



Studies on the copper extraction in a channel-based packed extraction device

Somen Mondal, Subrata Kumar Majumder*

Department of Chemical Engineering, Indian Institute of Technology Guwahati, Guwahati 781039, India



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ABSTRACT

The present study aims to investigate the effect of copper ion concentration in the solution, extractant concentration in the solvent, solution pH, mixture velocity of the phases and temperature on copper extraction from a copper-laden solution. Copper is separated from a mixture of copper, nickel and cadmium solution as hydroxide by changing the pH of the solution. Copper is then extracted from the aqueous phase to the organic phase using a commercially available extractant D2EHPA [Bis-(2-ethylhexyl) phosphate] dissolved in the organic phase (benzene). The effect of pH and the other parameters on the stripping process of copper from the organic phase to the acidic aqueous phase has also been studied introducing a rectangular serpentine packed millichannel as a novel extracting device. The performance study of the packed and unpacked serpentine channel reveals that extraction efficiency of the packed channel is higher (85–99%) than the unpacked channel (80–91%). The equilibrium constant for the copper extraction by D2EHPA has also been incorporated in the study. The present work will be helpful for further understanding about the separation and extraction of copper from a mixture of metal ions solution and also be useful for the development of the channel based extracting devices.

1. Introduction

Copper is an important engineering metal having wide industrial applications. Despite the importance, it can also be a toxic metal for living creatures and water pollutant having a limit of 0.05 mg/l for drinking water (WHO, 1984). Maximum recovery of copper from industrial waste effluent would be economically and environmentally beneficial. Solvent extraction is one of the attractive separation techniques used in the industries for the selective separation and purification of metals from their mixtures as well as from their ores. In the present day scenario, researchers and engineers are interested in miniaturization or scaling down approach for the chemical process intensification. An increase in the surface to volume ratio increases the reaction and mass transport rate which is significant in case of millichannel. Millichannels have the potential to overcome the shortcoming associated with the large-scale equipment by reducing the analyte to reagent volume. Along with the millichannel based and other types of extraction devices, the researchers have used the different types of extractants in the copper extraction, mainly oximes and organophosphorus compounds from the Cyanex and Lix® group of companies are shown in Table 1. The extraction efficiency of an extractant for a

particular metal ion in a mixture mainly depends on the solution pH. Therefore, the determination of the optimum pH value for a particular metal ion and the extractant pair attracted the attention of the researchers (Sridhar and Verma, 2011; Pradhan et al., 2014). When the pH of the solution is optimized for a metal ion, the extraction efficiency depends on the other parameters like extractant concentration, ratio of aqueous to organic (A/O) content, temperature and equilibrium time (Devi et al., 2014). Furthermore, the scientists have been trying to find out the effects of diluents on the extraction processes using various types of diluents like 1-hexanol, 1-heptanol and xylene for the dilution of Cyanex-272 (Biswas and Singha, 2007). Along with the optimization of the extraction parameter, selection of appropriate extractant for a metal ion is also a challenge to the researchers. In this regard the studies on the extraction efficiency of various types of extractant on a fixed metal ion considering the optimum parameters are commendable (Gupta et al., 2003; Gouvea and Morais, 2010).

The extraction efficiency of D2EHPA, Cyanex 272, LIX 63, LIX 984N, LIX 612N-LV on copper (II) extraction was investigated and concluded that D2EHPA is the best extractant for Cu(II) from the aforementioned (Gouvea and Morais, 2010). In the present study, D2EHPA has been used as the Cu (II) extractant diluted in benzene.

* Corresponding author at: Chemical Engineering Department, Indian Institute of Technology Guwahati, Guwahati 781039, Assam, India.

E-mail address: skmaju@iitg.ernet.in (S.K. Majumder).

URL: <http://www.iitg.ernet.in/chemeng/skn/home> (S.K. Majumder).

Nomenclature			
A	cross sectional area of the channel, m^2	r_p	diameter of the largest drawn circle possible inside the particle, m
A/O	ratio of aqueous to organic volumetric flow rate, –	T	temperature, $^{\circ}C$
C_m	copper concentration in the solution, $mol\ l^{-1}$	U	actual velocity, $m\ s^{-1}$
C_e	extractant concentration in the solvent, $mol\ l^{-1}$	$V_{channel}$	total volume of the channel, m^3
C_a^{in}	solute concentration in the aqueous phase at the inlet, $mol\ l^{-1}$	w	width of the channel, m
C_a^{out}	solute concentration in the aqueous phase at the outlet, $mol\ l^{-1}$	<i>Greek symbols</i>	
C_o^{out}	solute concentration in the organic phase at the outlet, $mol\ l^{-1}$	ρ	density, $kg\ m^{-3}$
$C_{a,Eqb}^{in}$	solute concentration in the aqueous phase in equilibrium with the solute concentration of the organic phase at the inlet, $mol\ l^{-1}$	μ	viscosity, $N\ s\ m^{-2}$
$C_{a,Eqb}^{out}$	solute concentration in the aqueous phase in equilibrium with the solute concentration of the organic phase at the outlet, $mol\ l^{-1}$	ε or ε_{eff}	effective porosity, –
d_H	hydraulic diameter, m	%E	percentage extraction, –
d_p or $d_{p,eff}$	average or effective particle diameter, m	%S	percentage stripping, –
h	height of the channel, m	% η	percentage efficiency, –
K_D	distribution coefficient, –	τ	residence time or interaction time of the phases, s
K_e	equilibrium constant, –	χ	volumetric flow fraction or holdup, –
$K_L a$	overall volumetric mass transfer coefficient, s^{-1}	φ_p	sphericity of the packing material, –
L	length of the channel, m	Δ_{LMC}	log mean concentration difference, $mol\ l^{-1}$
M	molarity of the solution, $mol\ l^{-1}$	[]	concentration of each species, $mol\ l^{-1}$
Q	volumetric flow rate of the liquid, $m^3\ s^{-1}$	<i>Subscripts</i>	
R_c	radius of curvature of the channel, m	m	multiphase flow or mixture flow through unpacked channel
r_c	diameter of the smallest drawn circle surrounding the particle, m	m,p	multiphase flow or mixture flow through packed channel
		o	organic phase
		a	water or aqueous phase

In the liquid-liquid extraction processes, the extent of mixing between the two phases is important as it enhances mass transfer. So, an appropriate economic design of a mass transfer device based on the mixing characteristics of the phases is required. Researchers have recommended various type extraction devices like two-impinging-jets reactor (Dehkordi, 2002), micro-batch glass extraction chamber (Diniz et al., 2004), pulse sieve plate column (Ferreira et al., 2010), dual-valve sequential injection system (Škrliková et al., 2011), T-junction micro-channel, a serpentine microchannel and a split-and-recombine micro-channel (Darekar et al., 2014; Sen et al., 2014; Singh et al., 2015) to fulfil the purposes. Further details on the various other types of reactors used for extraction processes are tabulated in Table 2.

From the literature, it is clear that a few studies on extraction have been done on the serpentine rectangular millichannel with (Su et al., 2010) and without packing materials (Darekar et al., 2014; Sen et al., 2014; Singh et al., 2015). The objective of our present study is to optimise the extraction parameters of copper after the separation of it from an aqueous phase mixture using a commercially available extractant diluted in benzene through a rectangular serpentine packed and unpacked millichannel. The present study also reveals the performance difference between the two channel types. The results from this work directly help in designing the rectangular serpentine packed bed micro-reactors.

2. Materials and experimental methods

2.1. Materials used

Copper remains as a mixture of various metal ions in the industrial effluents. Initially, lab-made mixtures of copper, nickel and cadmium of different concentrations were taken as the experimental solutions. The chemicals: sodium hydroxide (NaOH), hydrochloric acid (HCl), ammonia solution (NH_4OH), D2EHPA, benzene, copper sulphate, nickel

sulphate and cadmium sulphate, sodium sulphite and dimethylglyoxime (DMG) purchased from Sigma-Aldrich were used for the experiment.

2.2. Estimation of packed material properties

The average particle diameter ($d_{p,eff}$), effective porosity of the packed bed (ε_{eff}) and sphericity of the packing material (φ_p) are the three important parameters to govern the hydrodynamics as well as mass transfer in the packed bed. The average particle diameter was determined by the sieve of sizes ASTM No. 35 and 40 (U.S. Standard). The average particle size as per the current experiment is 462 μm . The effective porosity was calculated by the ratio of the volume of the pores to the total volume of the bed. The total volume of the bed is calculated by the channel dimensions. The wetted solid volume is measured by liquid displacement method. The pore volume is obtained by subtracting the volume of the wetted solid from the total volume of the bed. The solid is taken in wetted condition to minimize the liquid absorption during the measurement. The effective porosity estimated experimentally in the present work is 0.36. The sphericity is calculated by using the modified procedure of Krumbein and Sloss (1963). It is defined as the ratio between the diameters of the largest drawn circle possible inside the particle (r_p) to the diameter of the smallest drawn circle surrounding the particle (r_c). It is determined by analyzing a snapshot of the sand particle taken by Sony DSC-H400 Point and Shoot Camera using Digimizer[®]4 software. The mathematical form of sphericity of particle is expressed as Eq. (1).

$$\varphi_p = \frac{r_c}{r_p} \quad (1)$$

From the analysis, it is found that the sphericity of particle is 0.80, which is within the range 0.50 to 0.90 for natural sands reported by Cho et al. (2006). The holdup of each phase inside the channel was

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