Stability Study of Emulsion Liquid Membrane via Membrane Breakage on Lactic Acid Extraction from Aqueous Solution Using TOA Avinash Thakur¹, Parmjit Singh Panesar², Manohar Singh³, Anil Kumar^{4*}

 ^{1,4}Research Laobratory-III, Department of Chemical Engineering, Sant Longowal Institute of Engineering and Technology, Longowal 148106, India. Email*: <u>anilchaudharyaec03@gmail.com</u>
 ²Biotechnology Research Laboratory, Department of Food Engineering and Technology, Sant Longowal Institute of

Engineering and Technology, Longowal 148106, India.

³Global Group of Institutes, Amritsar, Punjab India.

Tel.: +91 8427173309

Abstract. The main aim of this current research work is to examine about the "Emulsion Liquid Membrane" (ELM) stability via membrane breakage (%) for lactic acid (LA) extraction from the feed phase. This research article mainly discusses about the detailed experimental study of the various process parameters affecting the membrane breakage and its performance. The ELM formulation is done by using organic phase constituents containing extractant (tri-octylamine (TOA)), diluents i.e., hexane and surfactant Span 80), and internal phase (0.1 M sodium carbonate solution). The optimal numbers of several process variables for gaining a stable ELM are as follows: emulsification time: 20 min, emulsification speed: 2000 rpm, span 80 concentrations: 4% (v/v), internal phase concentration: 0.1 [M], extractant (TOA) concentration: 10%, phase ratio: 1.0 (v/v), treat ratio: 2 (v/v), and stirring speed: 200 rpm. The percentage (%) lactic acid efficiency stripped into the ELM with the lowest membrane breakage of 4.5 % was found 95 %. Overall, the findings of this research related to ELM formulation having good stability suggest that this ELM based technology for lactic acid extraction from aqueous feed phase has great potential.

KEYWORDS: Stability; Parameters; Emulsion liquid membrane; Span 80; Hexane

1. Introduction

Since last few years, membrane practices are branded as a possible substitute to the other separation technologies (such as adsorption, ion-exchange, precipitation, electro coagulation, liquid-liquid extraction, and solid phase extraction) for treating and recovering the solute molecules from the various aqueous waste systems [1]. However, these other separation technologies/processes are ineffective, expensive, complex process, long extraction time, and risk of secondary pollution. Therefore, researchers are continuously exploring new and effective separation and purification methods based on emulsion liquid membrane (ELM) and also modification/developments in this technique are going on to make it more energy efficient, economic, and time saving [2,3,4,5]. Among all, the advanced ELM based separation technology has published promising results and findings for the separation and purification of waste streams owing to its process simplicity, large surface area for interfacial reaction, minimum membrane thickness, and high efficiency [6,7]. ELM based separation technology is a novel and efficient technology for dealing with the separation of several substances (hydrocarbons, metal ions, organic acids, biologically compounds, lignin recovery from pulping wastewater, gaseous mixtures) from the waste dilute streams [2,8,9]. Usually, emulsions are classified into two categories: single emulsions (oil-in-water (O/W), water-in-oil (W/O), and water-in-water (W/W)) and double emulsions (water-in-oil-in-water (W/O/W) and oil-in-water-in-oil (O/W/O)) [10]. Double emulsions are capable to produce huge interfacial areas for mass transfer between aqueous phase and stripping phase through the dispersion of emulsion globules into the aqueous solution [11]. Double emulsions need both water-soluble and oil-soluble surfactants in water and

oil phases for the synergetic interfacial stabilization [12]. These emulsions are known as metastable colloids and formed through two non-miscible liquids. Generally, in the process of emulsification one liquid is spread into the second liquid in the presence of an emulsifying reagent. Presently, two key techniques of emulsification are applied for the ELM preparation: Low and high energy techniques. High energy technique generates strong mechanical energy either through high shear stirring or high-pressure homogenizers [13,14,15]. Since some previous time, homogeneous and fine dispersed stable emulsions have practical importance in ELM based various separation technologies [13,16]. At present, the major hindrance in ELM scaleup/industrial applicability is its stability which measured in terms of either in membrane breakage (%) or in emulsion swelling. Emulsion swelling and membrane breakage (%) are the most uninvited processes which generally happen during the various ELM based separation/purification operations [15,17]. The again and again usage of organic phase has contrary effect on the ELM performance because it decreases stability of ELM due to further the declining in the emulsifying agent's (such as Span 80) surfactant properties [2,18]. Hence, the selection and formulation of the various ELM organic phase constituents for obtaining the stable emulsion is still a problematic. The ELM stability can be improved by the selection and incorporating the most appropriate organic phase constituents [15]. The ELM stability can also be improved through optimizing the values of various operating process variables. These emulsions have complex and diverse composition [19]. An ELM based separation process involves following key ingredients; surfactants, co-surfactants, carriers, ionic liquids, and diluents. A number of surfactants (mainly nonionic surfactants) were applied for the stabilization of emulsions through the both mechanisms i.e., steric and electrostatics. Both mechanisms take place, but generally, one of them is dominant and finally plays key part in the ELM stability. But the usage of various additional emulsifying additives (e.g., co-emulsifiers) during emulsion stabilization is problematic due to their unpredictable stabilizing effect [13,20]. Another important component of ELM phase is the diluent whose selection depends various factors viz. ELM stability, extraction efficiency (%), and cost.

Also, the application of carriers/extractants in the ELM organic phase formulation smooth the transfer of solute from the aqueous solution to the stripping solution and accelerates the separation process [21]. Therefore, there is a pertinent need of appropriate choice of several constituents of organic phase for the ELM preparation and formulation. Till date, emulsion stability is a key problem in the ELM based separation processes, hence, a thorough parametric research work on the stability performance in term of membrane breakage (%)) and later its successful application for lactic acid extraction has been examined experimentally. Lactic acid is a bio-chemical mainly used as a platform chemical for various applications such as generating products appearing in various sectors, such as food (for mineral fortification of stable food and as a buffering agent), chemical (as a descaling agent, pH regulator, and neutralizer), medical industries (to make screws, plates, pins, and rods clip for wound closing) [22,23,24]. The key purpose of this current experimental investigation is to optimize the several process variables viz. emulsification speed and time, surfactant concentration, type of stripping agents, stripping phase concentration, types of diluents, extractant (TOA) concentration, phase ratio, treat ratio, and agitation speed using one-factor-at-a-time (OFAT) optimization technique for gaining stable ELM by measuring its performance in terms of membrane breakage (%). The efficiency of these optimized process parameters assessed experimentally by making a stable ELM system. The effect of these process variables has been estimated to gain maximum extraction efficacy of lactic acid.

2. Experimental methodology

2.1. Reagents

Span 80 was brought from MERCK. Internal phase agents (NaOH, NaCl, NH₄OH and Na₂CO₃) procured from S.D. fine Chem. Ltd, India. Diluents (such as n-heptane, n-hexane, and kerosene), lactic acid (as a solute), extractant (n-tri-octyl amine (TOA)) were brought from S.D. fine Chem. Ltd, India.

2.2. ELM formulation and preparation

Normally, there are two types of ELMs i.e., water-in-oil-in-water (W/O/W) and oil-in water-in-oil (O/W/O). These ELMs mainly contain membrane, stripping, and aqueous phases. The key constituents used during ELM organic phase are as follows: solvents, nonionic surfaceactive agent (Span 80), and extractant (Trioctylamine, TOA). The preparation of organic phase was done through mixing suitable quantity of Span-80 (used as an emulsifying agent) in hexane (selected from other diluents) and TOA (extractant) in a beaker at the temperature of 25 °C for 5 min using the magnetic stirrer at the stirring speed of 500 rpm. After that the stripping phase was formulated using various stripping agents (such as NaOH, NaCl, NH₄OH and Na₂CO₃). The aqueous solution of stripping phase was made by selecting appropriate quantity of sodium carbonate (Na₂CO₃). ELM was formed by mixing the two phases (organic and stripping) using high-speed overhead Homogenizer at various homogenizing speed through varying time. Aqueous solution of Na₂CO₃ poured in dropwise fashion to organic solution. After the ELM formation, the emulsion was put in 200 mL glass batch reactor in the external aqueous phase for measuring its performance in terms of membrane breakage (%). The schematic representation of ELM formulation and preparation for LA extraction from aqueous solution has been shown in Fig.1. Experimental conditions and operating process parameter with range have been discussed in Table no. 1 for ELM formulation and preparation with the purpose of optimizing ELM stability in terms of membrane breakage (%).

2.3. Estimation of membrane breakage (%)

The main aim of exploring the performance of the numerous variables towards the ELM stability in terms of membrane breakage (%) was to elucidate the utility of ELM for various industrial applications mainly in separation and purification area [17,25]. The ELM stability performance in terms of membrane breakage (%) was measured by calculating the relative change in emulsion volume before and after extraction process as given by Eq.1.

Figure 1.

Table 1.

Swelling/Breakage (%) =
$$\frac{V_{final} - V_{initial}}{V_{initial}} \times 100$$
 (1)

where, $V_{initial}$ is the original emulsion volume before agitation, V_{final} is the volume after agitation. Negative and positive values of difference between V_{final} and $V_{initial}$ was measured as breakage (%) and swelling (%) [26]. Moreover, the membrane breakage (%) was calculated by determining the initial and final aqueous phase pH. Membrane breakage (%) was calculated using Eq. (2) and Eq. (3). Membrane breakage (%) = $\frac{\text{internal phase volume leaked into external aqueous phase by splitting }(V_s)}{\text{initial volume of internal phase }(V_{int})} \times 100\%$ (2)

 V_s can be determined by using mass balance. $V_s = V_{Ext} \frac{10^{pH_0-14} - 10^{pH-14}}{10^{pH_0-14} - C_{OH^-}^{oH^-}}$

(3)

Where: $C_{OH^-}^{int}$ is the initial concentration of OH^- in the internal phase,

 V_{Ext} is the initial aqueous solution volume

 pH_0 is the initial pH of aqueous phase,

pH is pH of aqueous solution after in contacting with the membrane for a certain time of stirring, respectively [11].

3. Results and discussion

3.1. Effect of emulsification time and speed

Emulsification speed and time have been considered as the key process parameter in the discussion of both the membrane stability as well as extraction efficacy through emulsion liquid membrane (ELM) based removal processes [1,11]. The combined effect of both factors on the membrane breakage (%) has been shown in Fig.2. At any given emulsification speed, the ELM stability in the terms of membrane breakage (%) increases with an increase in the emulsification time because longer emulsification time produce emulsion of smaller diameter and more stable emulsion till a critical time limit [27]. After that the emulsion tends to become less stable because of high internal shear force for a longer time period to encapsulate large number of small size internal droplets in a unit volume. It causes coalescence phenomenon due to increase in the collision frequency between small droplets leads emulsion breakage and makes the emulsion droplets bigger, which facilitates the water transport phenomena into the emulsion. For low emulsification time (<10 min), large breakage was found due to the droplets have a big size, which is conducive to their coalescence [28].

Figure 2.

3.2. Effect of surfactant concentration

Surfactants or emulsifying agents are known as the important organic components. They assist during formulation of water emulsion in oil (W/O). The main function of these surfactants is to reduce interfacial tension between water and oil by getting adsorbed at L-L (liquid-liquid) interface [17,29]. The influence of emulsifying agent concentration (span 80) on membrane breakage (%) was illustrated in figure 3. The higher concentration of surfactant leads to good emulsion stability. After 6 % (v/v) span 80 concentration, the ELM stability tends to decrease because the saturation of surfactant molecules at the interface between oil-water. The high surfactant concentration causes a decrease in interfacial tension at the macro droplet surface which leads to sharp breakage of macro droplets [11]. Moreover, at higher surfactant concentration (CMC) of surfactant, the molecules of surfactant tend to make large masses in the solution leading to emulsion swelling owing to transportation of water molecules from feed to stripping solution and hence causes breakage.

Figure 3.

For small values of span 80 concentration, the availability of span 80 molecules is inadequate and the coverage of membrane interface is incomplete, due to which coalescence takes place and making larger size of the droplets [30,31]. Span 80 concentration of 3% (w/v) [32], and 3-5% (w/w) [33] had been reported sufficient for the production of the stable emulsion. Thus, keeping in view the demulsification for the recovery of solute, 4% (v/v) of Span 80 concentration has been considered to be enough for preparing stable ELM.

3.3. Effect of stripping agents

Types of stripping agents (NaOH, NaCl, NH₄OH and Na₂CO₃) and their concentration in the organic solution have a vital role on the ELM stability and the extraction performance during the treatment of low solute molecules from the aqueous industrial waste solution. The key role of these internal phase reagents is mainly to create a driving force between feed solution and stripping solution for the transportation of molecules from aqueous solution to stripping phase through the membrane phase [7]. The consequence of stripping reagents (such as NaOH, NaCl, NH₄OH and Na₂CO₃) on the ELM stability in terms of membrane breakage (%) has been depicted in figure 4.

Figure 4.

Among the different internal phase reagents tested, membrane phase containing sodium carbonate (Na_2CO_3) has been shown the best results i.e., stable emulsion. Other internal phase reagents such as NaOH, NaCl, and NH₄OH have shown the poor ELM stability. More specifically, strong bases e.g., Sodium hydroxide (NaOH) and ammonium hydroxide (NH₄OH) make the emulsion unstable. It is further reported that sodium hydroxide (NaOH) in the stripping solution causes membrane breakage because emulsion swelling and hydrolysis of the ester bonds of Span 80 [11]. Therefore, in the following ELM experiments, only Sodium bicarbonate (Na₂CO₃) was used as the stripping agent for the extraction of lactic acid through ELM based separation process.

3.4. Effect of stripping phase concentration

Sodium carbonate (selected as a stripping reagent) and its concentration play an imperative role in ELM for making ELM based separation technique more effective. Both have impact on ELM stability and extraction performance. Experiments were performed using Na₂CO₃ (selected as most desirable stripping agent from Fig. 4) as internal phase reagent for concentrations (0.05, 0.10, 0.25, 0.50 N). The findings have been illustrated in figure 5. Membrane breakage (%) decreases with the rise in sodium carbonate (Na₂CO₃) concentration in stripping solution (as shown in Fig.5). The plausible reason for this may be the reaction between sodium carbonate and the emulsifying agent (Span 80) causing a partial drop in the surfactant properties which further reduces membrane stability [33]. Increasing Na₂CO₃ amount in the stripping phase also increases the difference in the ionic strength between external phase and internal solution which further may be responsible for emulsion swelling and finally, promotes excess emulsion leakage [25,34].

Figure 5.

The rise in stripping reagent concentration also increases the water transportation because of the enhancement in ionic strength difference between stripping and aqueous solution and hence thereby resulting in swelling causing coalesces [1,7]. Therefore, 0.1 M sodium carbonate concentration in the stripping phase has been chosen as an optimum value for obtaining the better ELM stability for further its applications in various extraction processes to treat/separate the various toxic and valuable substances from the industrial streams.

3.5. Effect of diluents

The selection of the organic diluent from the available diluents (such as hexane, heptane, and kerosene) in which the selected extractant (in this case n-trioctyl amine (TOA)) is dissolved depends on the concentration of extractant employed, local availability, and safety, health, and environmental consideration [2,35]. The effect of various petroleum based organic solvents (such as hexane, heptane, and kerosene) on the emulsion stability mainly in terms of breakage (%) has been investigated and shown in Fig.6.

Figure 6.

The frequently used diluent in the formulation and preparation of ELM organic phase is hexane in comparison to other diluents, followed by heptane and kerosene. Additionally, viscosity (pertinent property of solvent for making ELM stable) of hexane is more (0.31 cP) with respect to the other solvents [2]. Emulsion formed using n-heptane and kerosene were found more unstable in comparison to n- hexane may be owing to the formation of bigger droplets owing to their higher viscosities than that of n-hexane [33]. Moreover, longer duration stability behavior had been depicted by the emulsion formed by using n-hexane.

3.6. Effect of extractant concentration

The addition of n-trioctyl amine (TOA) as an extractant agent in ELM organic solution increases the permeation of molecules to be extracted from the external solution to stripping solution by the membrane phase. Though, the availability of both extractant as well as surfactant in the same phase enhances competitive adsorption between them. The addition of the emulsifying agent lessens the interfacial tension of emulsion solution which increases with rise in extractant concentration [1,7]. Therefore, optimum concentration of stripping reagent (sodium carbonate), surfactant (Span 80) and extractant (TOA) are essential to gain better separation performance of ELM. The selection of tri-octylamine (TOA) as an extractant was based on author's preliminary experimental study. Beside it, tri-octylamine (TOA) is also most widely used extractant during ELM formulation and preparation in the previously published research articles related to ELM [15]. It was observed from Fig.7 that the highest extraction efficacy (%) and good emulsion stability in terms of membrane breakage (%) were obtained at 4 % addition of extractant (TOA) to the given organic phase of ELM.

Figure 7.

Supposedly, extraction performance and permeation rate enhance with an increase in carrier concentration. With the increase in the n-trioctyl amine concentration up-to 5% (v/v), the emulsion stability was marginally affected (as shown in Fig. 7). It may be due to the interfacial properties of the extractant [2]. However, further increment in the amount of n-trioctyl amine (>5%, v/v) leads to rise in membrane breakage (%) and subsequently, reduces the ELM stability.

The higher extractant concentration increases the viscosity which further helps in forming larger globules and also promotes permeation swelling due to water transport phenomena [36]. An appropriate quantity of extractant was needed to separate and transport the lactic acid molecules to membrane solution, which then entrapped them from being released back into the aqueous solution [17]. Hence, carrier (TOA) concentration (4%, w/w) was found to give the best membrane stability.

3.7. Effect of phase ratio

Phase ratio (PR) is the Volumatic ratio of internal phase (IP) to the organic phase (OP). Phase ratio is an integral part of the ELM formulation and preparation along with the optimized process conditions. Any change in stripping phase volume not only influences the emulsion properties but also separation efficacy (%) of ELM-based separation processes for the extraction/purification of several effluent streams [17,37]. The effect of phase ratio (0.5, 0.75, 1, 1.33, 1.5 v/v) on the ELM stability measured in terms of membrane breakage (%) was illustrated in Figure 8. The volume of the organic solution (means hexane) was kept fixed (15 ml) but the sodium carbonate (Na₂CO₃) amount was changed for gaining the suitable phase ratio for further obtaining the stable emulsion liquid membrane. It was observed that with an increase in phase ratio, ELM stability was found to be reduced (as shown in Fig.8) due to increase in membrane breakage (%) with the phase ratio. The enhanced internal phase volume causes a reduction in membrane bending moment due to unavailability of sufficient organic solution volume for encircling all the stripping phase resulting in leakage of the stripping phase into the feed [38].

Figure 8.

Moreover, increasing the volume of internal solution, changes the stripping phase droplet size distribution toward bigger size, hence the thickness of film in the emulsion got reduced owing to dispersion of stripping phase in the membrane by mixing leading to instability of globules [30,34]. Henceforth, phase ratio of 1 (v/v) was selected as an optimal value for obtaining stable ELM in terms of membrane breakage (%) and also to gain the homogeneous distribution of internal reagent (Na₂CO₃) small droplets in the aqueous solution.

3.8. Effect of treat ratio

The effect of treat ratio (v/v) on the membrane breakage (%) of emulsion liquid membrane was depicted in Fig.9. The ratio of membrane phase to external phase is defined as treat ratio which further has influence on the ELM stability in terms of membrane breakage (%) and ELM extraction performance. Membrane breakage (%) was measured for various values of treat ratio (such as 1:1, 1:2, 1:3, 1:4). The treat ratio (volume ratio of membrane phase to feed phase) limits the reaction zone through the distribution of emulsion globules in the aqueous solution of various industrial streams [1]. With the rise in treat ratio provide larger space for the emulsion dispersion enhances the contact area between two phases due to the formation of several small emulsion droplets [23].

Figure 9.

But further increment in the treat ratio values may cause emulsion swelling of emulsion consequential from water transportation from external solution into internal solution due to osmosis which further causes breakage of emulsion [11]. Henceforth, the treat ratio of 1:2 (v/v) was chosen as the optimal figure for obtaining stable emulsion liquid membrane in terms of minimum membrane breakage (%) and also to have a better dispersion of emulsion phase in the feed phase which have the solute molecules to be separated.

3.9. Effect of stirring speed

Stirring speed is known as a very significant factor due to it further controls the ELM performances during the separation/treatment of several waste streams. It is a noteworthy parameter which affects the ELM stability and, subsequently, the overall extraction percentage [1]. In this current research work, the agitation speed varies between from 100 to 300 rpm as elucidated in Fig.10. The emulsion stability was found to be maximum at 200 rpm.

Figure 10.

At lower value of the stirring speed (<200 rpm), the emulsion breakage is more pronounced and leakage of the stripping solution happens because of enhanced size of the emulsion droplets. While at higher stirring speed (>200 rpm), the width of membrane phase decreases promoting the diffusing process which in turn causes the rise in the osmotic swelling of membrane. High stirring speed also developed large shear force acting on the emulsion globule further causes rupturing of the membrane affecting ELM stability [17,26]. Therefore, the stirring speed of 200 rpm was considered as to ensure a better ELM stability and to increase the interfacial area available for mass transfer.

4. Lactic acid (LA) extraction

The LA extraction efficacy (%) under the obtained process conditions of various process variables according to membrane breakage (%) was explored. The ELM prepared under these obtained process conditions (such as emulsification time: 20 min, emulsification speed: 2000 rpm, span 80 concentrations: 4% (v/v), internal solution concentration: 0.1 [M], extractant (TOA) concentration: 10%, phase ratio: 1.0 (v/v), treat ratio: 2 (v/v), and stirring speed: 200 rpm) is found stable because of the formation of the small size droplets to give the poor membrane breakage (%). Table 2 has been described the summary of the findings. To gain the highest possible percentage LA extraction by using the ELM based separation technology, a stable ELM formulation is essential [39]. The ELM stability has strong influence on the extraction process due to the unstable phenomena (i.e., leakage of stripping agent causes a reduction in driving force) of ELM process lowers the extraction performance [11,27]. The leakage of stripping agent from the stripping phase was not only disturb the extraction process but also pollutes the aqueous solution with an inner phase reagent (sodium carbonate (Na_2CO_3)). The attained findings of the ELM stability study have elucidated that the highest extraction efficiency was 95%, along with the minimum membrane breakage of 4.5%. The lactic acid (LA) extraction efficiency i.e., 95% of this current manuscript has been found comparable/or in the range with the other previous published LA isolation methods [17, 40].

Table 2

5. Conclusion

An approach of one-factor-at-time (OFAT) used in this experimental research work reveals the contribution of several factors affecting the breakage of an ELM based separation system. The findings of membrane breakage (%) were described by investigating several parameters. Throughout this research work, the best possible process conditions were reported to be at the emulsification time: 20 min, emulsification speed: 2000 rpm, span 80 concentrations: 4% (v/v), stripping phase concentration: 0.1 [M], extractant (TOA) concentration: 10%, phase ratio: 1.0 (v/v), treat ratio: 2 (v/v), and stirring speed: 200 rpm. Based on the best process conditions obtained, the findings give membrane breakage of 4.5 % with the removal efficiency of 95%. This experimental research work would be very supportive to manage the membrane breakage (%) in order to gain the optimum performance of ELM systems.

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Figure Captions

Figure 1. Schematic representation of ELM formulation and preparation for LA extraction from aqueous solution [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 2000 rpm, Diluent: n-hexane, Stripping agent: Sodium carbonate (Na₂CO₃), and Emulsion volume: 20 ml]

Figure 2. Effect of emulsification time and speed on emulsion stability [Experimental conditions: Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Diluent: n-hexane, Stripping agent: Sodium carbonate (Na₂CO₃), and Emulsion volume: 20 ml]

Figure 3. Effect of surfactant concentration on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Diluent: n-hexane, Stripping agent: Sodium carbonate (Na₂CO₃), and Emulsion volume: 20 ml]

Figure 4. Effect of stripping agent on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Diluent: n-hexane, and Emulsion volume: 20 ml]

Figure 5. Effect of sodium carbonate concentration on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Diluent: n-hexane, and Emulsion volume: 20 ml]

Figure 6. Effect of diluents on emulsion stability [**Experimental conditions:** Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Stripping agent: Sodium carbonate (Na₂CO₃), and Emulsion volume: 20 ml]

Figure 7. Effect of extractant (TOA) concentration on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Stripping agent: Sodium carbonate (Na₂CO₃), Diluent: n-hexane, and Emulsion volume: 20 ml]

Figure 8. Effect of phase ratio on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Treat ratio: 2 (v/v), Stirring speed: 200 rpm, Stripping agent: Sodium carbonate (Na₂CO₃), Diluent: n-hexane, and Emulsion volume: 20 ml]

Figure 9. Effect of treat ratio on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Stirring speed: 200 rpm, Stripping agent: Sodium carbonate (Na₂CO₃), Diluent: n-hexane, and Emulsion volume: 20 ml]

Figure 10. Effect of stirring speed on emulsion stability [Experimental conditions: Emulsification time: 20 min, Emulsification speed: 2000 rpm, Span 80 concentration: 4% (v/v), Stripping phase concentration: 0.1 M, Extractant (TOA) concentration: 10%, Phase ratio: 1.0 (v/v), Treat ratio: 2 (v/v), Stripping agent: Sodium carbonate (Na₂CO₃), Diluent: n-hexane, and Emulsion volume: 20 ml]

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 Table 1. Operating parameters for ELM stability through membrane breakage (%)

 Process conditions

S. No.	Parameters	Values
1	Emulsification speed, (rpm)	1500, 2000, 2500
2	Emulsification time, (min)	10, 15, 20, 25, 30
3	Span 80 concentration, (%, v/v)	3, 4, 5, 6, 7, 8
4	Type of stripping agents	NaOH, NaCl, NH ₄ OH and Na ₂ CO ₃
5	NaOH concentration, [M]	0.05, 0.10, 0.25, 0.50
6	Type of organic diluents	n-hexane, n-heptane, and kerosene
7	Extractant (TOA) concentration, (%, v/v)	0, 5, 10, 15, 20, 25, 30
8	Phase ratio (PR), (v/v)	0.5, 0.75, 1, 1.33, 1.5
9	Treat ratio (TR), (v/v)	1:1, 1:2, 1:3, 1:4
10	Agitation speed, (rpm)	100, 150, 200, 250, 300

PR (phase ratio) = Internal phase (IP): Organic phase (OP), TR (treat ratio) = Emulsion phase (EP): External phase (EP)

S.NO.	Process Parameters	Optimum Values
1.	LA concentration	0.05, [M]
2.	Na_2CO_3 concentration	0.1 [M]
3.	Span 80 concentration	4% (v/v)
4.	TOA concentration	10 % (v/v)
5.	Phase ratio	1, v/v
6.	Treat ratio	2, v/v
7.	Agitation speed	200 rpm
8.	Internal phase reagent	Sodium carbonate (Na_2CO_3)
9.	Type of diluent	n-hexane
Results	Membrane Breakage	4.5 %
	LA extraction efficiency	95%

Table 2 Summary of final results based on process parameters examined

A Brief on Technical Biography of Authors

First Author Technical Biography:

Dr. Avinash Thakur is Professor of the Department of Chemical engineering at Sant Longowal Institute of Engineering and Technology (Deemed to be University: Established by Govt. of India), Longowal-148106, Punjab. India. He also served as Head of the Department. He has 26 years of experience in teaching and research. Dr. Avinash Thakur has supervised 01 Ph.D. Scholar, 35 B.Tech. and 10 M. Tech. projects and currently supervising 08 Ph.D. scholars. He has one process and two design patents to his credit. Two no of externally funded research projects by Govt. agencies are being looked after by him. He is life member of Biotechnology Research Society of India, Indian Society for Technical Education etc. His articles have appeared in journals such as Waste and Biomass Valorization, Journal of Cleaner Production, International Journal of Food Engineering, Separation and Purification Technology, Journal of Industrial and Engineering Chemistry, Biomass Conversion and Biorefinery, Reviews in Environmental Science and Bio/Technology, Journal of Dispersion Science and Technology etc. His research is focused in the area of Biochemical Engineering and downstream separation processes especially green solvent extraction and value addition of agro-industrial wastes. Prof. Thakur has h-index of 13 (Google scholar) and more than 583 citations. He has visited countries such as UK, Switzerland, New Zealand, Australia for academic assignments.

Second Author Technical Biography:

Prof. Parmjit S. Panesar is currently working as Professor, Department of Food Engineering & Technology, Sant Longowal Institute of Engineering and Technology. Prof. Panesar has more than 26 years of teaching & research experience and also served in administrative positions. In 2005, he has been awarded BOYSCAST fellowship by Department of Science & Technology (DST), Govt. of India, to carry out advance research at Chembiotech labs, University of Birmingham Research Park, UK. In 1999, Prof. Panesar was awarded Young Scientist Fellowship. He has published 180 international/national scientific papers, 50 book reviews in peer-reviewed journals, 50 chapters and has authored/edited 10 books. He has supervised 16 Ph.D. students & more than 30 M.Tech students. He is a member of the editorial advisory boards of various national/international journals. He is now serving as a member of the National level Scientific Panel on "Alcoholic Beverages" constituted by FSSAI, India. In recognition of his work, Prof. Panesar was elected as 'Fellow 2018' by The Biotech Research Society of India (BRSI), 'Fellow 2019' of 'National Academy of Dairy Sciences', 'Fellow 2021' by the 'International Society for Energy, Environment & Sustainability (ISEES)' and 'Fellow 2021' of 'Academy of Microbiological Sciences'. Prof. Panesar was also listed consequently four times in the most coveted list (2020, 2021, 2022, 2023) of "World Ranking of Top 2% Scientists" published by Stanford University, USA. His research is focused in the area of value addition of food industry by products, circular economy, green extraction of bioactives, nanoencapsulation, functional food products. Prof. Panesar has h-index of 47 (Google scholar) and more than 7000 citations.

Third Author Technical Biography:

Presently, he is working as Director, at Global Group of Institutes Graphic, Amritsar, Punjab, India. He has worked as Scientist/Engineer at Propellant Fuel Complex, Vikram Sarabhai Space Centre, ISRO, Trivandrum from March 1982 to March 1994. Afterward, he also served as Asstt. Professor & Head at SLIET Longowal in March 1994 & became Professor there in 2000. Then for two year (2002-2003 & 2006-2007) he worked as Professor abroad. I also worked as Dean for one year at PTU Jalandhar (2004-05). He also worked here as Director, Guru Nanak Dev Engineering College, Ludhiana since Jan. 2008. Graduated from BIT Sindri (Established in 1949), Post Graduated (Chemical Eng.) from India's number one (Premier Institute) IISc, Bangalore & MBA from Madurai Kamaraj University and Doctorate (Ph.D.) from Thapar Institute of Engineering & Technology (now known as Thapar University).

Corresponding Author Technical Biography:

Dr. Anil Kumar is working as guest faculty in the Department of Chemical Engineering, Sant Longowal Institute of Engineering and Technology (Deemed to be University, Under MHRD, Govt. of India), Longowal-148106, Sangrur since 24 January, 2023. He has published 16 articles in the peer-reviewed journals (such as Journal of Cleaner Production, Separation and Purification Technology, Journal of Industrial and Engineering Chemistry, Reviews in Environmental Science and Bio/Technology, Journal of Dispersion Science and Technology, etc.) with the total impact factor (TIF) of 84.541. He also got one Indian patent granted. He has 420 Citations with h-index 10. I got SLIET Quality Publication Award 6 times with the cash prize of 30,000. He visited 3 International conferences, various national conferences, and participated in many more short-term courses and other academic activities. He is also a life member of Biotechnology Research Society of India (BRSI) and Indian institute of Chemical Engineers (IIChE). His areas of interest are Green chemical Engineering, Green extraction, applications of green solvents in place of toxic and costly petroleum-based solvents in liquid membranes, separation and purification of low concentrated solutes (such as toxic metal ions and organic acids) from industrial waste streams, mathematical modeling, and process optimization.