



Investigation the efficiency of emulsion liquid membrane process for malachite green dye separation from water

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ABSTRACT

The main aim of this work is to remove the cationic malachite green dye (MG) from wastewater using the emulsion liquid membrane (ELM) process. The used emulsion liquid membrane contains *n*-hexane as a diluent and a surfactant (Span 80) as an emulsion stabilizer, 0.5 M nitric acid (HNO₃) solution as a stripping phase, and MG dye solution as a feed phase. Amongst the main factors that affect the separation of dyes, treat ratio, agitation speed, surfactant concentration, agitation time, emulsification time, phase ratio, and initial dye concentration have been investigated. The results showed that almost 100% of the cationic MG dye in the feed phase were separated under the best experimental circumstances. These conditions are a 1:2 treat ratio, 350 rpm agitation speed, 2% surfactant (Span 80), 3 min agitation time, 30 s emulsification time, 1:2 phase ratio, and 20 mg/L initial dye concentration. The ELM revealed a highly efficient and straight forward method for removing cationic dye from water.

Keywords: Malachite green; Emulsion; Nitric acid; Liquid membrane; Hexane

1. Introduction

One of the major environmental problems in textile factories is discharging wastewater, including improper concentrations of dyes [1]. Even in low concentrations, the most noticeable sign that the water is polluted is the presence of dyes [2]. Dye is extremely carcinogenic and poisonous, and the possibility of water organisms causing skin irritation. So that there is noxious to human beings and the environment [3]. Due to their photostability and thermal resist biodegradation, dyes remain in the environment for unlimited time. The presence of high concentrations of dyes in water cuts off sunlight and restrict the receiving water's ability to reoxygenate. Thus, dyes may hurt the photosynthesis

process of aquatic plants and biological activity in marine life [4,5].

According to the charges of dyes, they can be categorized as cationic, anionic, or non-ionic dyes [6]. Cationic dyes are more toxic than anionic dyes, [7] such as the malachite green (MG) dyes which is mostly used in the textile industry. There are various methods for separating the dyes from wastewater, such as biological treatment methods (sedimentation, crystallization, gravity separation) and conventional treatment methods (solvent extraction, reverse osmosis, ion exchange, electrodialysis, electrolysis, and adsorption) [8–10].

In recent years, the liquid membrane (LM) technique has been known in different scientific fields, including chemical

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engineering, biotechnology, biomedical engineering, and wastewater treatment. A process known as LM is the transfer of solutes through an immiscible membrane phase from one aqueous phase to another [11,12]. LM has many advantages compared with the other separation methods, such as high selectivity, efficiency, high flux, reusability, and low energy consumption [13,14]. LMs can be categorized as bulk liquid membranes (BLM), support liquid membranes (SLM), and emulsion liquid membranes (ELM). ELM was introduced as an alternate method to solid polymeric membrane separation and liquid–liquid extraction. ELM is a double emulsion made by emulsifying two immiscible liquid phases (such as a water droplet in an oil or vice versa). The created emulsion is then distributed into a different feed phase that contains a solute that needs to be removed [15]. Emulsion liquid membrane (ELM) has been known as one of the most attractive kinds of liquid membrane technology due to key attributes, including its simplicity, good selectivity, quick extraction, and cheap energy. Conversely, emulsion instability and the challenge of demulsification following extraction are drawbacks of ELM [16]. ELM method was applied successfully for recovering various acids/bases [17], metals [18], organics [19], and dyes [20].

Emulsification, dispersion, and extraction are the three basic stages of an ELM process. An emulsion is created in the first stage by emulsifying the stripping and membrane phases. The second stage is the injection of the produced emulsion into the feed phase containing the extractable solute. In the last stage, the settling process separated the solute solution from the emulsion [21].

There are three phases in this technique: membrane phase, stripping phase, and feed phase. The membrane phase of emulsion drops contains low-viscosity organic diluents such as (hexane, heptane, kerosene, etc.), a surfactant to keep the emulsion stable, and occasionally a carrier [22]. The stripping phase contains a stripping agent, and the feed phase includes the dye solution we desire to eliminate from water [23]. Raval et al. [24] is the only research investigating MG dye separation from water. A carrier (D2EHPA) was used to assist the dyes molecules in transporting from the external feed phase (10 mg/L of MG dye) to the stripping phase (acetic acid/hydrochloric acid/sulphuric acid in the deionized water) through the membrane phase (Span 80 and D2EHPA in cyclohexane). The maximum MG removal percentage achieved was 94.99% at an emulsification time of 5 min. The highest rate of MG dye removal was 94.99%.

The novelty of the current study is using the ELM for extracting MG dye from an aqueous solution without needing to use a carrier in the membrane phase. The experiments were carried out to investigate the efficiency of the ELM method in MG dye separation from water. The effects of treat ratio, emulsification time, surfactant concentration, phase ratio, agitation speed, agitation time, and initial dye concentration were studied. The best operation conditions for the MG extraction were evaluated experimentally.

2. Materials and method

2.1. Materials

Chem-Lab NV, Belgium, supplied normal hexane. Nitric acid (HNO₃) (95% purity) was procured from Sigma-Aldrich

Company. The surfactant sorbitan mono-oleate (Span 80) was obtained from Wuhan Kemi-Works Chemical Co., Ltd. The powder of malachite green (MG) dye ((C₂₃H₂₅N₂Cl), MW 364.9 g/mol) was supplied by (HiMedia). All the chemicals were used without further treatment. The water used in the experiments was distilled water (pH = 7).

2.2. Experimental method

The carried experiments included a preparation of three solutions: the first solution (stripping phase solution) was nitric acid of 0.5 M. The second solution (membrane phase solution) was prepared by adding the surfactant (Span 80) to hexane. At the same time, the third solution (feed phase solution) was a dye solution (20 ppm) prepared by dissolving MG dye in distilled water. The stripping phase was mixed with the membrane phase using a high-speed homogenizer (Ultra-Turrax Janke & Kunkel KG) at 10,000 rpm to obtain a milky colour liquid (W/O emulsion). Then the produced mixture was mixed with the feed phase to obtain double emulsion (W/O/W emulsion) using a mechanic stirrer (Heidolph). Finally, the three-phase solutions W/O/W were added directly to a separation funnel, Fig. 1. The solutions of the treated water samples were analyzed by UV spectrophotometer (Thermo GENESYS 10S UV-Vis Electron Corporation Madison, WI 53711, USA) at a wavelength of 618 nm. All the samples were collected after 8 min of the experiment.

Fig. 2 shows the transport mechanism for the extraction of cationic MG dye in the ELM technique. Through the membrane, MG dye molecules are transferred from the feed phase to the stripping phase and concentrated in the stripping phase droplets. The stripping phase of the process contains a stripping agent (nitric acid) that combines with the solute (MG dye molecules) to produce a membrane insoluble product, increasing the mass transfer rate across the membrane phase. Diffusion and reaction serve as the motors for MG dye molecules movement.

The removal efficiency was calculated using the following equation:

$$\text{Dye removal \%} = \frac{c_0 - c_i}{c_0}$$

where c_0 : the initial concentration of MG dye in the feed phase mg/L. c_i : the concentration of MG dye in the feed phase at any time mg/L. Each experiment was repeated two times to use an average value in the calculations. All the experiments were done at ambient temperature.

3. Results and discussion

3.1. Impact of treat ratio

The treat ratio is defined as the volume ratio of the membrane to the feed phase and serves as a gauge of the system's emulsion holdup. The effect of the volume ratio of the membrane to feed phase (treat ratio) on the MG removal efficiency is shown in Fig. 3. It was observed that the removal efficiency increased from 72.56% to 99.38% with increasing the volume ratio of the membrane to feed phase from 1:4 to 1:2. The 1:2 treat ratio produced the highest removal

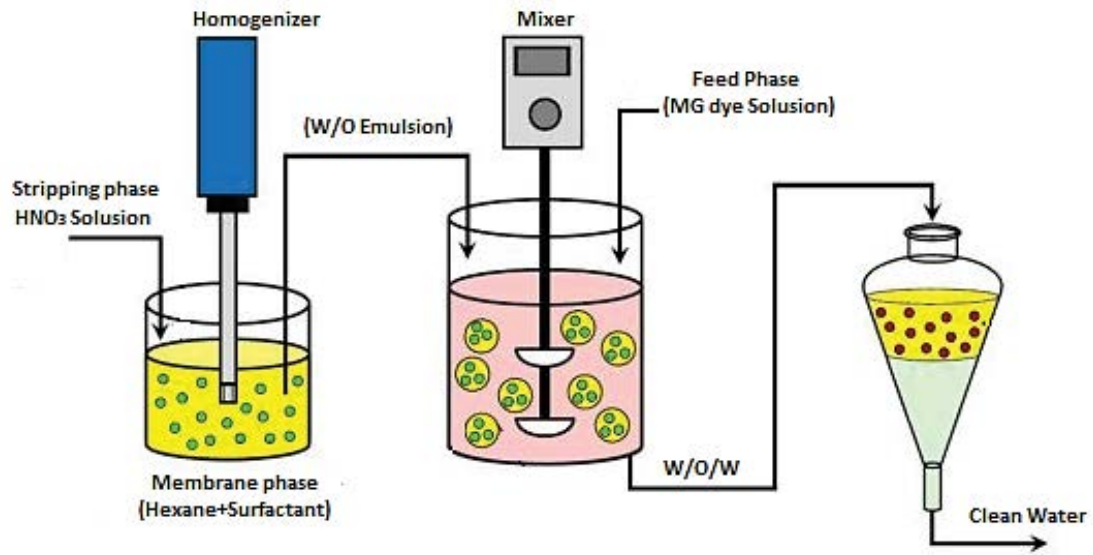


Fig. 1. Emulsion liquid membrane process for cationic malachite green dye separation.

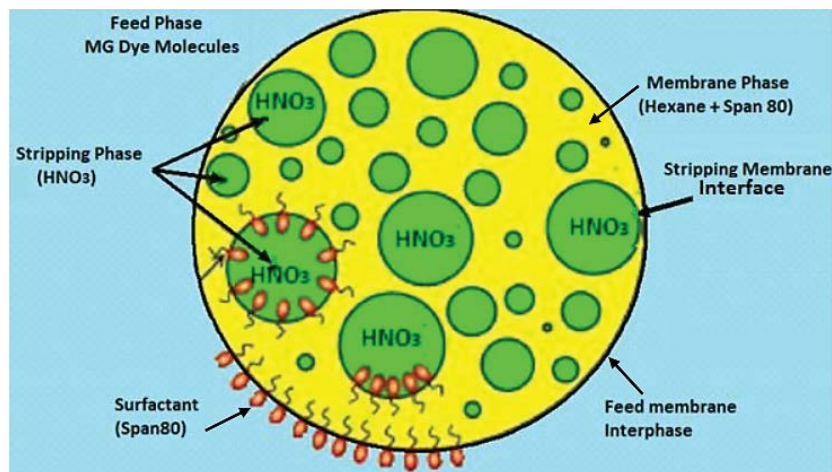


Fig. 2. An illustration of the malachite green dyes' mechanism by emulsion liquid membrane.

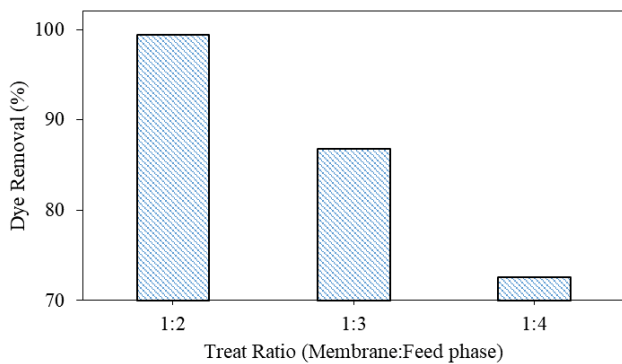


Fig. 3. Impact of treat ratio on the percentage of dye removal. Conditions: agitation speed: 350 rpm, surfactant concentration: 2%, agitation time: 3 min, emulsification time: 30 s, phase ratio: 1:2, and initial dye concentration: 20 mg/L.

percentage. The volume of the membrane was constant during the experiments, but the volume of the feed phase was changed. Emulsion holdup rises with an increase in the treat ratio. Due to a rise in the amounts of the feed phase, the emulsion's overall extraction capacity is increased or vice versa. Because an increase in the treat ratio also leads to an increase in the system's overall surface area [20,25], with a decrease in the treat ratio, the mass transfer surface area decreases, preventing more emulsion globules' growth. To obtain adequate emulsion dispersion in the dye solution, a treat ratio of 1:2 was determined to be the optimal.

3.2. Impact of agitation speed

Through the emulsion liquid membrane approach, the proportion of dye removal is significantly influenced by the agitation speed. It is observed in Fig. 4 within the

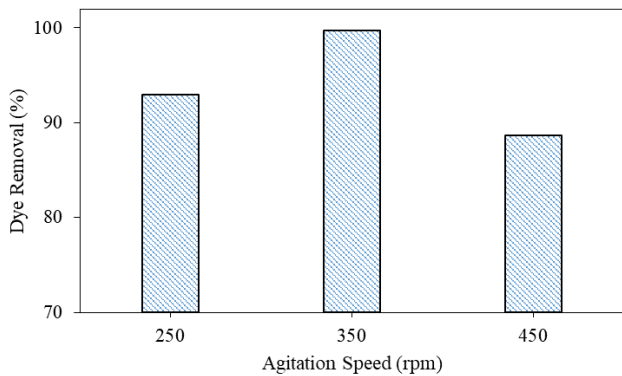


Fig. 4. Impact of agitation speed on the percentage of dye removal. Conditions: treat ratio: 1:2, surfactant concentration: 2%, agitation time: 3 min, emulsification time: 30 s, phase ratio: 1:2, and initial dye concentration: 20 mg/L.

range of 250–450 rpm that at low agitation speed 250 rpm, the emulsion globules are large, which would reduce the mass transfer area; thus, the removal efficiency decreases. Increasing the agitation speed from 250 to 350 rpm increased the percentage of dye removal. This increasing speed level would raise the mass transfer and the interfacial area to enhance the removal percentage. Raising the agitation speed higher than a critical value (350 rpm) results in the breaking of the emulsion droplets, an inefficient extraction and makes the emulsion [17,26].

The swelling phenomenon for higher agitation speeds becomes notable, resulting in more significant amounts of water permeating through the membrane, which causes swelling and coalescing for the stripping droplets [23,27]. Therefore 350 rpm was chosen as the most suitable agitation speed.

3.3. Impact of surfactant concentration

The stability and breakdown of the emulsion liquid membrane are both directly impacted by the surfactant concentration (Span 80), which is a critical component. Based on Fig. 5, the results show that at low surfactant concentrations of less than 2%, the emulsion is unstable and breaks easily, leading to poor removal because there was insufficient surfactant to decrease the interfacial tension between the oil and the water. The stability of the emulsion was greatly improved by increasing the surfactant concentration to 2%. At high surfactant concentrations, the dye removal is reduced because of the increase of the emulsion's viscosity which is not desirable in the ELM technique. Also, higher surfactant at the interface between the feed and membrane phases reduces the amount of dye molecules transferred to the stripping phase. Previous investigations also noted comparable results for this surfactant proportion [19,25,28–30].

3.4. Impact of agitation time

According to Fig. 6, the variation of MG dye removal with agitation time between membrane and feed phases indicated that increasing the agitation time increases the

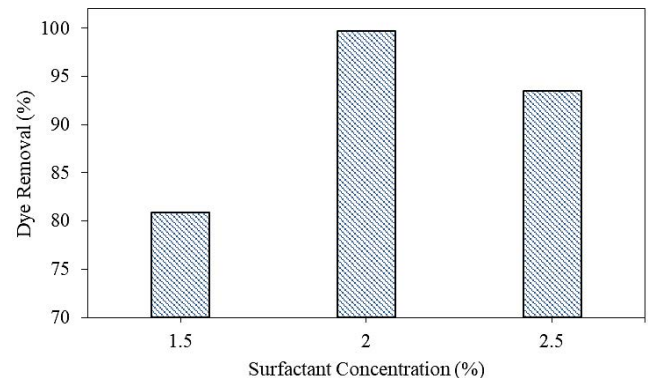


Fig. 5. Impact of surfactant (Span 80) concentration on the percentage of dye removal. Conditions: treat ratio: 1:2, agitation speed: 350 rpm, agitation time: 3 min, emulsification time: 30 s, phase ratio: 1:2, and initial dye concentration: 20 mg/L.

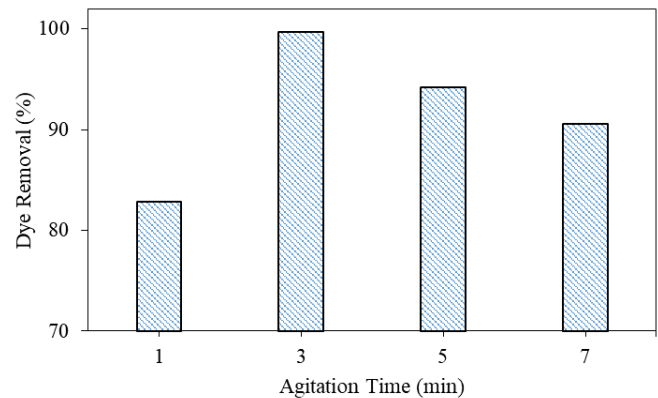


Fig. 6. Impact of agitation time on the percentage of dye removal. Conditions: treat ratio: 1:2, surfactant concentration: 2%, agitation speed: 350 rpm, emulsification time: 30 s, phase ratio: 1:2, and initial dye concentration: 20 mg/L.

percentage of dye removal until 3 min due to the contact area for mass transfer which was increased because of the reduction in the size of the globules. After 3 min, the dye removal decreased over time; a further increase in the agitation time resulted in the rupture of the emulsion liquid membrane leading to an out flow of MG dye extracted into the feed phase. It was also observed by Mortaheb et al. [31]. Thus, 3 min agitation time is needed to produce the most stable emulsion.

3.5. Impact of emulsification time

The preparation time for the emulsion, or “emulsification time”, is another parameter that has a considerable impact on the stability of the W/O emulsion and, consequently, the effectiveness of the extraction process. It is one of the most critical in the emulsion liquid membrane technique. Fig. 7 shows the effect of emulsification time on the dye for three different emulsification times (15, 30, and 45 s).

At low emulsification times (insufficient emulsification time), the removal percentage was low due to the formation of large droplets that can coalesce quickly and induce

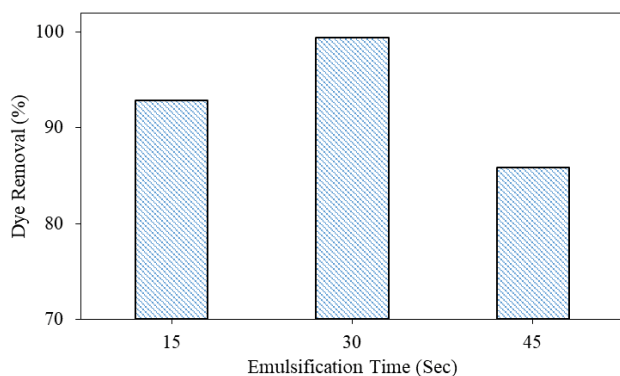


Fig. 7. Impact of emulsification time on the percentage of dye removal. Conditions: treat ratio: 1:2, agitation speed: 350 rpm, surfactant concentration: 2%, agitation time: 3 min, phase ratio: 1:2, and initial dye concentration: 20 mg/L.

the membrane's film to break. This can be explained by the internal phase's shear, which means a small transfer surface area. For higher than 15 s. It was found that the removal percentage rose as a result of the high internal shearing, which produced a large number of tiny droplets and encouraged their diffusion into the feed phase [32]. In addition, any further increase in emulsification time beyond 30 s decreases the removal percentage [33]. Therefore, a suitable emulsification time might be 30 s, a more effective time to increase the removal of MG dye and simultaneously minimise emulsion swelling.

3.6. Impact of phase ratio

Phase ratio refers to the volume ratio between the membrane and stripping phases. It is an important parameter in the emulsion liquid membrane process. To investigate the influence of the volume ratio of the membrane to stripping phases, different experiments were performed with constant 5 mL of membrane phase and HNO_3 volume from 5, 10, and 15 mL (i.e., M/S = 1:1, 1:2 to 1:3). The MG dye removal rate increases with an increase in the amount of stripping phase (HNO_3) in the emulsion. Since the phase ratio increases, the emulsion barrier's durability and resistance to breaking improve. So, by raising this ratio, we anticipate having a more stable emulsion. Fig. 8 illustrates the effect of the stripping phase (HNO_3) amount on the dye removal percentage.

On the other hand, a further increase in the membrane phase's volume to the stripping phase's volume produces a more viscous mixture. Therefore, the amount of extracted MG dye is decreased because of insufficient membrane volume, which prevents the stripping agent droplet from being completely entrapped. As a result, the stripping agent droplet then tends to seep outside the emulsion bubble into the feed phase [19,30]. Increasing the stripping phase means increasing the amount of HNO_3 , which resulted in losing the surfactant capabilities of Span 80 and consequently decreasing the dye removal efficiency.

3.7. Impact of initial dye concentration

The effect of initial dye concentration on dye removal is illustrated in Fig. 9. Due to an increase in driving force, it

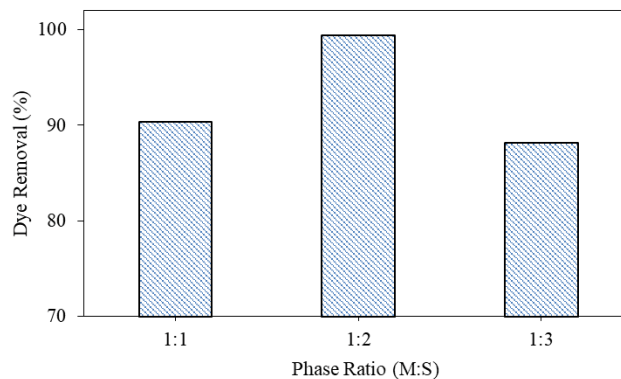


Fig. 8. Impact of phase ratio of the membrane to stripping phase (M:S) on the percentage of dye removal. Conditions: treat ratio: 1:2, agitation speed: 350 rpm, surfactant concentration: 2%, agitation time: 3 min, emulsification time: 30 s, and initial dye concentration: 20 mg/L.

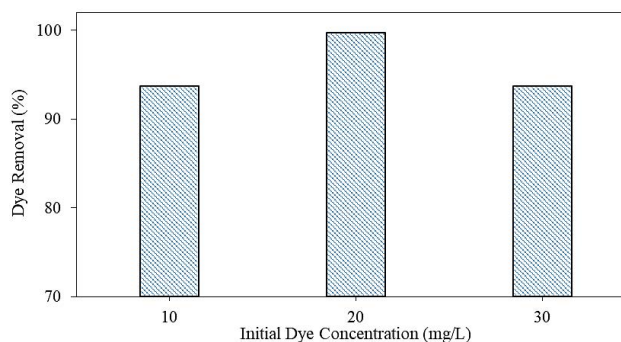


Fig. 9. Impact of initial dye concentration on the percentage of dye removal. Conditions: treat ratio: 1:2, agitation speed: 350 rpm, surfactant concentration: 2%, agitation time: 3 min, emulsification time: 30 s, and phase ratio: 1:2.

was found that the removal efficiency increased when the MG dye concentration rose from 10 to 20 mg/L. The swelling phenomenon was expected at the lower concentrations of MG dye [28]. The difference in osmotic pressure between the feed and stripping phases mainly drives water transport from the feed to the stripping phases. The removal percentage from 20 to 30 mg/L tends to decrease due to the instability of the emulsion; this may be brought on by membrane saturation and a smaller effective membrane area. The initial MG dye concentration was taken at 20 mg/L to increase the interfacial area available for mass transfer [34,35].

4. Conclusion

The goal of this work was the removal of malachite green dye from an aqueous solution of water. The results showed that the prepared ELM using nitric acid, hexane, and Span 80 could quickly separate the existing cationic MG dye in the feed phase. The effects of treat ratio, agitation speed, Span 80 concentration, agitation time, phase ratio, emulsification time, and initial dye concentration were studied. The most effective ELM formulation for this study was discovered at a treat ratio of 1:2, agitation speed of 350 rpm, 2%

of surfactant concentration, 3 min of agitation time, phase ratio of 1:2, and emulsification time of 30 s with an extraction efficiency of almost 100%. The results showed that the ELM process was a very attractive method for separating cationic dyes from water because it is a highly effective, simple, and quick technique.

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