



## Emulsion stabilization using ionic liquid [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> and performance evaluation on the extraction of chromium

Rahul Kumar Goyal, N.S. Jayakumar, M.A. Hashim\*

Department of Chemical Engineering, University of Malaya, Malaysia

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### ABSTRACT

This study focuses on the role of a hydrophobic ionic liquid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide, [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> in the preparation of emulsion liquid membrane (ELM) phase containing kerosene as solvent, Span 80 as surfactant, NaOH as internal phase and TOMAC (tri-n-octylmethylammonium chloride) a second ionic liquid as carrier. The first time used [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> in ELM was found to play the role of a stabilizer. The emulsion prepared using [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> has a long period of stability of about 7 h (at 3% (w/w) of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup>) which otherwise has a brief stability up to only 7 min. The stability of the emulsion increases with the increase in concentration of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> up to 3% (w/w). Nevertheless, with further increase in concentration of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup>, a reduction in the stability occurs. The extraction experiments were carried out after holding the ELM for 2 h after the preparation and a removal efficiency of approximately 80% was obtained for Cr. The destabilization of the emulsion was studied by observing the change in the interface height. An empirical correlation for the stability of the emulsion has been proposed.

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### 1. Introduction

Liquid membrane technology is well known for its wide application in extraction processes to separate hydrocarbons [1–3], heavy metals [4–6], amino acids [7,8] and biological compounds [9,10]. The potential advantages of liquid membrane techniques are low capital and operating costs, low energy and extractant consumption, high concentration factors and high fluxes. This technology has an edge over solvent extraction because it requires less energy and operates in a single stage for extraction and stripping. The main types of liquid membrane systems include emulsion liquid membrane, supported liquid membrane and bulk liquid membrane. However, the liquid membrane techniques have not been adopted for large scale industrial processes primarily due to problems in maintaining its stability.

Emulsion liquid membrane (ELM) was invented by Li [11] to separate hydrocarbons and this technique has been utilized for many other applications such as metal extraction [5,6], wastewater treatment [6] and bio-medical separation [9].

Stability of emulsion is a major concern in the effective use of ELM either in laboratory scale or industrial scale. The resistance to rupture of liquid membrane at high shear stress defines the stability of the emulsion liquid membrane. Repeated coalescence of

the internal droplets on the interface, creaming due to density difference, Ostwald Ripening and flocculation cause the instability of the emulsion. Several techniques to overcome the stability problem have been proposed, and these include the use of aliphatic solvent instead of aromatic solvent [12], the increment of the carbon chain length of the aliphatic solvent [13], the increment of the surfactant concentration [14], the increment of membrane viscosity [13,15], the use of co-surfactants [16], non-Newtonian conversion of the membrane phase [17], the use of Janus particles as stabilizers in emulsion polymerization [18] and the use of functionalized silica particles for high internal phase emulsion [19]. All of the remedies have their own tradeoffs and compromises with the overall extraction efficiency.

Room temperature ionic liquids (RTILs) possess unique and exceptional properties such as negligible vapor pressure, inflammability, thermal stability even at high temperatures, highly polar yet non-coordinating solvent and application based adjustable miscibility/immiscibility in chemical processes [20–25]. These properties have made them potentially useful in a wide range of applications in industries as well as in research. Ionic liquids possess a very negligible vapor pressure that has enabled them to be used as a “green solvent” in synthesis [23,24,26–28], separation and purification [29–34], and electrochemical applications [35]. RTILs being stable and in the liquid form at room temperature, are made of organic cation and organic/inorganic anion. The physical and chemical properties of RTILs can be altered by changing the cation or anion or both to facilitate a particular task, hence they are

\* Corresponding author. Fax: +60 3 79675319.

E-mail address: [alishashim@um.edu.my](mailto:alishashim@um.edu.my) (M.A. Hashim).

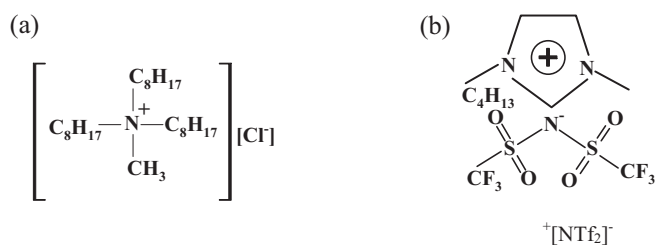


Fig. 1. Structures of TOMAC and [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup>.

sometimes referred as “task specific” ionic liquids [24]. However, very few studies have been reported on the application of ionic liquids in emulsion liquid membrane. Hence, an effort to incorporate ionic liquids with emulsion liquid membrane has been made by investigating the stability and % removal efficiency of emulsion liquid membrane in the presence of ionic liquids. Chromium metal was selected to investigate the removal efficiency of emulsion liquid membrane.

The present study focuses on enhancing the stability of ELM, identification of the role of a hydrophobic ionic liquid [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> in it and extraction efficiency of the ELM. Ionic liquid [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> was chosen over other ionic liquids due to its hydrophobicity, minimum toxicity, relatively less viscosity and density.

## 2. Materials and methods

### 2.1. Chemicals

Ionic liquids [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> and TOMAC, with structural formulae illustrated in Fig. 1, were directly obtained from Merck (Germany) and used without any further purification while kerosene of boiling point ranged from 180 °C to 280 °C was received from ACROS (USA). Span 80 (Sorbitan oleate or Sorbitan (Z)-mono-9-octadecenoate) a non-ionic surfactant with a ratio of 4:3 of hydrophilic to lipophilic (HLB), was purchased from Merck (Malaysia). Sodium hydroxide pellets, potassium dichromate and hydrochloric acid were procured from R&M Chemicals (UK). The solution of sodium hydroxide of desired normality was prepared by dissolving appropriate weight of pellets in de-ionized water. Similarly, Cr solution of 500 mg/L was prepared by mixing suitable amount of potassium dichromate in de-ionized water. The prepared Cr solution was diluted with de-ionized water according to the required concentration.

### 2.2. Analytical instruments

An ICP-spectrophotometer (Perkin Elmer, model: Optima 7000 DV) was used for the measurement of the Cr concentration. The emulsion was prepared using a high speed homogenizer (IKA, model: T25 digital Ultra Turrax) and the dispersion of the emulsion in the feed phase was carried out by a stirrer (IKA, model: RW11 Lab Egg). pH values were measured using a CyberScan 510 pH meter while photographs were taken using a digital camera (NIKON, model: DSLR D3000). Surface tension was measured by a tensiometer (Fisher Scientific, model: Tensiomat 21<sup>®</sup>) using a Pt/Ir Du Noüy ring.

### 2.3. Procedure

#### 2.3.1. Preparation of emulsion and stability analysis

The emulsion was prepared in a 100 mL un baffled beaker by mixing organic solvent and an appropriate amount of non ionic surfactant Span 80. Subsequently, the carrier and ionic liquid (sta-

**Table 1**  
Physical and thermodynamic properties of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> [36].

Property	Temperature (°C)	Value
Density (g/ml)	25	1.43
Viscosity (cP)	25	52
Surface tension (dyne/cm) (Water equilibrated <sup>a</sup> )	25	36.8
Thermal decomposition temperature (°C) (Water equilibrated <sup>a</sup> )		394
Water content (mg/l) (Water equilibrated <sup>a</sup> )	25	3280
Melting point (°C) (Dried <sup>b</sup> )		4

<sup>a</sup> Water equilibrated denotes that [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> was kept in contact with water.

<sup>b</sup> Dried stands for water equilibrated [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> that was dried at 70 °C for 4 h on a vacuum line.

bilizer) were added to the mixture. The mixture was homogenized for up to 5 min by the homogenizer at 8400 rpm. NaOH was added drop-wise into the mixture, keeping the whole mixture homogenized for the next 5 min. The ratio of internal to organic phase (I/O) was kept at 1:3 for all the experiments. The surfactant concentration (Span 80) (wherever applicable) was kept 3% (w/w) which is an optimized concentration in order to avoid swelling and to provide sufficient stability. Photographs of the beaker containing the emulsion were taken at regular intervals to analyze its stability. The photographs were analyzed by AUTOCAD to determine the phase separation rate of the emulsion.

#### 2.3.2. Extraction of chromium

The prepared emulsion was poured into another 250 mL beaker containing the Cr solution of 100 mg/L. The ratio of emulsion to feed phase (E/F) was kept at 1:2 for all the extraction experiments. The pH of the feed phase was maintained below 1.5 to establish a pH difference between the internal and external phases, hence maintaining a driving force for Cr to diffuse through the membrane. The whole mixture was gently stirred by a mechanical stirrer, and an agitation speed of 300 rpm was found to be the best to generate fine globules of emulsion with lowest possible breakage. Samples were taken at a regular interval using disposable syringes and the syringes were kept left undisturbed for some time until the emulsion and the feed phase were separated. The feed phase was then taken out, filtered and analyzed using ICP-spectrophotometer.

## 3. Results and discussion

Physico-chemical properties of this ionic liquid are as shown in Table 1.

### 3.1. Identification of the role of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> in emulsion without TOMAC

As the first stage of this study, the role of ionic liquid [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> is conjectured to behave as one of the following: either as a carrier, surfactant, solvent or stabilizer. In order to substantiate its role, the following experiments were conducted and discussed below.

#### 3.1.1. Consideration of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> as a carrier

In order to identify the role of [BMIM]<sup>+</sup>[NTf<sub>2</sub>]<sup>-</sup> as a carrier, emulsion was prepared by taking kerosene as solvent, Span 80 as surfactant, NaOH (0.1 N) as internal phase and varying amount

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