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Application of Used Cooking Oil Based Emulsion Liquid Membrane for Rhodamine B Extraction

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Abstract

Used cooking oil is utilized as a nontoxic and environmentally friendly organic diluent without the need for complicated pretreatment. Cationic dye rhodamine B was chosen as a sample because of its ability to decolorize water and its use as a base material in toxic, carcinogenic, and mutagenic textile dyes. This study investigates the extraction of rhodamine B from water using cooking oil-based Emulsion Liquid membrane (ELM) methods. This approach addresses issues associated with petroleum-based organic diluents commonly used in ELM processes. The research aimed to evaluate the effect of rhodamine B concentrations ranging from 5 to 20 ppm and D2EHPA concentrations from 0.5 to 2.5% on extraction efficiency. The emulsion was prepared using used cooking oil as the diluent, Span 80 as the surfactant, D2EHPA as the carrier, and HCl as the internal phase. The concentration of rhodamine B in the external phase was determined using a UV-Vis Spectrophotometer. Both rhodamine B concentration and D2EHPA concentration significantly affected extraction efficiency. The highest extraction efficiency of 92.83% was achieved with a rhodamine B concentration of 15 mg/L and a D2EHPA concentration of 2.5% (v/v).

Keywords: emulsion liquid membrane, used cooking oil, rhodamine B, D2EHPA, extraction.

1. Introduction

The textile industry is a significant sector in Indonesia, contributing substantially to both export and the country's GDP. Notabley, there was significant growth in production within the textile industry during the second quarter of 2021 (Uchi et al., 2022). The growth rate of 7.07% per year was higher compared to the first quarter of 2019, which saw a growth rate of 4.45%. This expansion in the textile sector correlates directly with the increasing volume of textile waste generated. Textile waste, primarily liquid waste, arises during the dyeing process when chemicals and water are used to clean textiles. Improper disposal of textile dye waste in rivers can harm aquatic ecosystem because of its persistence, potential conversion into cancer-causing substances, and limited degradation by aerobic processes (Haryono et al., 2018). Textile dyes containing metal ions such as chromium, copper, nickel, mercury, cadmium, and lead are particulatly concerning because of their carcinogenic properties and inability to degrade naturally (Kumar et al., 2019a).
Dyes may be categorized into anionic and cationic types (Panda et al., 2021). Rhodamine B, a positively charged artificial dye belonging to the xanthenes group (Salsabila et al., 2022), is a crimson crystalline powder with a molecular weight of 479 g/mole. Solutions with high concentrations exhibit a purplish-red tint, while those with lower concentrations are brilliant-red tint and fluorescent (Saigl, 2021). Rhodamine B is a commonly used in the production of food items, textiles, paper, printing materials, and pharmaceuticals (Ahmad et al., 2021). Despite its widespread used, rhodamine B poses environmental concerns because of its persistence, potential toxicity, and adverse effects on aquatic ecosystems (Yusuf et al., 2022).

Various methods exist for removing dyes from textile waste, including coagulation and flocculation (Le et al., 2020), biological purification (Kanagaraj et al., 2015), electrochemical purification (Yuniarti and Widayatno, 2022), electrocoagulation (Wani et al., 2022), electrocoagulation via ion exchange (Joseph et al., 2020), adsorption (Permatasari, Sutriah & Khotib, 2023), catalytic ozonation (Chen et al., 2024), and filtration using membranes (Hube et al.,

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2020). Adsorption has drawbacks including short solvent life, low capacity, and high costs with pure adsorbents (Kumar et al., 2019a). Catalytic ozonation alone is insufficient for decomposing by-products because of difficulties in the decomposition process, low ozone reactivity, and high ozone production costs (Rame et al., 2017). Conventional separation techniques have high operating costs, expensive materials, are time-consuming, have low efficiency, and geneate secondary pollutants (Rosly et al., 2020).

Emulsion Liquid Membrane (ELM) is a technique for separating emulsions that are immiscible with the feed solution (Sujatha et al., 2021a). ELM can invlove water-in-oil-in-water (W/O/W) or oil-in-water-in-oil (O/W/O) emulsions (Kumar et al., 2019a). An emulsion is a three-phase dispersion system consisting of the membrane phase, internal phase, and external phase (Meilinda et al., 2021). ELM was first used by Li in 1968 for isolating hydrocarbons (Shokri et al., 2020). ELM technology has been successfully applied to separate phenol compounds and their derivatives, such as nitrophenol with 98% efficiency, Hg (II) ions, heavy metals, and hydrocarbons in water treatment, pharmaceutical, and electrolytic industries (Admawi and Mohammed, 2024), removal of malachite green dye (Raval et al., 2022) and acid orange (Ferdenache et al., 2024), separation of Ag metal ions (Djunaidi et al., 2021), uranium, lactic acid, succinic acid, citric acid, acetic acid, as well as penicillin G (Kumar et al., 2019b). Currently, ELM is a promising alternative for dye separation because of its simplicity, energy efficiency, high separation factor (Sulaiman et al., 2020), low chemical consumption, and effectiveness in removing toxic pollutants (Purtika et al., 2022a).

The ELM mechanism invloves two immiscible liquid: the membrane phase, consisting of surfactant, diluent, and carrier, and the internal phase (Othman et al., 2019). An emulsion is formed by combining the membrane phase with the internal phase. Cationic dye ions in the external phase are transferred to the emulsion phase with the help of a carrier (Zereshki et al., 2018). The emulsion phase is then separated from the external phase using a separating funnel and undergoes demulsification, breaking down and returning to the membrane phase (Purtika et al., 2022a). Carriers ficilitate the transport of components by selectively reacting with solutes and having reversible properties (Ge et al., 2018). Di-(2-Ethyl Hexyl) phosphoric acid (D2EHPA) was selected as the carrier because of its anionic properties, making it suitable for extracting the cationic rhodamine B. The choice of diluent impacts emulsion stability, with used cooking oil being used as because of its potential as an ecologically acceptable substitute for synthetic solvents derived from hydrocarbons, promoting a greener ELM (Sujatha et al., 2021b).

Few studies have investigated the efficiency of using used cooking oil as a diluent for removing rhodamine B, specifically concerning extraction performance and membrane stability. Utilizing used cooking oil in the ELM process can transform this food waste into a valuable resource. This research aims to explore the effects of D2EHPA and rhodamine B concentrations on extraction performance, breakage level, and membrane stability.

2. Methodology

2.1. Materials

The Kolb brand non-ionic surfactant Sorbitan monooleate, also known as Span 80, was used to stabilize emulsions. Used cooking oil served as a diluting agent. The internal phase consisted of 37% hydrogen chloride solution from Merck. Di-(2-Ethyl Hexyl) phosphoric acid (D2EHPA 99%), produced by Eagles, was used as the carrier. Rhodamine B, supplied from MAE, was used as the cationic dye in the external phase. Figure 1 shows the molecular structures of D2EHPA, rhodamine B, and Span 80.

2.2. Emulsion Preparation

The membrane phase, a water-in-oil (W/O) emulsion, was prepared in a 500 mL beaker at ambient temperature, using the specified mixing speed and duration. The organic phase was prepared by dissolving Span 80 (surfactant) and D2EHPA (carrier) in used cooking oil (diluent). The internal phase was prepared by dissolving a suitable quantity of hydrogen chloride. The internal phase was gradually added to the organic phase while continuously mixing with a homogenizer. The resulting water-in-oil (W/O/W) emulsion was then added to the external phase containing rhodamine B, mixed for a specific time, and allowed to separate. The sample was then analyzed for rhodamine B concentration.

2.3. Analysis

The concentration of rhodamine B in the external phase was measured using a Thermo Scientific Genesys 10 UV-Vis

Spectrophotometer at a wavelength of 557 nm, using a calibration curve. The emulsion's stability was monitored, and the efficiency of the extraction process was calculated. Table 1 displays the operating settings for the ELM, whereas Figure 2 illustrates the extraction of rhodamine B using the ELM technique. Equations (1) through (4) are used to calculate the extraction efficiency, degree of breaking, and stability of the membrane.

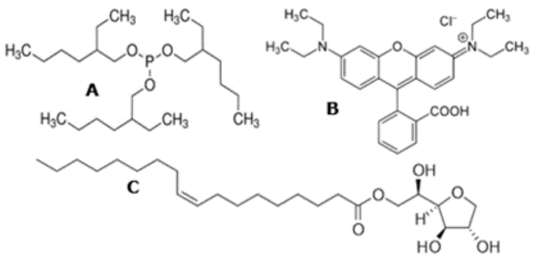

Figure 1. Structural formula A: D2EHPA, B: Rhodamine B, C: Span 80

$$E\ (\%) = \frac{C_{in} - C_{out}}{C_{in}} \times 100\% \quad (1)$$

Emulsion breakage is found by Equation 2.

$$\varepsilon = \frac{V_s}{V_i} \quad (2)$$

Where,

$$V_s = V_{eks} \frac{10^{pH_0} - 10^{pH}}{10^{pH} - C_{H^+}^{int}} \quad (3)$$

Remark:
E (%) : extraction
C_{in} : initial concentration of external phase (ppm)
C_{out} : final concentration of external phase (ppm)
ε : breakage
V_s : final volume of internal phase (mL)
V_i : initial volume of internal phase (mL)
pH_0 : initial pH of the external phase
pH : final pH of the external phase
$C_{H^+}^{int}$: initial concentration of internal phase (M)

Table 1. ELM operating conditions

No	Variable	Mark
1.	Diluent	Used cooking oil
2.	Surfactant	Span 80
3.	Carrier concentration, D2EHPA (%v/v)	0.5; 1; 1.5; 2; 2.5
4.	Internal phase	HCl
5.	Internal phase volume	8.333 mL
6.	External phase volume	100 mL
7.	External phase concentration (ppm)	5; 10; 15; 20; 25
8.	Emulsification time	25 minutes
9.	Homogenizer speed	1200 rpm
10.	The volume ratio of the internal phase to the organic phase	1:3
11.	The volume ratio of the membrane phase to the external phase	1:3
12.	Extraction speed	200 rpm
13.	Extraction time	7 minutes

2.4. Demulsification

The separation of the emulsion and external phase is achieved using gravity and settling in order to retrieve the membrane phase. The membrane was placed into a 100 mL beaker and heated to a temperature of 60°C for a duration of 10 minutes. The application of heat causes a fraction of the membrane phase to split into an organic phase and a transparent liquid internal phase. The calculation of stability may be determined using the Equation (4).

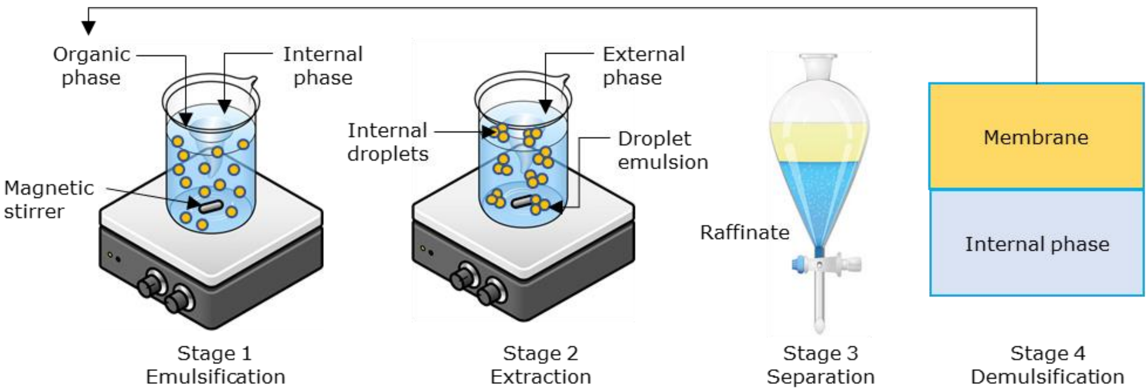

Figure 2. ELM process

$$\text{Stability } (\%) = \frac{V_t - V_w}{V_t} \times 100\% \tag{4}$$

Remark:
Vt : initial membrane volume
Vw : volume of internal phase clear fluid

2.5. Statistical Analysis

The rhodamine B solution's concentration was measured using a Thermo Scientific Genesys 10 UV-Vis Spectrophotometer set at a wavelength of 557 nm. Data were analyzed using ANOVA to determine statistical significance. Implementing Two-Factor with Replication using an α value of 0.05. The factor of D2EHPA concentration (%v/v) is composed of five levels: 0.5, 1.0, 1.5, 2.0, and 2.5. The concentration of rhodamine B is measured in parts per million (ppm) and is categorized into four levels: 5, 10, 15, and 20. The treatment combination consists of 5 levels and 4 repeats, resulting in a total of 60 experimental units.

3. Results and Discussion

3.1. Effect of Rhodamine B Concentration

Figure 3 displays the outcomes of the rhodamine B extraction process conducted in a separating funnel. Rhodamine B is capable of dissolving in oil due to its hydrophobic nature, allowing it to diffuse inside the membrane and reach Cl- ions present in the internal phase. Meanwhile, protons disperse into the surrounding phase in order to preserve the overall neutrality of the solution. Triglycerides, the primary element in used cooking oil, serve as a complexing agent that binds dyes and enhances the permeability of solute transfer (Kumar et al., 2019a).

Transport in ELM involves the use of carriers from the exterior phase to the internal phase, which occurs via the membrane phase (Figure 4). The carrier facilitates the dissolution of the external phase into the organic phase, allowing it to enter the internal phase by a reversible process (Shokri et al., 2020). The extraction process occurs in four distinct steps: 1) Rhodamine B's diffusion over the outer phase boundary layer. 2) Partitioning of rhodamine B between the membrane phase and the exterior phase. The study investigates the process of rhodamine B diffusion via a liquid membrane. 4) Process of removing the external layer to access the interior phase. Reaction (7) may be used to identify the cation exchange mechanism, which involves the creation of a complex between the exterior phase and the carrier across the membrane.

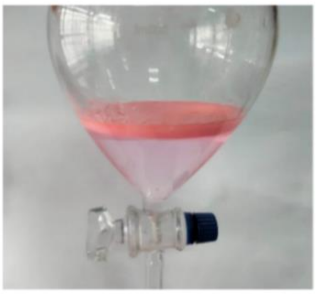

Figure 3. Results of rhodamine B extraction in a separating funnel

$$RhB^{+}_{aq} + [RH]_{2org} \rightarrow [RhB(R.HR)]_{org} + H^{+}_{aq} \tag{5}$$

Where $[Rh]_{2org}$ is the carrier and $[RhB(R.HR)]_{org}$ is the complex of the organic phase. If the internal phase is hydrogen chloride, then:

$$[RhB(R.HR)]_{org} + H^{+}Cl^{-}_{aq} \rightarrow [Cl^{-}RhB^{+}]_{org} + [RH]_{2org} \tag{6}$$

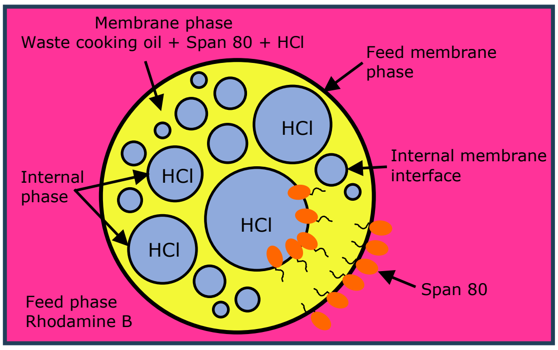

Figure 4. Simple transport mechanim of rhodamine B to the membrane phase and internal phase

Carrier concentration plays a crucial role in extraction by serving two major functions: 1) facilitating the transfer of dissolved molecules from the exterior phase to the internal phase by generating a complex, and 2) enhancing the stability and selectivity of ELM (Emulsion Liquid Membrane) by raising the viscosity of the membrane (Zereshki et al., 2021). Furthermorse, the diffusion of rhodamine B is influenced by the concentration gradient between the internal and exterior phases (Sujatha et al., 2021). Figure 5 displays the relationship between the concentration of rhodamine B (measured in parts per million) and the absorbance (measured in angstroms) on a calibration curve. The correlation coefficient (R^2) for this curve is 0.9974. The

Equation (8) represents the known calibration plot.

$$\text{Final concentration(ppm)}=\frac{\text{Å}-(-0.0249)}{0.0945} \qquad (7)$$

Where Å is absorbance.

Figure 6 illustrates the efficiency of extracting rhodamine B at different concentrations of both rhodamine B and D2EHPA as the carrier. The extraction efficiency improved as the concentration of the external phase rose from 5 ppm to 15 ppm, reaching a peak performance of 92.83% at 15 ppm. However, the extraction efficiency dropped at a concentration of 20 ppm, except for the D2EHPA concentration of 0.5% (v/v). An increasing concentration of the external phase inside the internal phase leads to a leftward shift in the equilibrium. This leads to an elevation in the concentration of complex molecules on the surface of the membrane.

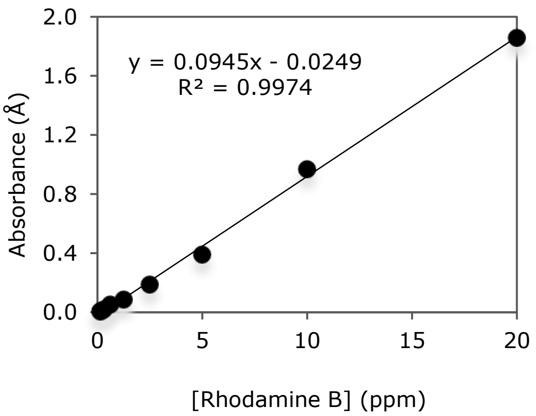

Figure 5. Rhodamine B extraction calibration curve

Raising the concentration of rhodamine B in the internal phase leads to an increase in the diffusion rate on the membrane surface, resulting in improved extraction performance (Qureshi et al., 2018). The decline in extraction efficiency at a concentration of 20 ppm can be attributed to the carrier's inability to adequately transport all the molecules from the external phase, as well as the quicker saturation of the droplets in the internal phase within the emulsion. This leads to a longer diffusion pathway and a reduction in overall effectiveness (Purtika et al., 2022b). This decline is primarily caused by the saturation of the internal phase with the carrier and the limited membrane surface available for efficient transport owing to the creation of a concentration boundary layer at the membrane interface. As a result, transport is reduced with increasing concentrations of the external phase.

Furthermore, the elevated concentration of the external phase enhances the ionic strength of the solution, hence diminishing the external phase's ability to form complexes (Isanejad et al., 2016). The findings align with a study previously used the Supported Liquid Membrane (SLM) technique to recover rhodamine B (Isanejad et al., 2016). Extraction efficiency of rhodamine B was 80% at a concentration of 50 mg/L, and increased to 95.3% at a concentration of 100 mg/L owing to efficient interaction beL and the external phase and the carrier. The extraction efficiency increased with increasing rhodamine B concentration, reaching 92.3% at a concentration of 150 mg/L. This was primarily caused by the carrier concentration being inadequate to transport molecules from the external phase. Additionally, the membrane became saturated with carrier molecules and the membrane area decreased due to the formation of a concentration boundary layer at the membrane interface. This boundary layer facilitated the retention of the feed, thereby reducing the transport efficiency at higher concentrations.

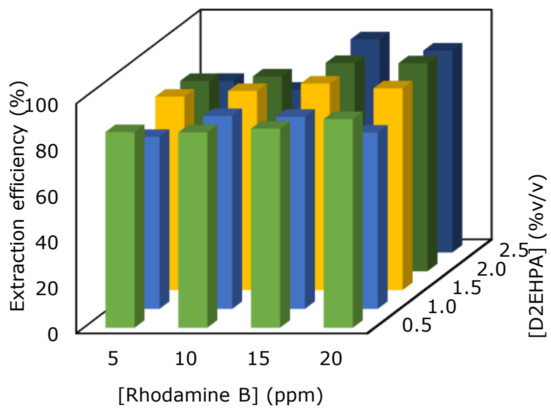

Figure 6. Rhodamine B extraction performance

Figure 6 demonstrates that the extraction performance declined when the carrier concentration was 1% (v/v), but improved as the carrier concentration increased to 1.5% (v/v). The extraction performance reached its peak at a carrier concentration of 2.5% (v/v), achieving a performance of 92.83%. The decline in the extraction of rhodamine B dye at a concentration of 1% (v/v) was correlated with an elevation in the viscosity of the membrane phase. Elevating the viscosity of the membrane hampers the movement of rhodamine B dye and the creation of complexes on the membrane, resulting in the generation of larger emulsion sizes and impeding the pace at which rhodamine B dye transfers from the external phase to the membrane phase. Subsequently, the osmotic pressure gradient intensifies between the internal and exterior phases, leading to an accelerated influx of water from the external phase into the internal phase, resulting in the dilution of the internal phase (Raval et al., 2022). The extraction performance improved as the carrier concentration increased from 1.5% (v/v). This increase in carrier concentration in the membrane phase led to an increase in interfacial tension, resulting in higher viscosity of the membrane phase and the formation of larger emulsion droplets. These larger droplets could promote permeation swelling, thereby enhancing the extraction process (Mohammed et al., 2020).

Augmenting the concentration of the external phase in the membrane phase results in the presence of an ample quantity of carrier molecules for complex formation. The number of complexes between D2EHPA-rhodamine B generated in the membrane phase and external phase at the external phase contact also shown an increase (Zereshki et al., 2021). The same occurrence was documented in the retrieval of rhodamine B by the use of the Supported Liquid Membrane (SLM) technique (Isanejad et al., 2016). At a carrier concentration of 20%, the extraction efficiency was 67.6%. However, at a carrier concentration of 35%, the extraction efficiency increased to 95.3% owing to its enhanced capacity to absorb and transport larger molecules in the feed. Moreover, when the carrier concentration reached 50%, the extraction rate reduced to 92.3% as a result of the increased viscosity of the membrane, thus leading to an increase in membrane resistance.

Table 2. Anova results of rhodamine B extraction with ELM

Source of Variation	SS	df	MS	F	F crit
[D2EHPA]	523.27	4	130.82	4992.18	2.61
[Rodhamin B]	681.81	3	227.27	8672.93	2.84
Interaction	927.53	12	77.29	2949.64	2.00
Error	1.05	40	0.03		
Total	2133.65	59			

The results of the Anova are shown in Table 2. Two-Factor with Replication analysis using a 5% level of significance, shown in Figure 7, indicate that the D2EHPA concentration, rhodamine B concentration, and their interaction significantly affect the extraction of rhodamine B using the ELM method. This means that these variables and their interaction have a significant impact on the extraction performance.

3.2. Breakage Level

Excessive carrier concentration leads to the rupture of the membrane, resulting in osmotic swelling (Laki and Kargari, 2016). Figure 7 illustrates that the rate of breaking for the membrane emulsion is below 20%. The minimum amount of breakage observed was 0.890% when the carrier concentration was 0.5% (v/v). The highest amount of breakage seen was 18.44%. Meanwhile, the breakage level at the highest extraction, 92.83%, was observed at a value of 8%. It was reported that the instability of the emulsion and the leakage of the internal phase solution to the external phase are produced by the higher concentration and volume of carrier in the membrane phase (Mohammed et al., 2020). The breakage was caused by the interface characteristics shown by the complexes and carriers generated with solute molecules, which led to the loss of reagents from the internal phase (Kumar et al., 2019a).

3.3. Membrane Stability

The stability of the emulsion is a crucial component that affects the process of permeation. Membrane demulsification results in a reduction in the extraction percentage because of the escape of separated solutes from the internal water phase to the exterior water phase (Dâas and Hamdaoui, 2010). The stability of an emulsion is a result of viscoelastic surfactants forming a film at the interface, which acts as a barrier to prevent the aggregation and coalescence of emulsion droplets (Kumar et al., 2019a). The settings that result in the best extraction performance provide a stability of 60%.

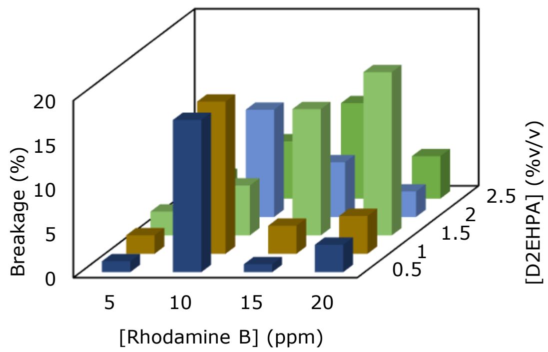

Figure 7. Level of breakage to rhodamine B extraction

This outcome may be asttributed to the permeation of the membrane layer and the expansion of the emulsion, leading to the release of the receptor agent into the external phase during the extraction process. The swelling of the emulsion occurs due to the trapping of the external phase inside the internal phase, which is generated by the recurrent dispersion of droplets of the internal phase. One additional factor that contributes to the instability of the emulsion is the disruption of the internal phase droplets due to shear pressures during extraction (Kumar et al., 2019a).

4. Conclusion

This study demonstrated that used cooking oil can be effectively utilized as a diluent in ELM processes for extracting rhodamine B. The ELM was prepared using D2EHPA as a carrier, SPAN 80 as a surfactant, and used cooking oil as the diluent. The ELM method demonstrated the effective extraction of rhodamine B dye, with a 92.83% extraction efficiency. This was achieved with a membrane breakage level and stability of 8.0% and 60%, respectively. The highest extraction efficiency of 92.83% was achieved with 15 ppm rhodamine B and 2.5% D2EHPA. The study concluded that an organic emulsion made by the used cooking oil green diluents may be an effective way to eliminate rhodamine B dye from aqueous solutions. However, a thorough investigation is required to optimize factors that impact the effectiveness of extraction and stability of emulsions. A water treatment technology based on a lot of dye waste with lesser environmental concerns is worth pursuing economically.

Author contribution statement

Conceptualisation, N.H.,; Formal analysis, A.A.K,; Investigation, H.P.,; Project administration, A.A.K,; Supervision, N.H.,; Writing – original draft, H.P.,; Writing – review and editing, N.H.

Data availability statement

The data presented in this study are included in the article.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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