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Emulsion Liquid Membrane Screening for Ibuprofen Removal from Aqueous Solution

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ABSTRACT: *Ibuprofen (IBP) has become increasingly widespread in our environment, posing a major problem for treatment by its high occurrence in low concentrations. Emulsion liquid membrane (ELM) technology has emerged as an alternative solution for removing low concentration pollutants from contaminated sources. This study screened ELM formulations for the removal of IBP from an aqueous solution. Emulsion screening was conducted using a series of reactants, including petroleum-based diluent, green diluent, and acidic/alkaline stripping agent. The ELM system, which included hexane (diluent), 0.1 M sodium carbonate, Na_2CO_3 (stripping agent), 2 wt% Aliquat 336 (carrier) and 4 wt% Span 80 (surfactant), achieved the highest IBP extraction efficiency of 97%.*

Keywords: ibuprofen, emulsion liquid membrane, extraction efficiency

1. INTRODUCTION

Ibuprofen (IBP) or known as a pain reliever is a group of non-steroidal anti-inflammatory drugs (NSAIDs) that have become the most commonly used painkiller in the world.[1] IBP emerged as one of the best options for headaches, migraines, fever, and inflammation due to the requirement of immediate medication. IBP can be consumed by living organisms such as humans, animals, and aquatic organisms. As a result, IBP consumption resulted in a high occurrence in various water sources, which became toxic to the surrounding environment. Furthermore, IBP has been considered a contaminant of emerging concern (CEC) due to its bioactive nature, which has the potential to degrade water quality and the environment.[2] Several studies found IBP presence in the wastewater, soil, surface water, groundwater, and aquaculture in concentrations ranging from ng/L to µg/L.[2,3]

The presence of IBP at low concentrations will put pressure on the existing wastewater treatment plant due to IBP toxicity. In addition, ozonation, photodegradation, and nanofiltration methods had shown less efficient on the removal of IBP from water sources.[4–6] Therefore, an emulsion liquid membrane (ELM) emerged as a potential alternative solution for removing pollutants from wastewater. ELM is a simple method that uses the liquid-liquid extraction principle to separate specific pollutants from contaminated sources.[7] Several pharmaceutical products, including acetaminophen (painkiller), ciprofloxacin (antibiotics), diclofenac (painkiller), tetracycline (antibiotics) have been reported to achieve at least 95% of extraction.[8–11] The common use of the painkillers and antibiotics are to treat inflammatory diseases and fight the infection caused by bacteria, respectively. Not to mention, ELM has a wide range of applications in the removal of organic compounds such as dye, ethylparaben, electronic waste and lactic acid.[12–17] Ahmad et al. reviewed that copper, chromium, nickel, cobalt, silver, cadmium and lead are potentially removed by using ELM.[18]

ELM works on the basis of the facilitated type II mechanism in which the carrier and stripping play vital roles in transporting the targeted pollutants from the feed phase to the internal phase. According to Zaulkiflee et al., type of diluent, carrier, stripping agent and surfactant used in the formulation influences the extraction of the targeted pollutants.[19] Several studies have found that green diluents such as rice bran oil (RBO), corn oil (CO) and sunflower oil (SFO) had achieved a high extraction efficiency of the pollutants.[20–22] However, the compatibility of different formulations with ELM performance varies. Therefore, the current study will screen a suitable formulation for IBP extraction in the presence of various diluents/stripping agents systems, carrier concentration, surfactant concentration, and stripping agent concentration.

2. EXPERIMENTAL

2.1 Chemicals

Ibuprofen purchased from Sigma Aldrich, Darmstadt, Germany was used to represent synthetic pharmaceutical wastewater. SFO, CO, RBO bought from supermarket were used as green diluent while kerosene, hexane, and heptane bought from Emsure, Darmstadt, Germany were used as petroleum-based diluent. Aliquat 336 (Acros Organics, New Jersey, United States) and Sorbitan monoleate, Span 80 (Merck, Hohenbrunn, Germany) were used as a carrier and surfactant, respectively. Sodium hydroxide, NaOH (Merck, Darmstadt, Germany), Ammonia solution, NH_3 (Emsure, Darmstadt, Germany), Sodium bicarbonate, Na_2CO_3 (Sigma Aldrich, Darsmtadt, Germany) were used as alkaline stripping agent while hydrochloric acid, HCl and sulphuric acid, H_2SO_4 bought from Emsure, Darmstadt, Germany were used as acidic stripping agent. The feed phase containing IBP is fixed at pH 2 throughout the experiment. All the chemicals were dissolved by using deionised water.

2.2 Screening Procedure

The screening experiment is started by preparing the emulsion consisted of membrane and internal phase. The emulsion is prepared based on previous work reported by Ahmad et al.[23] Internal phase or known as stripping agent is dissolved in the mixture of diluent, carrier and surfactant which made up the membrane phase. The emulsification is assisted by Ultrasonicator (Telsonic Ultrasonix, Mumbai, India) for 10 min at room temperature. The prepared emulsion is then transferred into the feed phase contained 20 mg/L IBP. The emulsion and the feed is mixed by using mixer (IKA, Germany) at 250 rpm for 15 min.[18] The IBP concentration in feed is measured by using UV-Vis spectrophotometer (Spectroquant Pharo 300, Merck, Darmstadt, Germany) at 220 nm wavelength.[24] Figure 1 shows the experimental setup for the emulsion preparation and extraction of IBP.

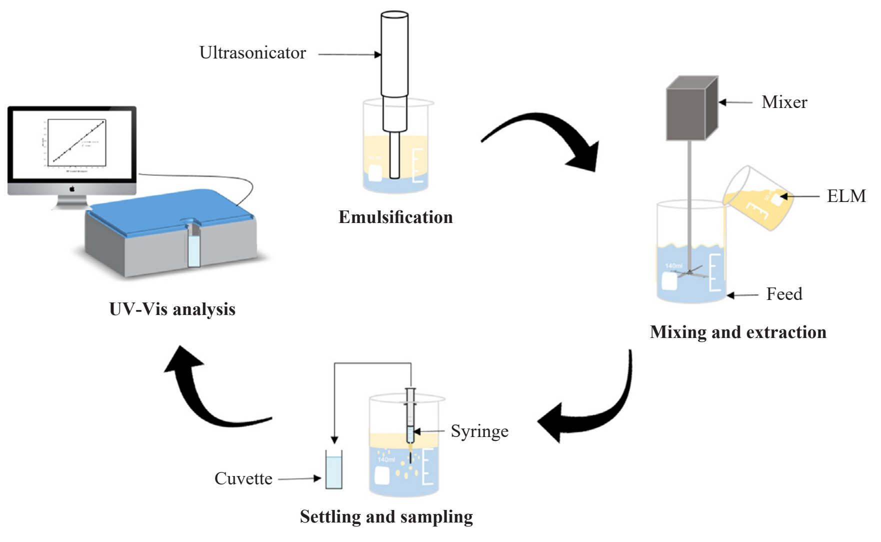

Figure 1: Screening procedure for the extraction of IBP using ELM.

A series of investigations have also been conducted to investigate the effects of main parameters, such as carrier, surfactant and stripping agent, using the best formulation from screening. The experiment was based on one factor at a time (OFAT) in which one main parameter was studied while other parameters such as organic to internal phase volume ratio (O/I), feed to membrane phase volume ratio (F/M), emulsification time and extraction contact time were kept constant. Table 1 lists the parameters for examining the effects of the main parameters. Each experiment was repeated three times to determine the average extraction efficiency.

Table 1: Parameters of study for ELM performance

Parameters		
Span 80	:	2–4 wt%
Aliquat 336	:	1–3 wt%
Na_2CO_3	:	0.05 M–0.15 M
Hexane/Na_2CO_3, O/I	:	3:1
IBP/Hexane, F/M	:	4:1
Extraction stirring speed	:	250 rpm
Emulsification time	:	10 minutes
Extraction contact time	:	15 minutes
IBP in feed	:	20 mg/L

2.3 Analysis of IBP removal

Absorbance at 220 nm for different IBP concentrations in the range of 20 mg/L–2 mg/L is determined. Figure 2 shows a calibration linear fitted graph of absorbance (Abs) against IBP concentration. Based on linear equation of the calibration curve, the final concentration of IBP can be calculated. The extraction efficiency of IBP from the feed is calculated by using (1).

$$E(\%) = \frac{IBP_{initial} - IBP_{final}}{IBP_{initial}} \times 100\% \quad (1)$$

Where E is denoted as extraction efficiency whereas IBP_{intial} and IBP_{final} are concentration of IBP before and after extraction, respectively.

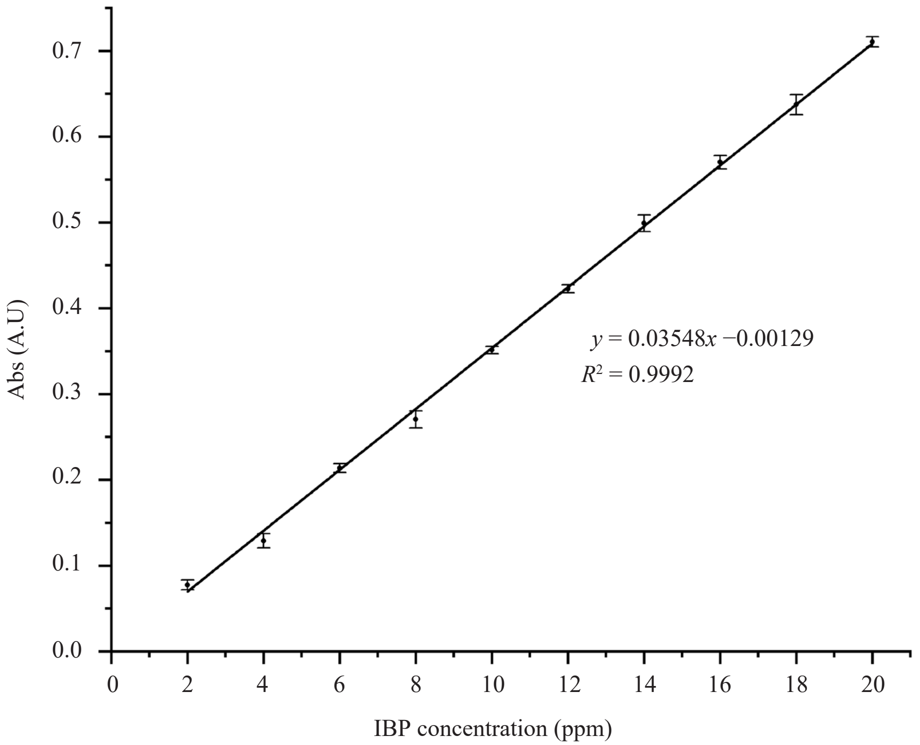

Figure 2: Linear-fit graph of absorbance at different IBP concentration ($R^2 = 0.9992$).

3. RESULT AND DISCUSSION

3.1 Diluents and Stripping Agents Screening

The screening materials included petroleum/green diluents and acidic/basic stripping agents. Diluent is an important parameter because it contributes to a major component of the ELM system. The petroleum-based diluents were heptane, hexane, and kerosene, while the green diluents were RBO, CO, and SFO. Each diluent will be screened using different stripping agents in the presence of Aliquat 336 as an ionic liquid carrier. Figure 3 depicts the extraction of IBP at various stripping agents using petroleum-based and green-based diluents.

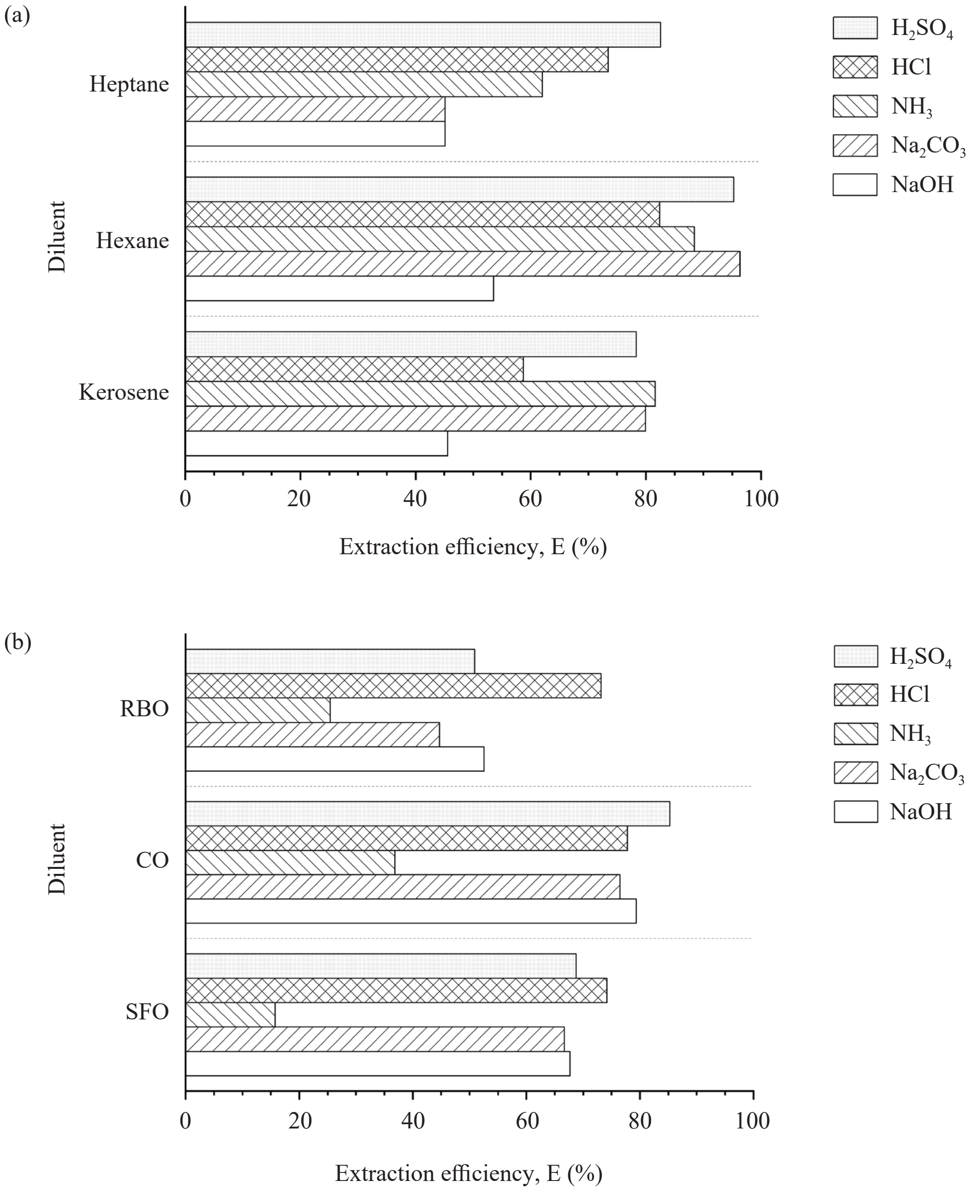

Figure 3: Screening of type of diluent, (a) petroleum, (b) green, and internal phase by using Aliquat 336 as a carrier.

Overall, petroleum-based diluent has a higher extraction efficiency than green-based diluent, with hexane-Na_2CO_3 as the best diluent-internal system. The following are the trends of increasing extraction efficiency by using Na_2CO_3 as a stripping agent such that Hexane>Kerosene>CO>SFO>Heptane>RBO.

This is due the IBP transport rate through the membrane phase containing hexane is faster than that of other diluents. Chaouchi and Hamdaoui made a similar observation, discovering that hexane has a higher transport rate than kerosene and heptane.[25] On the other hand, the optimal viscosity of the diluent is also important in maintaining ELM stability.[26] Hexane has the lowest viscosity of 0.3 cP compared to other petroleum and green diluents.[18,27,28] Djenouhat et al. state that cavitation bubbles form more readily in a low viscous diluent.[29] As more cavitation bubbles is formed, the greater the total contact area to transport IBP from the feed phase to the internal phase. Dâas and Hamdaoui found that an ELM system with hexane as the organic phase produced the most stable emulsion when compared to kerosene and heptane.[30]

The type of stripping agent used determined the internal phase's ability to trap the extracted pollutant from the feed phase. For the screening, acidic stripping agents such as H_2SO_4, HCl, and alkaline phase components such as NH_3, Na_2CO_3, and NaOH were used. Except for the H_2SO_4-RBO system, the results for green diluent indicated that extraction efficiency increased for HCl, H_2SO_4, NaOH, Na_2CO_3, and NH_3. The low extraction of the system when compared to using HCl as the internal phase could be attributed to a difference in ionic strength between the feed and internal phases, which causes emulsion breakage.[31] Stripping agent in petroleum-based diluent had shown an inconsistent trend, which was due to the interaction with surfactant, diluent and carrier in the ELM. Kohli et al. reported that the stripping agent can cause the emulsion breakage which is due from the hydrolysis with the surfactant thus lowering the extraction efficiency.[14] Thus, IBP removal is suitable to be removed by using formulation of Na_2CO_3 and hexane as stripping agent and diluent, respectively.

3.2 Effect of Main Parameters

The current study investigates parameters that affect the IBP, such as carrier, surfactant, and stripping agent. From of the screening in Section 3.1, hexane and Na_2CO_3 is selected as diluent and stripping agent, respectively. The parameters such as emulsification time, stirring speed, extraction time, O/I, and F/M are fixed throughout the experiment.

3.2.1 Effect of carrier concentration

Carrier, or known as extractant plays a vital role in extracting the IBP from the feed phase. Aliquat 336 is selected as a carrier due to its non-pollutant properties. Figure 4 illustrates the extraction efficiency of IBP at Aliquat 336 concentration of 1 wt%–3 wt%.

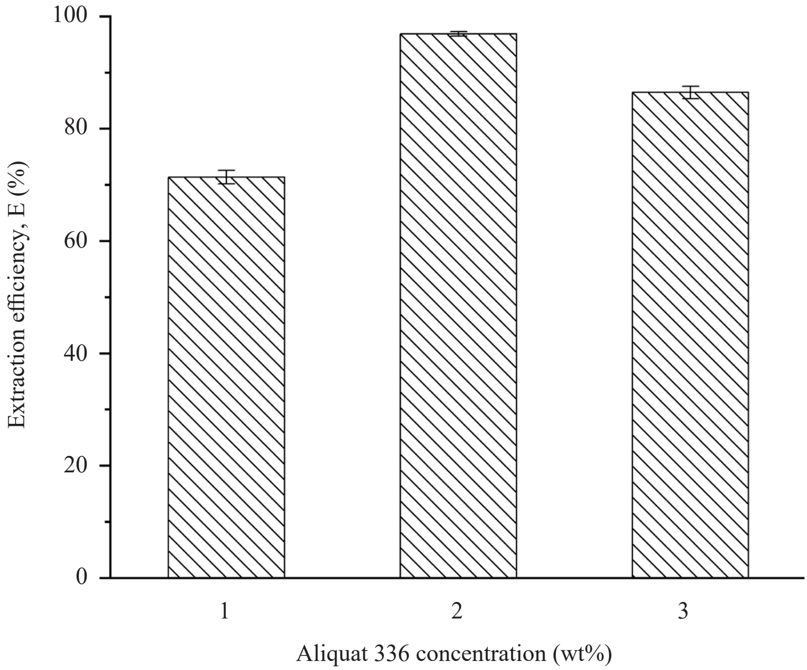

Figure 4: Effect of Aliquat 336 on extraction efficiency of IBP [Span 80 concentration = 4 wt%, Stripping agent = 0.1M Na_2CO_3].

In this study, 2 wt% Aliquat 336 showed the highest extraction efficiency of IBP. The IBP transport is improved as the carrier is increased from 1 wt%–2 wt%. However, further increased the concentration of Aliquat 336 wt%–3 wt%, the extraction efficiency decreased to 86%. According to Kumbasar, the decrease in extraction efficiency is caused by an increase in the viscosity of the membrane phase as a result of the presence of more viscous carriers in this phase.[32] The stability of the emulsion suffers when the carrier used exceeds a certain limit, reducing the extraction efficiency of IBP.[33,34] Thus, Aliquat concentration of 2 wt% is sufficient to extract the maximum amount of IBP from the feed phase.

3.2.2 Effect of surfactant concentration

With the optimised Aliquat 336 concentration of 2 wt%, the effect of surfactant concentration was studied from 2 wt%–6 wt%. Span 80 is used as a surfactant to reduce interfacial tension between phases and thus maintain emulsion stability. Figure 5 shows the result of extraction efficiency of IBP at different Span 80 concentration.

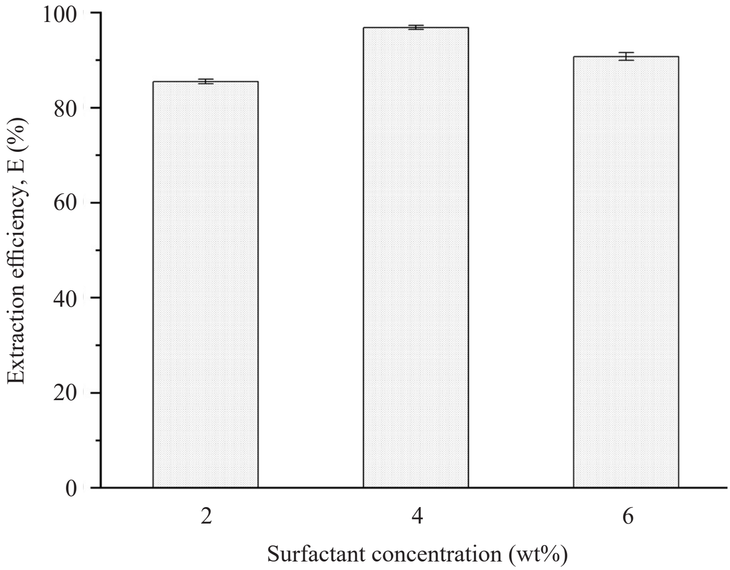

Figure 5: Effect of Span 80 on extraction efficiency of IBP [Aliquat 336 concentration = 2 wt%, Stripping agent = 0.1M Na_2CO_3].

The result shows that increasing the surfactant concentration to 4 wt% increases extraction efficiency to 97%. The stabilisation of the emulsion increases as the surfactant amount increases due to a reduction in the interfacial tension between the phases.[33] According to Ahmad et al., the minimal surface tension between the immiscible phases results in smaller globules, which increases the total contact area of the extraction.[35] Extraction performance deteriorated when Span 80 concentrations exceed 4 wt%. The swelling of the membrane caused by the amphiphilic property of Span 80 is responsible for the decline of the extraction efficiency at 6 wt%. Water was spontaneously transported across the membrane-external phase by the Span 80, which had a low hydrophile-lipophile balance (HLB) value of 4.3.[23,36] As a result, higher Span 80 caused more water to be transported to the internal phases, resulting in swelling.[37] Thus, 4 wt% Span 80 results in minimal emulsion swelling and high extraction efficiency.

3.2.3 Effect of stripping agent concentration

Stripping agent is used to strip the transported IBP from the external phase to the internal phase. While keeping the optimised Aliquat 336 and Span 80 constant, the effect of the stripping agent was investigated, as shown in Figure 6. The concentration of Na_2CO_3 is varied from 0.05 M–0.15 M with 0.05 M increments.

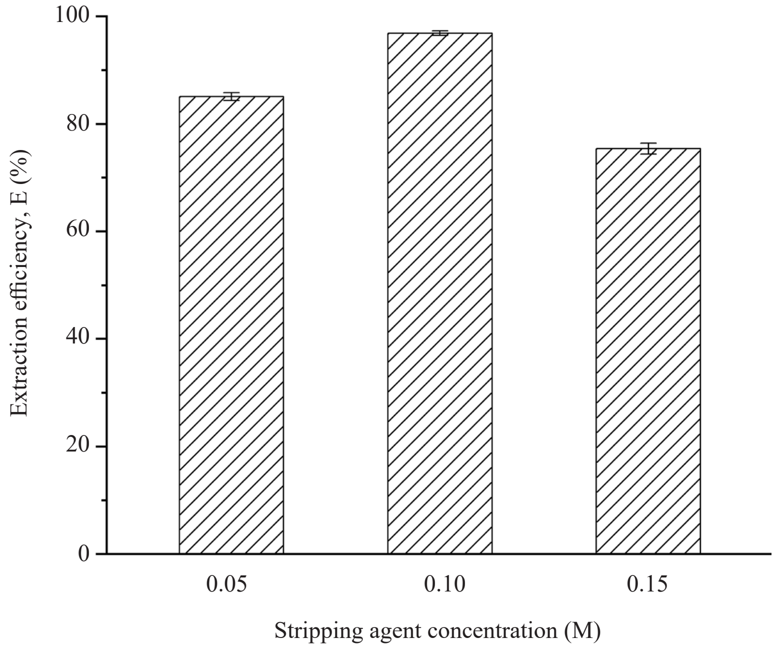

Figure 6: Effect of Na_2CO_3 on extraction efficiency of IBP [Aliquat 336 concentration = 2 wt%, Span 80 concentration = 4 wt%].

Based on Figure 6, enhancement of extraction efficiency occurred by increasing the Na_2CO_3 from 0.05 M–0.10 M. Low extraction efficiency at 0.05 M is due to the insufficient Na_2CO_3 to remove IBP from the feed phase. This finding is agreement with Chaouchi and Hamdaoui, who found that the stripping process has slowed as due to the saturation of the pollutants in the internal phase.[25] The extraction efficiency is maximum at 0.10 M Na_2CO_3 and decreases as the Na_2CO_3 concentration increases. The decrease in IBP extraction efficiency from 97%–75% is due to increased ionic strength between the internal and external phases. The significant difference in ionic strength increased the volume of the internal phase, resulting in emulsion leakage.[38] Another observation made by Sulaiman et al. was that the high concentration of Na_2CO_3 caused an interaction with the Span 80, reducing the Span 80 properties and causing the emulsion to become unstable.[39] Therefore, 0.1 M Na_2CO_3 is sufficient enough to achieve high extraction IBP from the feed phase.

4. CONCLUSION

In conclusion, a series of screenings has been done. The results showed that an ELM system consisted of petroleum-based diluent and Na_2CO_3 achieved a higher extraction efficiency than green diluents. The ELM formulation consisted of 2 wt% of Aliquat 336, 4 wt% of Span 80, hexane, and 0.1M Na_2CO_3 has shown a high extraction efficiency of about 97%.

5. ACKNOWLEDGEMENTS

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