Journal of Water Process Engineering xxx (2016) xxx–xxx

Contents lists available at ScienceDirect

Journal of Water Process Engineering

journal homepage: www.elsevier.com/locate/jwpe

Utilization of environmentally benign emulsion liquid membrane (ELM) for cadmium extraction from aqueous solution

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ARTICLE INFO

Article history:
Received 31 October 2015
Received in revised form 9 May 2016
Accepted 24 May 2016
Available online xxx

Keywords:
Emulsion liquid membrane
Vegetable oil
Cadmium separation
Demulsification

ABSTRACT

Utilization of Emulsion Liquid Membrane (ELM) for cadmium removal from aqueous solution was explored. Basically, ELM consists of 2 phases; internal and membrane phase that form the primary W/O (water-in-oil) emulsion which to be dispersed in the external phase. In this work, Aliquat 336 and Span 80 were used as carrier and surfactant, respectively whereas corn oil was used as an environmentally benign diluent in the membrane phase. Influence of operating conditions that affect ELM performance; ultrasonic power, emulsification time, treat ratio, stirring speed together with initial cadmium concentration were investigated. It was found that 20 W ultrasonic power, 12 min emulsification time, treat ratio of 1:5 and 300 rpm stirring speed were essential to achieve maximum removal efficiency (98.9%) after 15 min. The current study also exhibits the possibility of demulsification process assisted by ultrasonic probe. 48.8% emulsion breaking efficiency was recorded using ultrasonic power ≥ 33W, subjected to 5 min of irradiation time.

1. Introduction

Since its establishment in 1968 by Li [1], Emulsion Liquid Membrane (ELM) has found its usefulness in the field of separation, involving its numerous applications on heavy metal removal. This includes chromium [2], copper [3] and cobalt [4]. ELM is a multiple water-in-oil-in-water (W/O/W) emulsion system which was developed using 3 main phases. They are membrane and internal phase that were homogenized to form the primary water-in-oil (W/O) emulsion. The membrane phase consists of carrier and surfactant, dissolved in a diluent where the internal phase contains stripping agent. The existence of surfactant is important in such a way that it reduces the free energy as a result of low interfacial tension [5]. The primary emulsion was later dispersed with constant agitation in the third phase (external phase) which eventually, forms the W/O/W emulsion system [6]. Generally, heavy metal ions are insoluble in the membrane phase and it requires carrier to mediate its transportation across the membrane layer by forming a carrier-solute complex where this mode of transportation is named as Type II Facilitated Transport. In this system, the concentration gradient of solute-carrier complex across the membrane phase is maximized by reactions with stripping agent at the interface of membrane-internal [7]. This process involves solely chemical energy as a driving factor and does not need membrane pressure as well as voltage [8].

Cadmium is known as a highly toxic and non-biodegradable material, hence persistent [9]. It can be easily found in waste water from industries that are dealing with alloys, ceramics, electronics, textiles and leaching [10]. Excessive and continuous human exposure to cadmium either by ingestion or inhalation could cause headache, nausea, diarrhea and abdominal cramps [11]. In fact, several studies identified cadmium as carcinogen [12]. These factors has triggered extensive efforts to be carried out to reduce cadmium content in aqueous media using various physico-chemical treatment for instance, chemical precipitation and ion exchange [13]. On the other hand, ELM acts as an alternative to effectively remove heavy metals from aqueous solution. Othman et al. [14] reported that ELM has been applied commercially in removing zinc from wastewater in Austria's viscous fibre industry and it recorded greater than 99.5% extraction efficiency, proving the feasibility of ELM. This is due to the beneficial characteristics that ELM offers; high surface area to volume ratio, highly selective and requirement of single unit for extraction and stripping process [15].

On the other hand, demulsification is one of the critical steps in ELM which separates the water and oil phases. Demulsification refers to the process of breaking the loaded emulsion [16],

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http://dx.doi.org/10.1016/j.jwpe.2016.05.010
2214-7144/

Table 1
List of chemicals used and their purposes.

Chemicals	Purpose
Aliquat 336 (Sigma Aldrich)	Carrier
Span 80 (Merck)	Surfactant
Corn oil	Diluent
Ammonia Solution (Merck, 25%)	Internal Phase
Hydrochloric Acid (Merck, 37%)	External Phase

thus enabling the recovery of membrane phase and its reuse [17]. Besides reducing secondary pollutant, W/O emulsion breaking step also lowers the operational cost of ELM [18]. According to Lu et al. [19], breakdown of emulsion is a three steps process. The first step is called flocculation where the dispersed droplets of internal phase flocculate into some large groups and the process was followed by the formation of larger internal phase drops. In this process, the flocculated droplets were said to adhere to one another, yielding even larger droplets [20]. Finally, the large internal phase drops sink at the bottom due to the act of gravity. Several demulsification methods were discovered previously and they were classified into physical and chemical treatment [21]. Normally, chemical treatment involves the additional of demulsifier such as acetone, *n*-butanol and 2-propanol [21] whereas physical treatment available for demulsification process are; centrifugation [22], microwave radiation [23], ultrasonic exposure [24] and high-voltage electrostatic fields [25]. However, it is documented that a number of them were lacking in the aspect of high efficiency, slow and not clean.

In this study, corn oil was used as diluent in ELM formulation to replace the hazardous petroleum derivatives. Though the application of safer solvent was rapidly explored in the bulk and supported liquid membrane, but its application in the emulsion type is still limited. Since vegetable oils are lipid material derived from fruits and seeds [26], it is considered as a biological material and it is undeniably nontoxic, relatively cheap, biodegradable and renewable [27]. Basically, vegetable oils are non-polar solvent that composed of triglycerides, diglycerides and monoglycerides [26]. They possess high flash point and low melting point while their density difference with water suffices. The current study investigates the effect of several operational parameters; ultrasonic power, emulsification time, treating ratio, stirring speed as well as initial cadmium concentration on separation performance using environmentally benign ELM, followed by the evaluation on demulsification efficiency at various power of ultrasonic.

2. Experimental

2.1. Chemicals

Chemicals used in developing the ELM system and their purposes are provided in Table 1.

2.2. Procedure

2.2.1. ELM preparation

The primary emulsion was made with the assistance of ultrasonic probe, as illustrated in Fig. 1. Prior to this process, the membrane phase was prepared using 3 wt% Aliquat 336 and 3 wt% Span 80, dissolved in commercial grade corn oil while 0.1 M aqueous ammonia solution was used as the internal phase. These phases were homogenized to form a W/O emulsion where the volume ratio of internal:membrane phase is 1:3. A commercial ultrasonic (USG-150) equipped with titanium horn was used for this purpose at varying power (15 W, 20 W, 29 W). The power was identified *via* calorimetry by assuming that all of the power entering the solvent is dissipated as heat [28].

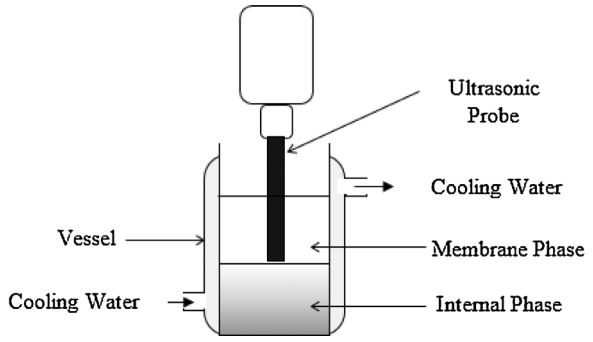

Fig. 1. Experimental setup for ultrasonic-assisted preparation of W/O emulsion.

2.2.2. ELM for cadmium extraction

Dispersion of W/O emulsion took place immediately as the emulsification process ended. The emulsion was poured in a vessel containing the external phase where the pH was adjusted to 1.0, at ratio 1:5. The content of the vessel was stirred at 400 rpm, otherwise mentioned. At the end of extraction process, the external phase sample was taken out using a syringe for cadmium ions concentration measurement and the removal efficiency, E(%) was calculated using the following equation:

$$\text{Cadmium Removal Efficiency, } E(\%) = \frac{(C_o - C_t)}{C_o} \times 100 \quad (1)$$

Where C_o is the initial concentration of cadmium in the external phase while C_t is the concentration of cadmium at the end of extraction process. The concentration of cadmium ions in the external phase was measured using Atomic Absorption Spectrophotometer model Shimadzu AA-6650 at wavelength of 228.8 nm.

2.2.3. Demulsification

Emulsion breakup was conducted using a commercial ultrasonic (USG-150) equipped with titanium horn. The spent emulsion was separated from the external phase due to the act of gravity and it was taken out using syringe where it was later placed under ultrasonic probe for a duration of time (2–10 min), at varying power (25 W, 33 W, 39 W). The broken emulsion was left for 5 min to allow complete separation of the immiscible phases before the organic membrane phase can be taken out for water content measurement. Based on the water content measured, the emulsion breaking efficiency was calculated using the following equation, as proposed by Chan and Chen [23]:

$$\text{Emulsion breaking efficiency, } \beta(\%) = \frac{V_b}{V_e} \times 100 \quad (2)$$

Where V_b and V_e is the volume of separated water phase (ppm) and initial volume of water in the emulsion (ppm), respectively. Water content in the membrane phase was measured using Metrohm 870 KF Trinitro Plus from Karl Fisher by using Hydranal as the reagent.

3. Results and discussions

3.1. Effect of ultrasonic power

Fig. 2 shows the dependence of cadmium removal efficiency, E(%) on ultrasonic power. From the figure, it is identified that ELM prepared with 15 W ultrasonic incapable to effectively remove cadmium from the external phase as it recorded the least E(%) throughout the extraction time studied. This is due to the fact that, the sound field provided is insufficient to yield enough energy to form a homogenized emulsion, thus producing large emulsion globules. As a consequent, low surface area to volume ratio was achieved. On the other hand, increment of the power to 20 W led

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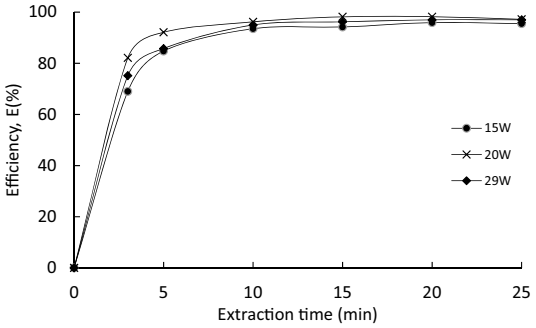

Fig. 2. Effect of ultrasonic power on cadmium removal efficiency, E(%). Experimental condition: 15 min emulsification time, treat ratio 1:5, stirring speed 400 rpm, 150 ppm cadmium.

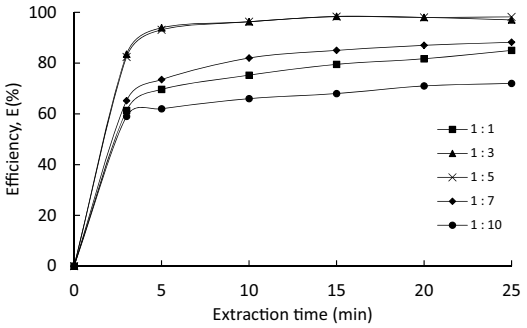

Fig. 4. Effect of treat ratio on cadmium removal efficiency, E(%). Experimental condition: 20 W ultrasonication, 12 min emulsification time, stirring speed 400 rpm, 150 ppm cadmium.

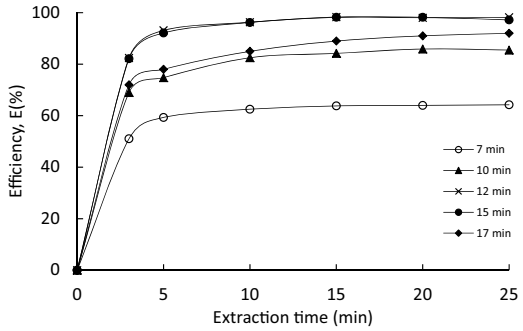

Fig. 3. Effect of emulsification time on cadmium removal efficiency, E(%). Experimental condition: 20 W ultrasonication, treat ratio 1:5, stirring speed 400 rpm, 150 ppm cadmium.

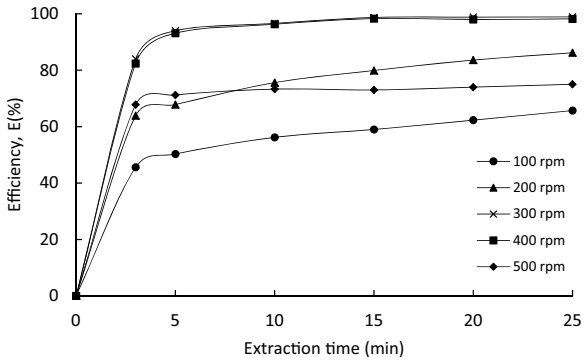

Fig. 5. Effect of stirring speed on cadmium removal efficiency, E(%). Experimental condition: 20 W ultrasonication, 12 min emulsification time, treat ratio 1:5, 150 ppm cadmium.

to a significant increment of E(%) (96.2% at 15 min), as shown in Fig. 2. As the emulsification process took place in the cavitation region, application of higher ultrasonic power increases the depth of this region [29]. This situation has evidently produced smaller emulsion globules, resulting in the increment of solute transportation area. However, employment of higher ultrasonic power (29 W) does not benefit the system either. As claimed by Djenouhat et al. [30], coalescence effect has predominate at high power of ultrasonication. Thus, 20 W is accepted as the optimum power to prepare the emulsion.

3.2. Effect of emulsification time

Fig. 3 shows the data of cadmium removal efficiency, E(%) using emulsion developed at various emulsification time. According to the figure, minimal E(%) was recorded with the application of short emulsification time (7 min). This is mainly due to the formation of large internal phase droplets resulting in a reduction of solute transportation area [31]. At this point, the time was said to be insufficient to produce a sufficiently small emulsion globule and as a consequent, poor extraction efficiency was recorded. On the other hand, sufficient time for emulsification process has allowed the production of smaller emulsion droplets [32]. This factor is highly possible to cause a significant increment of E(%) as the time for emulsification was prolonged to 12 min. It is reported that the formation of fine emulsion droplets is highly dependent on the ultrasonic irradiation time as it involves two steps process; (i) interfacial instability and (ii) droplets breaking at the interface due to cavitation effects [33]. However, insignificant increment of the efficiency was recorded as the time increases to 15 min and it was further reduced thereafter. This phenomena is caused by the coalescence of the small internal phase droplets produced [34]. Based on Fig. 3, 12 min is accepted as the appropriate time to form the emulsion.

3.3. Effect of treat ratio

Influence of treat ratio (emulsion:external phase) on cadmium removal efficiency, E(%) is shown in Fig. 4. Based on the figure, usage of neither high nor low emulsion volume has benefited the system. Low E(%) was recorded at treat ratio of 1:1, primarily due to the limited interfacial area for mass transfer. This is owing to the difficulties to disperse the emulsion as high volume of the emulsion has caused the total viscosity of W/O/W emulsion to increase, hence resulting an inverse in the interfacial surface area [32]. In fact, application of large volume of emulsion is not economically feasible. As the volume of viscous emulsion reduces, the system could be dispersed properly and the emulsion globules size is much smaller. This is the main phenomena that caused better E(%) to be noted as the volume of the emulsion reduces. According to Fig. 4, treatment ratio of 1:5 yields a maximum cadmium removal efficiency (98.3%) after 15 min of extraction. Though less emulsion is preferred from a processing point of view [35], but its usage at lower volume (ratio 1:7 and 1:10) has recorded significant E(%) reduction. This is owing to the fact that low number of emulsion globules present, hence reducing the surface area available for solute extraction purposes. As a consequent, the flux of cadmium ions transportation from the external phase to emulsion reduces [32]. To conclude, the optimum treat ratio selected is 1:5.

3.4. Effect of stirring speed

Removal efficiency, E(%) of cadmium from aqueous external phase at various stirring speed is reported in Fig. 5. The performance of ELM is directly dependent on the speed of stirring as it dictates the size of the dispersed emulsion globules. Based on the figure, increasing the speed from 100 rpm to 400 rpm has resulted in a

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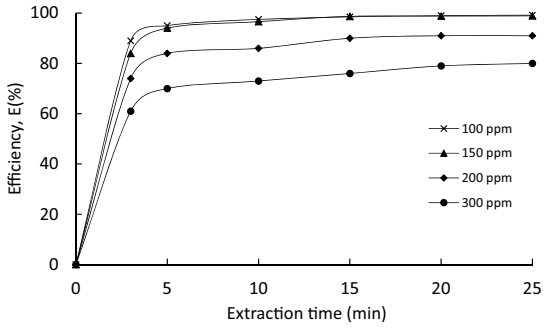

Fig. 6. Effect of initial cadmium concentration on removal efficiency, E(%). Experimental condition: 20 W ultrasonication, 12 min emulsification time, treat ratio 1:5, stirring speed 300 rpm.

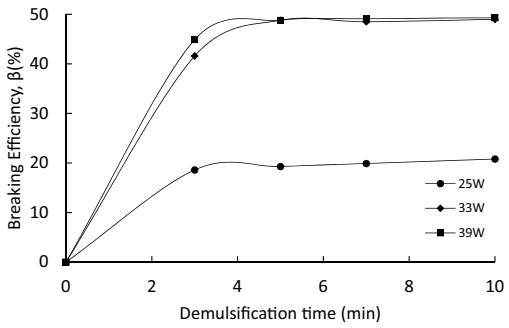

Fig. 7. Emulsion breaking efficiency, β(%) at various ultrasonic power.

large increment of E(%), although insignificant between 300 rpm and 400 rpm. Shear provided from the impeller during stirring reduces the emulsion globules size, hence providing high interfacial area for solute extraction. In fact, thinning of membrane wall could be achieved at higher speed of stirring. Due to this condition, the extracted solute need to travel shorter distance to be stripped at the membrane-internal interface. However, several authors reported on high occurrence of membrane breakage [36,37] due to excessive shear provided at high stirring speed [38]. It was followed by the expulsion of the extracted solute from the internal phase, causing the solute removal efficiency to be nullified. This is the main reason for poor extraction efficiency recorded at 500 rpm. Besides membrane breakage, swelling due to osmotic pressure gradient [39] could also contribute to the results obtained. For this parameter, 300 rpm is found to be the appropriate speed for stirring as at this point, highest cadmium removal efficiency (98.7%) was recorded.

3.5. Effect of initial cadmium concentration

Fig. 6 shows the influence of initial cadmium concentration on the removal efficiency, E(%). It is identified from the figure that external phase containing 100 ppm cadmium yields the highest E(%) throughout the period of extraction studied. Within 15 min, 98.9% of cadmium were removed. As the concentration increases beyond 150 ppm, E(%) recorded is much lower due to the saturation process [32]. As higher concentration of solute available, the internal phase needs to strip the targeted solute rapidly [40], otherwise the membrane-internal interface undergo saturation. This phenomena reduces the driving force of the system that is maximized by reactions with stripping agent at the membrane-internal interface [7], thus resulting in poor performance. Additionally, the phenomena was also accompanied by the ions strength difference between the internal and external phase that could cause emulsion swelling.

3.6. Demulsification

The possibility of emulsion breaking with the assistance of ultrasonic at various power (25 W–39 W) was investigated and the data obtained is shown in Fig. 7. Based on the data recorded, breaking of emulsion can only be carried out at power ≥ 33 W and the process requires at least 5 min. 48.8% breaking efficiency was recorded using ultrasonic power of 33 W while the application of higher ultrasonic power and prolong irradiation time has hardly improved the breaking efficiency. Demulsification is said to be impossible at low power of ultrasonic as higher energy input is necessary as the surfactant acts as a barrier to the breaking process of the emulsion where it must degrade first in order to destruct the emulsion [24]. One of the factors contributed to the success of demulsification in this study is the heat generated due to the absorption of ultrasonic energy [33]. As the temperature of the emulsion elevated due to the power dissipated by ultrasonication, the surfactant molecules were said to be degraded, hence increasing the surface tension between the two immiscible phases [41]. In addition, the acoustic streaming produced has disturbed the emulsion's stability and at the same time, allowing its destruction [42]. This situation led to the coalescence of water droplets and it sank to the bottom of the vessel once they are large enough [41].

4. Conclusions

The present work on ELM using environmentally benign diluent proves that the system developed able to effectively remove cadmium from the external phase. Nevertheless, the experimental work shows that the capabilities of ELM to extract cadmium is dependent on several operating conditions. From the data recorded, 98.9% cadmium was successfully removed at operating condition of 20 W ultrasonication, 12 min emulsification time, treat ratio 1:5 and stirring speed 300 rpm. Under the best operating condition, 15 min of extraction time is required to achieve maximum E(%). Another interest of this study is to explore emulsion breaking using ultrasonic probe. Based on the data available, ultrasonic power ≥33 W recorded high breaking efficiency (48.8%). In addition, this method allows high breaking efficiency to be achieved in short time (5 min) and it requires solely ultrasonication.

Acknowledgment

Financial support by the Membrane Science and Technology Research University Cluster from Universiti Sains Malaysia, Skim Tenaga Pengajar Muda UiTM and Ministry of Higher Education Malaysia are greatly acknowledged.

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