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The use of the emulsion liquid membrane technique to remove copper ions from aqueous systems using statistical experimental design

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ABSTRACT

Process water from mineral processing plants may contain a significant amount of dissolved metal ions, which may be recycled as process water, or discharged into the environment as effluent. If they are discharged into the environment they may cause significant problems for the local flora and fauna. One method of removing metal ions from aqueous systems, which has generated considerable interest over recent years, is the emulsion liquid membrane (ELM) technique, which incorporates solvent extraction and stripping. This work details the use of design of experiments applied to an ELM process for removing copper ions from a dilute aqueous solution. Initially, a fractional factorial design was used to screen out the most important factors; this was followed by a central composite design to obtain optimal operating conditions. The extraction percentage of copper was obtained as more than 99% under these conditions.
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1. Introduction

Every year a huge volume of waste water is discharged by the mining, metallurgy and smelting industries; these waste streams may contain significant amounts of heavy metal ions (Wan Ngah and Hanafiah, 2008; Fu and Wang, 2011). These metals ions should be recovered for both environmental and economic considerations. Thus, the removal of heavy metal ions from waste water has been an important topic for the past several decades. For waste streams released by the mining industry, acid mine drainage (AMD) is considered to be the biggest problem (Akcil and Koldas, 2006; Johnson and Hallberg, 2005).

There have been various ways for recovering heavy metal ions, the most commonly used methods being ion-exchange (IX) (Kang, 2004; Alyüz and Veli, 2009), adsorption (Jusoh et al., 2007; Bhattacharyya and Gupta, 2008; Babel and Kurniawan, 2003), solvent extraction and chemical precipitation (Ku and Jung, 2001; Huisman et al., 2006; Özverdi and Erdem, 2006). However, when facing AMD, these methods have some disadvantages. The concentration of heavy metal ions found in the drainage is usually less than 500 ppm (Kentish and Stevens, 2001), and a significant amount of reagents are usually required for treating such large scale waterbodies. Thus an effective way for extracting low concentration of heavy metal ions and requiring fairly lower cost must be developed.

One potentially effective method of recovering these heavy metal ions is the emulsion liquid membrane (ELM) technique (Kislik, 2009; Draxler et al., 1988; Ooi et al., 2015). This technique was introduced by Li in 1968 and has been researched ever since (Li, 1971; Li et al., 2011; Garti, 1997; Valenzuela et al., 2005, 2009; Yasser and Ibrahim, 2012; Kumbasar, 2010; Draxler and Marr, 1986). It works by forming a primary water in oil (W/O) emulsion as the first step in which the internal phase contains the stripping acid, and the oil phase contains the surfactant and metal extractant. The primary W/O emulsion is then sent for treatment with a solution containing heavy metal ions, and emulsion liquid membrane globules are formed during this process. In this project, the ELM globules are water (H_2SO_4 liquor) in an oil phase (copper extractant & surfactant in kerosene) in water ($CuSO_4$ solution) emulsions. The interfaces between water and oil phases are termed liquid membranes. The copper ions are extracted by the copper extractant in the oil phase and further stripped into the internal H_2SO_4 droplets. A schematic representation of extraction of copper ions using ELM technique is shown in Fig. 1.

The copper extractant is represented by HA, and CuA_2 represents the copper-extractant complex (Garti, 1997; Valenzuela et al., 2005). Copper ions will be extracted, stripped and concentrated at the same time (Valenzuela et al., 2005, 2009; Yasser and Ibrahim, 2012). After extraction, the water phase (effluent) will be sent for further treatment such as acid neutralization prior being discharged into environment; the emulsion phase will be sent for de-emulsification such as electrostatic coalescence (Sastre et al., 1998). The de-emulsification process will separate

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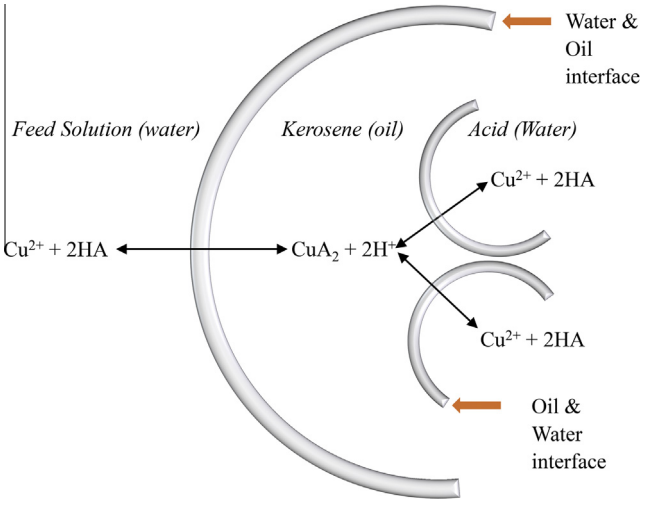

Fig. 1. A schematic representation of extraction of copper ions using ELM technique.

the oil phase from the stripping liquor phase. The stripping liquor which is loaded with copper ions will be sent for metal deposition such as electro-winning, and the oil phase will be recycled. Fig. 2 shows a schematic of the ELM process.

Economic evaluation has indicated that ELM extraction of copper using LIX 64N could be 40% cheaper than traditional solvent extraction (Frankenfeld et al., 1981), and many researchers have studied the practical operation of ELM technique recovering heavy metal ions for the past few decades (Cho et al., 1991; Wiencek and Hu, 2000; Kumbasar, 2009, 2008; Fouad, 2008; Ahmad et al., 2011; Wang, 1994; Vasconcelos and Carvalho, 1993; Uddin and Kathiresan, 2000). In this work, an investigation of the effects of operating parameters on copper extraction by ELM has been performed. Initially, a fractional factorial design was used to screen out the most important factors and the interactions between these factors were investigated. Then, a central composite design was used to obtain optimal lab operating conditions. Validation tests were performed to confirm the optimal operating conditions. Based on these results, the principles and theories behind this process were discussed.

2. Experiments

2.1. Materials

For the primary water in oil emulsion, kerosene (reagent grade, Sigma-Aldrich (USA)) was selected as the organic solvent. LIX 984N (BASF) was selected as the copper extractant, which was a 1:1 volume blend of LIX 860N-I (5-nonylsalicylaldoxime) and LIX 84N-I (2-hydroxy-5-nonylacetophenone oxime) in a high flash point hydrocarbon diluent. Sulfuric acid (98%, H_2SO_4) was purchased from Fisher Scientific (USA). Sorbitan monooleate (Span-80) was purchased from Sigma-Aldrich (USA) and used as the surfactant stabilizing the primary water in oil emulsion. It is a yellow viscous liquid with a hydrophilic/lipophilic balance of 4.3. Anhydrous cupric sulfate ($CuSO_4$) was purchased from Fisher Scientific (USA) to prepare $CuSO_4$ solution. All the aqueous phases were prepared using reverse osmosis (RO) purified water (pH of 5.8 at 25 °C). The pH of the $CuSO_4$ solution was adjusted using 1 M sulfuric acid and 1 M sodium hydroxide accordingly.

2.2. Experimental protocols

2.2.1. Primary W/O emulsion

In order to prepare the primary water in oil emulsion, H_2SO_4 solution was slowly added into kerosene during the process of ultra-sonication. The ultra-sonication was performed using a 24 kHz processor (Hielscher, Germany, UP400S). The ultrasonic horn tip was immersed in the oil layer before the sonication was turned on at the lowest power level (80 W). Cold water was

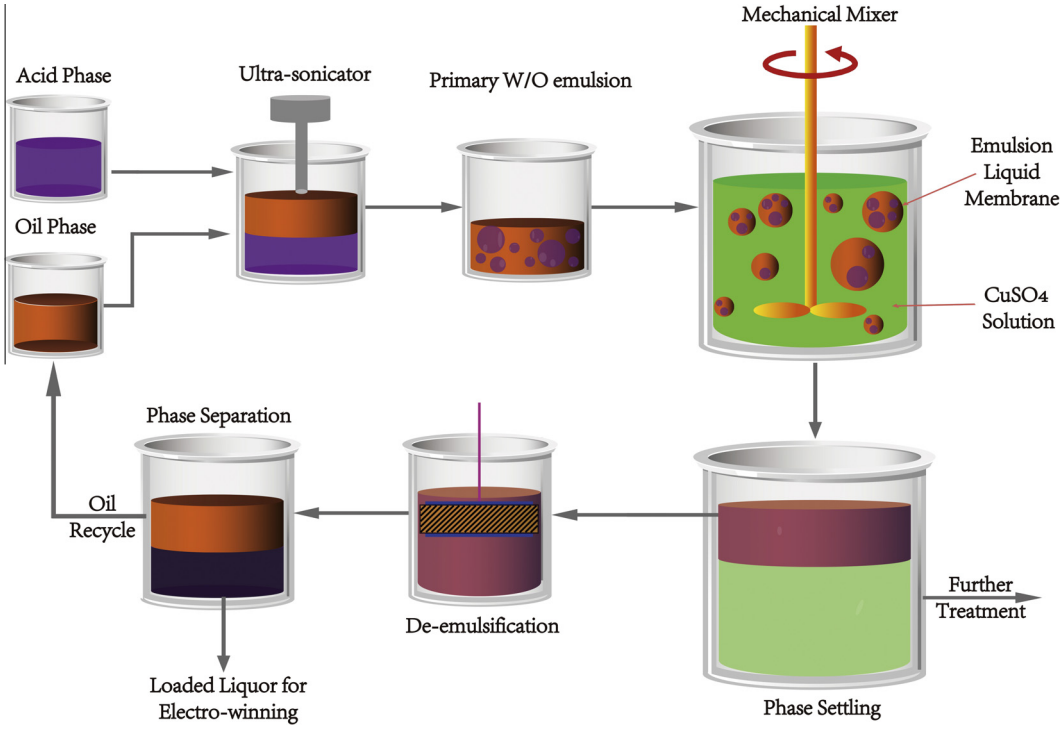

Fig. 2. A schematic of the ELM process.

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