containing external phase to form water in oil in water (double emulsion) system for extraction and stripping process to occur. During the extraction process, the sample was collected and analyzed. High voltage demulsifier was used to demulsify emulsion to recover dye in the receiving phase and recycle the liquid membrane. All experiments were performed at room temperature, 25 ± 1 °C. The same procedures were repeated for different parameters ranges as shown in Table 1.

Table 1 Parameters ranges on LS recovery using ELM process

Parameter	Range
Stirring Speed (rpm)	150, 250, 350, 450
Initial dye concentration(ppm)	5, 10, 15, 25
Surfactant concentration(%w/v)	1, 3, 5, 10
Treat ratio	1:3, 1:5, 1:7, 1:10,

2.3 Determination and Calculation

The concentration of dye in the external and internal phase after separation and demulsification was measured using a UV spectrophotometer at 650nm. The absorbance obtained from UV-vis Spectrophotometer was used to calculate the final concentrations by using the standard curve of Methylene blue dye. The performance of the ELM was measured by the following terms:

$$\%Extraction = \frac{[Dye]_i - [Dye]_f}{[Dye]_i} \times 100\% \quad (1)$$

$$\%Stripping = \frac{[Dye]_{int}}{[Dye]_{mem}} \times 100\% \quad (2)$$

$$\%Recovery = \frac{[Dye]_{int}}{2TR[Dye]_i} \times 100\% \quad (3)$$

$[Dye]_i$: Concentration of dye initial.
$[Dye]_f$: Concentration of dye final.
$[Dye]_{int}$: Concentration of dye in internal phase.
$[Dye]_{mem}$: Concentration of dye in membrane phase.
TR :Treat ratio

■3.0 RESULTS AND DISCUSSION

3.1 Effect of Stirring Speed

The effect of stirring speed was studied in the range of 150–450 rpm and the obtained results are shown in Figure 1. It was observed that increasing stirring speed from 150 to 450 rpm increases the rate of extraction. Higher stirring speeds lead to the formation of smaller sized globules, thereby increasing the interfacial area between the continuous phase and membrane phase causing an increase in the rate of mass transfer [20, 21]. The increase in agitation speed also reduces the thickness of the boundary layer at the interface of emulsion globules and internal phase. This leads to

good extraction efficiency. For the lower stirring speed (150 rpm), the extraction efficiency is low because the formation of larger emulsion globules involves a decrease in the area for mass transfer.

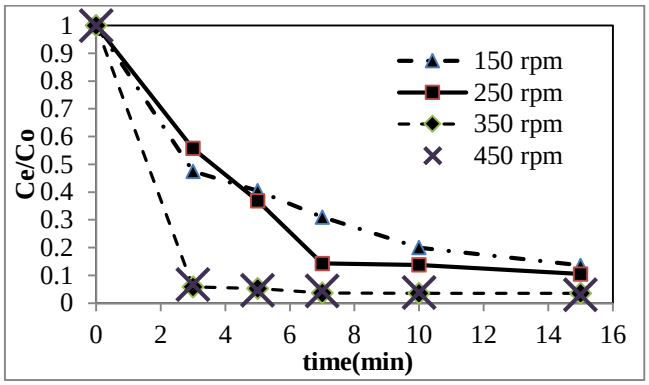


Figure 1 Effect of stirring speed on methylene blue dye extraction.in the continuous ELM process

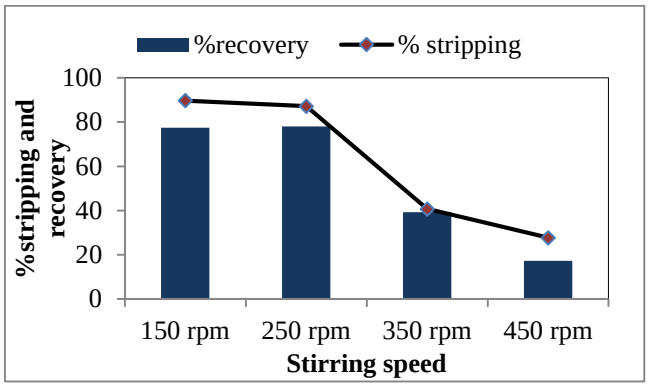


Figure 2 Effect of stirring speed on the stripping and recovery of dye in the continuous ELM process during extraction time of 15min

The performance of stripping was analysed after 15min of the extraction process. The result is shown in Figure 2 indicated that the stripping and recovery efficiency increased with an increase in stirring speed from 150 to 250 rpm. However, the stirring speed increases beyond 250 rpm causes the decreasing of stripping and recovery percentage. Increasing the agitation speed to a higher limit offers larger energy to rupture the emulsion drops compared to lower agitation speed [22]. For higher stirring speeds (350 and 450 rpm), the swelling phenomenon is expected to become notable, which resulted in greater amounts of water to permeate through the membrane causing the internal droplets to swell and coalesce. Therefore, a trade-off exists between these two effects. The breaking of swollen particles can be natural or is induced by shear force and thus allows more leakage of concentrated basic dye in the internal phase to diffuse back into the external phase [23, 24]. Thus, 250 rpm is recommended as the most appropriate stirring speed rpm due to the good stripping efficiency (78%) and excellent emulsion stability.

3.2 Effect of Initial Dye Concentration

The effect of initial dye concentration on the dye extraction is also illustrated in Figure 3. At the initial stage of extraction process within 3 minutes, it was observed that the extraction efficiency increases with the increase of methylene blue dye concentration from 5 ppm to 25 ppm. This may be attributed to an increase in the driving force, the concentration gradient, with the increase in the initial dye concentration. It has been observed that for the lower concentrations of methylene blue dye at 5 ppm, the phenomenon of swelling was expected to occur due to the low extraction performance. The transport of water from external phase to internal phase is mainly driven by the difference in osmotic pressure between the external and the internal phases [25]. By increasing the time up to 15 minutes, the extraction achieves maximum for all ranges of initial dye concentration. It is proven that dye extraction using ELM is promising for all ranges of initial dye concentration.

The influences of feed concentration of methylene blue dye on the stripping and recovery efficiency are also studied. The effect of the feed concentration of methylene blue dye in the external phase on the stripping of methylene blue dye is illustrated in Figure 4. It was observed that the stripping efficiency and recovery of methylene blue dye was not stable. The percentages stripping from 10 ppm to 20 ppm tend to decrease. This is due to the holding of some carrier–dye complex within the membrane phase which could not be stripped into the internal phase. This observation indicates instability of the emulsion. Hence, poor stripping reaction by internal phase cause small amount of dye cation to be released into internal phase. However, the stripping and recovery efficiency for 25 ppm reached 98.5% and 96.89% respectively. This indicates the stability of emulsion present in 25 ppm of initial dye. As a result, 25 ppm of initial dye is the optimum concentration for the stripping and recovery of dye solution using these operating conditions. Thus, 25 ppm of initial dye concentration is chosen for next parameter.

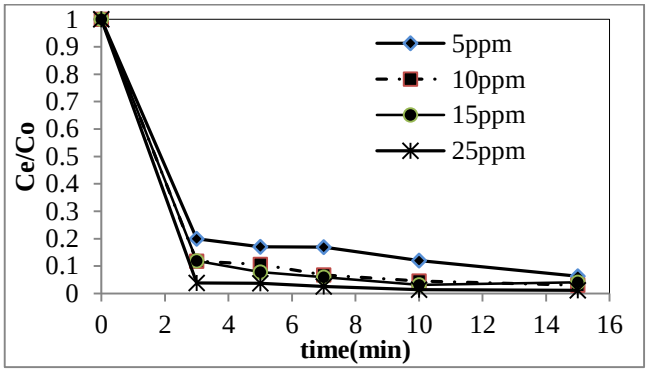


Figure 3 Effect of feed concentration of dye on the dye extraction in the continuous ELM process

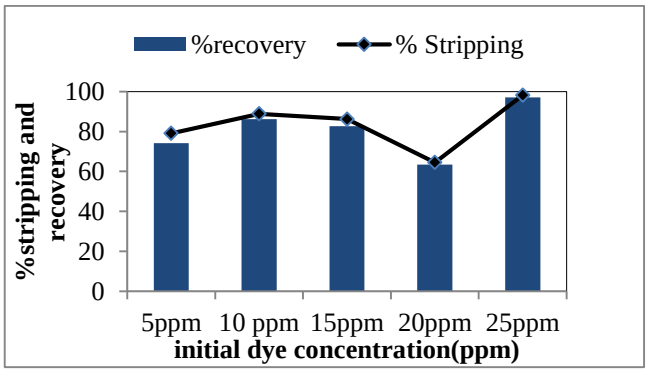


Figure 4 Effect of feed concentration of dye on the stripping and recovery of dye in the continuous ELM process during extraction time of 15min

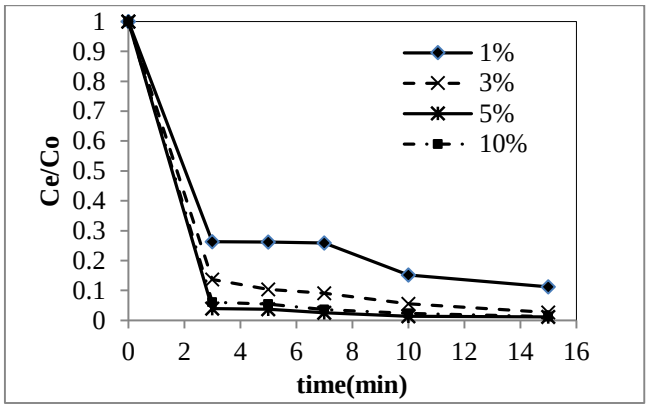


Figure 5 Effect of surfactant concentration on the extraction of dye in the continuous ELM process

3.3 Effect of Surfactant Concentration

Surfactant concentration is an important factor to an ELM system. While it ensures the stability of membrane during extraction, it also changes the mass transfer resistance [26]. It indicates that the breakup of emulsion or emulsion stability is strongly dependent on the surfactant concentration. Figure 5 represents the variation of extraction performance for various surfactant concentrations. It was observed that the extraction efficiency increases by increasing the surfactant concentration up to 5% (w/v) and the extraction rate decreases afterward. At 1 and 3% (w/v) surfactant concentration, the percentage extraction is low due to the fact that low surfactant concentration destabilize the membrane [26]. At lower surfactant concentrations (less than 3%), emulsions break easily because this amount is insufficient to surround all the internal aqueous phase. Consequently, the amount of surfactant in the membrane phase must be minimal but adequate to stabilize the emulsion. As the amount of surfactant was increased, interfacial tension between the phases decreases which favors the formation of finer droplets, thus producing more stable emulsion [24]. The increase in amount of surfactant beyond 5% (w/v) decreases the emulsion stability. Most of the surfactants tend to form aggregates in bulk of the solution above a particular concentration which is called critical micelles concentration (CMC). The surfactant aggregates act as a reservoir for water. They can promote the transport of water to the external phase or internal phase which is called swelling or breakage and ultimately affecting the stability of emulsions. Additionally, it is known that the increase in surfactant concentration leads to a higher viscosity of the W/O emulsion and causes higher mass transfer resistance and does not favor the extraction kinetics.

Figure 6 represents the effect of surfactant concentration on the stripping and recovery of dye during the extraction time of 10n minutes. It shows that by increasing Span 80 concentration from 1% (w/v) to 5% (w/v), the percentage of dye recovery increases to 91% but with further increment to 10% (w/v), the percentage drop down to 76%. Higher concentration of surfactant usually results in higher emulsion stability. At the same time, higher concentration of surfactant often causes a decrease in the transport rate of dye ions because the reaction between dye ions and carrier is inhibited by the adsorption of surfactant molecules at the reaction interface. Higher emulsion stability is also not desirable as it is needed to recover the strip phase by breaking the emulsion. Higher surfactant concentration means increasing thickness of resistance layer, which will affect the resistance on the dye transport process that leads to a decrease in the percentage of recovery and stripping. This effect is deduced by the amount of dye remaining in the external phase. Therefore, the surfactant concentration is an important factor which is beneficial to a certain limit but in excess it will destroy the process of recovery. Consequently, the optimum Span 80 concentrations was at 5% (w/v).

3.4 Effect of Treat Ratio

The treat ratio, defined as the volume ratio of emulsion phase (Vm) to the aqueous external solution (Vdye), plays a vital role in determining the effectiveness of ELM. Experiments were performed to study the influence of treat ratio on the extraction of methylene blue dye. Its variation cannot be random, because it is necessary to guarantee that the H2SO4 concentration that present in the internal phase is high enough to react with the external phase which is the methylene blue dye solution. The flow rate ratio of the dispersed phase to the continuous phase, were varied from 0.33 (Vm:Vdye =1:3) to 0.1 (Vm:Vdye = 1:10) by fixing the flow rate of the dispersed phase as 10 ml/min and changing the flow rate of the continuous phase, which is the external phase, from 30 to 100 ml/min. The effects of treat ratio on percentage extraction are shown in Figure 7. It is found that at a treat ratio of (1:5), the performance recovery of solute in external phase is high and further increase in treat ratio decrease the extraction efficiency. It is evident that the decrease of volume ratio of emulsion to the feed solution leads to a decrease in the extraction efficiency. Additionally, the influence of the volume ratio of emulsion to the external feed solution on the stability is found to be insignificant because the breakage increases very slightly with an increase of this volume ratio. An increase in the treat ratio with lower volume of external phase, 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 [27]. As a result, a higher degree of extraction was obtained. However, the lower the treat ratio means less emulsion is required to extract the solute, which is desirable from a processing point of view to ensure maximum enrichment with respect to the feed phase. The incomplete extraction of dye from the external phase would occur if the treat ratio is less than (1:5) at the present formulation of internal phase reagent.

The influence of the treat ratio of methylene blue on the stripping and recovery efficiency was also studied. The effect of the treat ratio of emulsion to external phase on the stripping of methylene blue dye is illustrated in Figure 8. It was observed that there was no significant difference appear in stripping and recovery for several treat ratios. The highest percentage of stripping and recovery was 85% for treat ratio 1:10. This is due to the stability of emulsion in that condition. Stripping percentages of treat ratio (1:5) are a bit lower than the treat ratio (1:10) .This is due to some holding of the carrier–dye complex within the membrane phase without getting stripped [24]. It is because the efficiency of stripping and recovery are almost the same, the extraction efficiency is considered to obtain the optimum parameter. Consequently, in order to ensure a good dispersion of emulsion in the feed solution and to enhance the concentration of the solute in the internal aqueous phase, the volume ratio of emulsion to the external feed solution of (1:5) was selected as the best treat ratio.

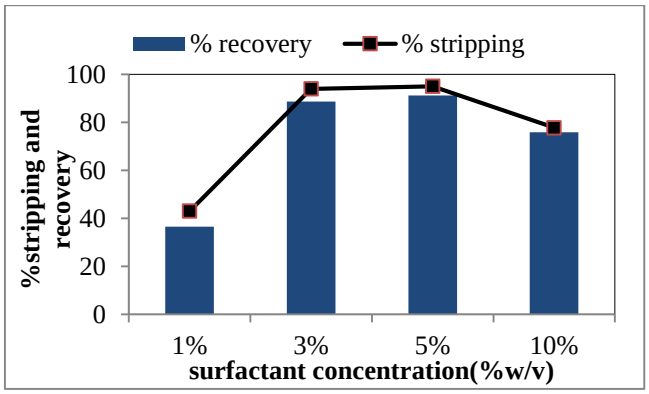


Figure 6 Effect of surfactant concentration on the stripping and recovery of dye in the continuous ELM process during extraction time of 10min

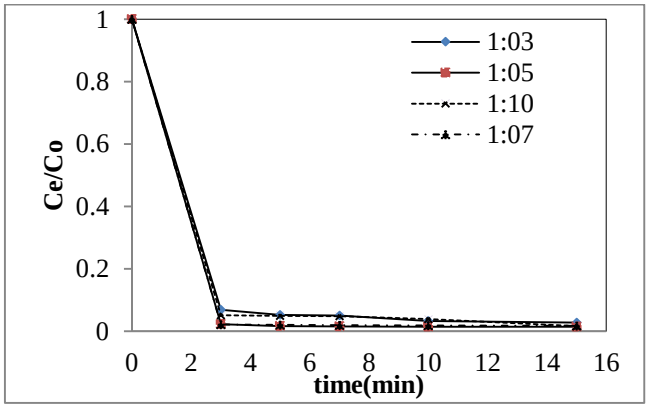


Figure 7 Effect of treat ratio on dye extraction in the continuous ELM process

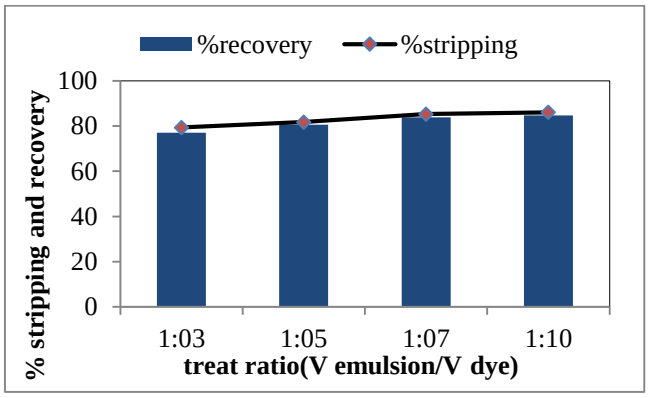


Figure 8 Effect of treat ratio on the stripping and recovery of dye in the continuous ELM process during extraction time of 15min

4.0 CONCLUSION

The feasibility of continuous ELM extraction and recovery of methylene blue was studied in this research. It was demonstrated that the continuous ELM technique was very promising in extracting and recovering methylene blue dye from simulated dye solution. The performance of the extraction column was good, and it was verified that the application of methylene blue extraction using the continuous ELM process was successful. The results showed that the optimum condition for 25ppm initial concentration of dye extraction are 15 minutes extraction time, 250 rpm stirring speed, 5%(w/v) Span 80 and treat ratio 1:5. At this condition, the percentage of dye extraction, stripping and recovery were 98 %, 82% and 81% respectively.

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