



Application of pseudo-emulsion based hollow fibre strip dispersion (PEHFSD) for the recovery of copper from sulphate solutions

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ABSTRACT

The transport of copper from acidic sulphate solutions using pseudo-emulsion based hollow fibre strip dispersion (PEHFSD) technology was investigated. The aldoxime Acorga M5640 was used as an extractant. The influence of several parameters like the flow rates, the initial concentration of Cu(II), the extractant amount, the pH, the presence of sodium sulphate, the use of a leach solution with Zn(II) and Fe(III) as the feed phase and lean electrolyte as strippant was studied. The analysis was made on the basis of the overall mass transfer coefficient of copper. This coefficient was also estimated by applying the resistance in series approach, the contribution of each resistance to the overall resistance to mass transfer being evaluated. Some experiments with zinc leach solution on emulsion liquid membranes (ELMs) were also conducted to compare the results of ELM and PEHFSD.

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1. Introduction

Separation and recovery of metals using liquid membrane technology, a promising alternative to conventional solvent extraction, particularly in case of dilute solutions, have been investigated by many researchers [1–16]. Although many configurations of liquid membranes (LMs) received deserved attention in recent years, but supported liquid membranes, particularly in hollow fibre and spiral wound modules, present the advantage of high surface area to volume ratios. However, emulsion liquid membranes (ELMs) technique, where extraction and stripping occur concurrently in single operation, also allows removal and recovery of solutes due to high surface area to volume ratios but the problems of emulsion stability and membrane swelling have constrained their commercial potential [13].

Recently, a number of different configurations using same hollow fibre module for extraction and stripping have been reported, namely; hollow fibre supported liquid membrane (HFSLM), hollow fibre contained liquid membrane (HFCLM) and the latest smart techniques with strip/organic dispersion methodologies, like hollow fibre renewal liquid membrane (HFRLM) and pseudo-emulsion fibre strip dispersion (PEHFSD) [17]. This new technology is attracting much scientific interests [7–12,14,15], as it presents the coupled advantages of emulsion liquid membrane and non-dispersive solvent extraction [13], where extraction and stripping oc-

cur simultaneously in a single hollow fibre contactor without membrane stability issues. In PEHFSD, a pseudo emulsion of organic and stripping phase runs through the shell side while the aqueous feed phase flows in the lumen side. The solute is transported from the feed to the membrane and then to the stripping phase simultaneously. As soon as the process stopped, the strip phase and organic phase get separated.

In literature, some reports regarding the recovery of Au(I) [7,15], Co(II) [14], Cr(III) [9], Cr(VI) [10], Fe(III) [11], Ni(II) [12] and U(VI) [8] are available, but no data on the recovery of copper using the PEHFSD technique has been published. However, an overview of current status of copper consumption and its supply worldwide indicates that copper production from primary sources is not keeping tempo with its continuously increasing demand and thus, it becomes necessary to explore and/or improve new hydrometallurgical processes to recover/recycle copper from wastes or secondary sources to meet the requirements. Therefore, the present work was aimed to recover copper from aqueous solutions by PEHFSD using a single hollow fibre module for extraction and stripping.

Based on previous studies [18,19], Acorga M5640 was selected as the extractant for the present study. Acorga M5640 is a commercial extractant, manufactured by Cytec, whose active substance is 5-nonyl-2-hydroxy-benzaldoxime. The extractant is very selective for Cu(II) over Fe(III) [20], which is a prerequisite characteristic of an extractant in case of a mixture of two or more competitive solutes. In fact, previous studies [18,19] demonstrated the efficiency of this reagent on the separation and recovery of copper from the

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pregnant leach solution obtained by acidic ferric sulphate leaching of a sphalerite concentrate. The approach of the present work includes the study of various experimental parameters like flow rates, metal concentration in feed, pH, extractant concentration, variations in volume ratio of feed to stripping and use of leach liquor with other metals, i.e., zinc and iron, as the feed phase. The modelling of mass transfer was performed and the overall mass transfer coefficient of permeation was related to the overall mass transfer coefficient of single extraction to analyse the contribution of each resistance to mass transfer. Finally, some experiments were carried out using the ELM technique to compare the efficiency of this liquid membrane technology with that obtained with the target technique PEHFS. D.

2. Experimental

2.1. Materials and methods

The aqueous solutions were prepared by dissolving the suitable reagents CuSO_4 , H_2SO_4 , Na_2SO_4 , $\text{Fe}_2(\text{SO}_4)_3$ and ZnSO_4 in deionised water. All the chemicals were of high purity analytical grade.

The solvent extraction reagents Acorga M5640 and ShellSol D70 (aliphatic diluent; aromatic content <0.01%) were received from Cytec (Netherlands) and Drogas Vigo (Portugal), respectively. A fatty ester, 2,4,4-trimethyl 1,3-pentanediol di-isobutyrate, was present in Acorga M5640 as a modifier. The amount of active reagent of Acorga M5640 was determined by using the maximum uptake of copper and was found 57%. Isotridecanol was obtained through the courtesy of Cognis (Ireland).

The pH of the fee solution was varied by adjusting the amount of H_2SO_4 added and its measurement was carried out with a pH meter from Metrohm (Metrohm 827) using a Metrohm 6.0228.010 glass electrode.

The aqueous solutions were analysed for metals concentration by AAS using a Perkin Elmer-AAAnalyst 200. A few organic phases were analysed using the Spectrometer 1100 from Perkin Elmer.

2.2. PEHFS. D. experiments

The experimental setup used for carrying out PEHFS. D. experiments is shown in Fig. 1. The same setup was used for copper extraction using single hollow fibre module.

In this schematic diagram HF represents the microporous fibre module, which was a Liqui-Cel® Extra-Flow 2.5 in. \times 8 in. membrane contactor from Celgard (USA). Further module details are given in Table 1.

The aqueous strip solution was dispersed in the organic membrane solution containing the extractant reagent. The pseudo-emulsion was then pumped into the membrane module flowing through the shell side of the fibres. The aqueous feed solution containing the target species to be extracted flowed through the lumen (tube side) of the fibres. The continuous organic phase of the dispersion readily wetted the pores of the hydrophobic microporous support (e.g., microporous polypropylene hollow fibres in the module), and a stable liquid membrane (the organic phase) supported in the pores was formed. A low pressure differential (minimum 30 kPa in this system) between the aqueous feed solution side and the strip dispersion side was applied to prevent the leakage of the organic solution of the strip dispersion to the aqueous side through the pores. The feed and strip dispersion phases were in recycling mode in their respective reservoir. As soon as the process stopped, the strip phase and organic phase get separated. All the tests were carried out at 25 °C.

The volume of pseudo-emulsion phase used in the experiment was 800 mL (400 mL of organic phase + 400 mL of stripping solu-

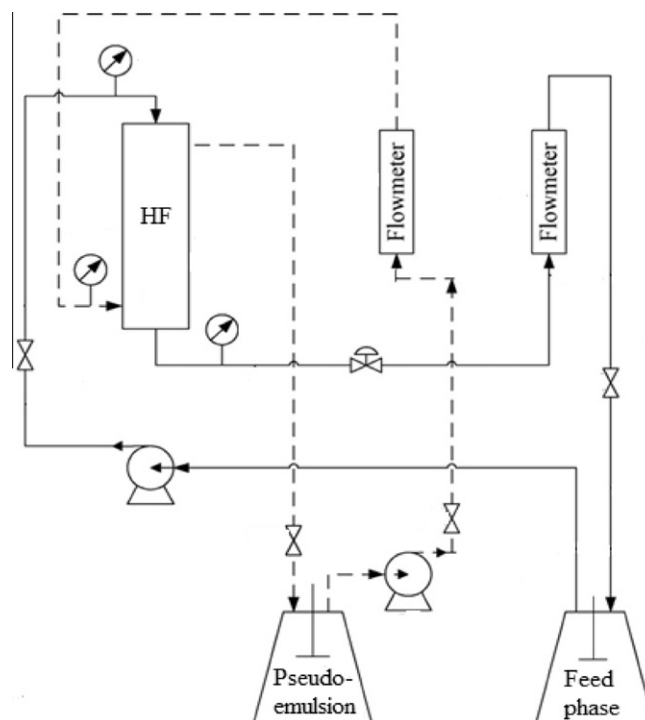


Fig. 1. PEHFS. D. experimental setup, a schematic representation.

Table 1
Characteristics of hollow fibre membrane module.

Type of module	G501 (contactor)
Module length (cm)	28
Module diameter (cm)	7.7
Case inner diameter (cm)	5.55
Centre tube diameter (cm)	2.22
Number of fibres	~10,800
Fibre	X50 – polypropylene
Effective fibre length (cm)	15.6
Inner diameter of the fibres (μm)	214
Outer diameter of the fibres (μm)	300
Pore size (μm)	0.03
Porosity (%)	40
Tortuosity	2.6
Inner interfacial area (m^2)	1.13
Area per unit volume (cm^2/cm^3)	28

tion), whereas 2000–8000 mL of feed solution of 0.1–1.0 g/L Cu(II) maintained at pH 1.4 (except in the study of the influence of pH on copper transport). A few tests were carried out with a zinc leach solution as the feed phase. The stripping solution had 180 g/L H_2SO_4 or 25 g/L Cu(II) + 180 g/L H_2SO_4 . At known intervals, small aliquot of the aqueous solutions were taken to be analysed.

2.3. ELM experiments

The emulsion was prepared by mixing the stripping aqueous solution (lean electrolyte: 25 g/L Cu, 180 g/L H_2SO_4) with the organic membrane phase using a rotor–stator type high-speed disperser (IKA Ultra Turrax T50) at 7000 rpm during 10 min. The organic phase contained 2 wt.% of surfactant (ECA 4360J, Essochem.), 5–10% Acorga M5640 (Cytec) and ShellSol D70. A predetermined volume ratio of 1:1 was maintained for the organic phase to the internal stripping phase. The emulsion was dispersed in the aqueous feed phase in a baffled glass reactor with 84 mm internal diameter and 20 cm height, immersed in a water bath with tem-

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