



Copper ion removal from dilute solutions using colloidal liquid aphrons



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ABSTRACT

The extraction of copper (II) from dilute aqueous solution by predispersed solvent extraction (PDSE) using colloidal liquid aphrons (CLAs) is described. The CLAs were made of LIX 984N as an extractant and an anionic, cationic, and nonionic surfactant. It was observed that the CLAs were in the micrometer size range, and that their size was controlled by the phase volume ratio. The phase volume ratio and extractant concentration in CLAs structure are the most effective parameters in this extraction process. CLAs were able to remove copper ions in less than one minute, and they remained stable for more than two months. The effect of surfactant type and concentration was also investigated, with the anionic surfactant found to produce the best results.

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1. Introduction

Industrial effluents discharged from the mining and metallurgical plants are considered to be a major environmental challenge. These effluents often contain dissolved heavy metal ions, usually at low concentrations and low pH. The release of heavy metal bearing effluents into the environment, whether intentional or otherwise, can lead to serious problems for the local environment because heavy metals, unlike organic contaminants, are not biodegradable and tend to be toxic or carcinogenic [1]. For example, excessive amounts of copper in water can cause health problems such as vomiting, convulsions, cramps, or even death [1]. Therefore, strict environmental regulations and increasing global demand for heavy metals have led researchers to study heavy metal removal techniques. A diverse range of physicochemical techniques have been proposed for removing heavy metals from waste water including: solvent extraction [2–8]; chemical precipitation [9–12]; electrochemical treatment [13–18]; ion exchange and adsorption [19–24]; membrane filtration [25–28]; and biosorption [29–31].

Amongst the various techniques, solvent extraction appears to be the most attractive, not only for its simplicity and versatility [32–35], but also for its ability to treat a wide range of concentrations and its selectivity [36]. It is also termed liquid–liquid extraction and is based on the transfer of a solute substance (metal ions) from one liquid phase (aqueous phase) into another liquid phase (organic phase), according to solubility. However, solvent extraction is most effective for treating relatively high concentrations (>500 mg/L) [37] and requires a large distribution ratio in the

treatment of dilute streams, which causes an increase in the organic phase volume to an unacceptable value, both from environmental and economic considerations [38].

Colloidal solvent extraction using colloidal liquid aphrons (CLAs), or predispersed solvent extraction (PDSE), is a modified solvent extraction technique which was first proposed by Sebba [39]. The technique was proposed in order to overcome major disadvantages of conventional solvent extraction methods such as: need for mixing–settling stage; high solvent/aqueous ratio; and need for high initial metal concentration. Michelsen et al. [40] reported that the effectiveness of this method for the removal of an organic compound (o-dichlorobenzene) from water was 5 to 10 times greater than conventional solvent extraction using the same quantity of the solvent, and that there was a considerable drop in solvent to feed ratio (1:500) [40]. In another study carried out by Wallis et al. [41], the PDSE method resulted in 20% higher ethanol extraction with CLAs in comparison with conventional solvent extraction.

In PDSE, organic solvents including the extractant are predispersed into micrometer sized globules termed colloidal liquid aphrons, which are believed to consist of an oil core encapsulated by a thin soapy (aqueous) shell as defined by Sebba [42]. This soapy shell makes the CLAs hydrophilic which allows them to disperse easily in the feed phase. The unique structure of CLAs makes them highly stable systems and their small size provides large interfacial area available for mass transfer. They show a significant resistance to coagulation and can be stored for long periods of time without any noticeable coalescence, and can also be transported by pumping without destroying the structure [42,43]. These properties make CLAs of interest for separation processes such as removal of metals [44,45]; extraction of antibiotics [46] and organic pollutants [47,48]; oil recovery [39,42]; and water remediation [49]. For

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a recent review of aphron research, please see Molaei and Waters (2015) [50].

The present work deals with the PDSE of copper from dilute solutions and investigate the effect of various experimental parameters on the extraction behavior as well as basic characteristics of CLAs.

2. Materials and methods

2.1. Materials

Copper (II) solutions were prepared by dissolving anhydrous cupric sulfate (CuSO_4) (Fisher scientific, India) in reverse osmosis purified water. For the CLA dispersion formulation, the oil phase comprised of Kerosene (Sigma–Aldrich, USA), LIX 984N (BASF, Germany), and the nonionic surfactant Tergitol 15-S-3 (Talas, USA) as the diluent, extractant and oil soluble surfactant, respectively. LIX 984N reagent is a 1:1 volume blend of LIX 860N (5-nonyl salicylaldoxime) and LIX 84 (2-hydroxy-5-nonylaceto-phenone oxime with the specific gravity of $0.935\text{--}0.955\text{ g/cm}^3$ at $25\text{ }^\circ\text{C}$ and the flash point greater than $76\text{ }^\circ\text{C}$). Fig. 1 shows the chemical structures of LIX 84 and LIX 860N.

The anionic sodium n-dodecyl sulfate (SDS) (99%, Avocado, UK), cationic hexadecyltrimethylammonium bromide (CTAB) (98%, Sigma, India) and non-ionic polyethylene glycol sorbitan monostearate (TWEEN 60) (Sigma, USA) were also used as water soluble surfactants in the formation of the aqueous phase. 1 M sulfuric acid (Fisher Scientific, Canada) and 1 M sodium hydroxide (Fisher Scientific, Canada) were used to modify pH and 4% nitric acid (Sigma–Aldrich, USA) was used to dilute samples before copper concentration analysis. All aqueous phases were prepared from reverse osmosis purified water at ambient temperature.

2.2. Preparation of CLAs

The CLA dispersion was prepared by the slow drop-wise addition of the oil (organic) phase containing LIX 984N, kerosene, and oil soluble surfactant into a foaming aqueous solution containing a water soluble surfactant. The aqueous phase was placed in beaker and mixed at a rate of 800 rpm for 10 min to make initial foams, followed by the gradual addition of the organic phase at a flow rate of 2 mL/min to the mixing aqueous phase, until the desired phase volume ratio was reached ($\text{PVR} = V_{\text{org}}/V_{\text{aq}}$). The resulting CLA dispersions were viscous and had a creamy white appearance, showing no phase separation over a period of days. The schematic view of the CLA generation process is shown in Fig. 2.

2.3. Size measurement and microscopic analysis

The size distribution of each the CLA dispersion was determined using a particle size analyzer, LA 920 (Horiba, Japan), using a refractive index of 1.44. Microscopic observations were obtained

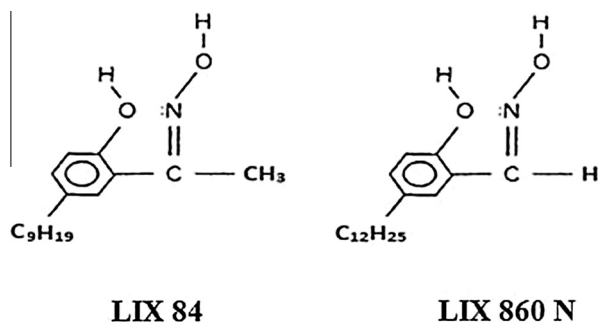


Fig. 1. Chemical structure of LIX 84 and LIX 860N. Adapted from [51].

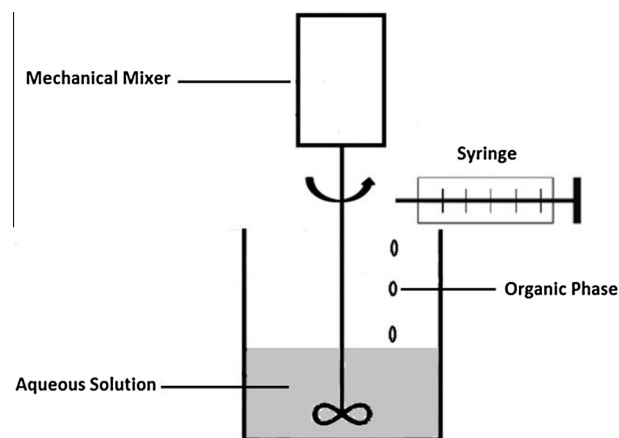


Fig. 2. CLAs generation process.

using light microscopy (Leitz Laborlux 12 Pol, Germany). CLA dispersions were generated with three different phase volume ratios, and then diluted with water (1:9), in order to reduce the turbidity of dispersions and have a clear distribution. Other parameters including the initial extractant concentration, anionic surfactant concentration, stirring time and speed, pH, and temperature were kept constant.

2.4. Extraction process

Copper extraction experiments were conducted in 50 mL centrifuge tubes, 6.5 g (equal to 10 mL at a PVR of 4) of CLAs were added to 10 mL of Cu (II) solution. The mixture was mixed for 30 s followed by phase separation in a centrifuge (5 min, 4400 rpm). After centrifuging, the raffinate was removed and diluted with 4% nitric acid in order to analyze the remaining copper concentration with inductively coupled plasma emission spectroscopy (ICP-ES, Thermo Scientific 6000 series). Ultimately, the percentage of copper removed was calculated using Eq. (1):

$$\text{Cu removal \%} = \frac{(C_0 - C)}{C_0} \times 100 \quad (1)$$

where C_0 is the initial concentration of copper (ppm) and C is the final concentration of copper (ppm) in the raffinate, as measured by ICP-OES. All the experiments were repeated a minimum of 4 times to ensure repeatability, and errors calculated at the 95% confidence level.

2.5. Phase separation

Phase separation experiments were performed in a column with a diameter of 5.7 cm and a height of 35 cm (Fig. 3). 150 mL of Cu solution was placed in column followed by gradual addition of 150 mL of CLAs to column with the rate of 7.5 mL/min. After addition of CLAs, phase separation process started and samples were taken over different time intervals. These experiments were carried out under two different conditions: introducing air using a glass frit for bubble generation at the bottom of column (air flow rate: $40\text{ cm}^3/\text{min}$); or without air, the same as the conventional solvent extraction process.

3. Results and discussion

3.1. Size and structure of CLAs

CLA dispersions were prepared using 0.5% (w/v) LIX, 0.24% (w/v) SDS, and 0.1% (v/v) Tergitol in three different phase volume

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