Solvent Extraction of Copper Enhanced by Mixing Cavities in Micromixer

Abdul Rauf SHEIK1,3,*, Amol A KULKARNI2,3 and Kali SANJAY1,3
1CSIR-Institute of Minerals and Materials Technology, Bhubaneswar-751013, India; 2CSIR-National Chemical Laboratory, Pune-411008, India; 3Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India

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A Y micromixer with a macroscopic mixing cavity at the inlet junction was developed. The effects of the mixing cavity on the improvement of extraction of copper by LIX 84I were investigated. The investigated parameters and their ranges were: overall flow rate: 10 – 200 mL/min; microchannel size: 0.5 – 1 mm and mixing cavity diameter: 5 – 20 mm. Extraction efficiency, volumetric mass transfer coefficient, kinetics and pressure drop were investigated. The extraction efficiency and the volumetric mass transfer coefficient in the cavity micromixer were significantly higher than the Y micromixer. The volumetric mass transfer coefficient ranged between 1 – 93 s⁻¹, and the pressure drop between 50 – 500 kPa. The residence time required in the micromixers was two orders of magnitude lesser than that of an agitated batch reactor.

1. Introduction

Solvent extraction is an important and major unit operation in hydrometallurgical production of base metals such as Cu, Ni, Co, and Zn. It is required to separate the metals present in the leach liquor into individual, purified solutions suitable for downstream operations such as electrodeposition. It is most widely carried out in mixer-settlers owing to their effectiveness, ease of construction and reliability. However, there are several aspects of mixer-settler operations which are disadvantageous. Solvent loss due to entrainment and evaporation, large floor space, sizeable dead volume leading to large solvent inventory, and sensitivity to impurities are some of the disadvantages [1]. The principles of microreactors offer a direct advantage over mixer-settlers in terms of reduced floor space, reduced residence volume and thereby, reduced solvent inventory. For instance, the solvent loss in mixer-settlers is estimated to be around 90 – 20000 mg/L of the solvent volume [2]. Applying microreaction technology can significantly reduce solvent loss through low solvent inventory.

Owing to these factors, the application of solvent extraction in microreactor systems has garnered interest from several researchers (Table 1). La was extracted using 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester (P507) in parallel flow. Parallel flow was maintained in the aqueous flow rate range of 0.833-41.7 µL/s. In this range, the increasing flow rate reduced the extraction efficiency [3]. Similarly, parallel flow was employed by Jiang et al. in a Y-Y microchannel to extract copper in the range of 0.01 – 0.1 mL/min [4]. While they obtained parallel flow in various phase ratios, the phase separation at the exit Y junction was found to be perfect only at 1:1 phase ratio. Employing a smaller angle of Y at the inlet was found to increase the stability of parallel flow in the instance of Co extraction with 2-nitroso-5-dimethylaminophenol [5]. A
tripropyl-monoacetic acid derivative of calix[4]arene (1Ac) was used to extract Li from seawater employing a T-junction microchannel made of glass. Compared to a conventional batch reactor, the kinetics in the microreactor was faster by a factor of $4.35 \times 10^4$ [6]. Dai et al. reported a mass transfer coefficient and extraction efficiency of copper in circular microchannels [7]. The volumetric mass transfer coefficient obtained was between 0.02 and 0.09 s$^{-1}$. Tokeshi et al. obtained parallel flow in a glass chip microextractor for extraction of Fe with tri-n-octylmethyl ammonium chloride with residence time of 45 – 60 s between flow rates of 0.03 – 0.1 mL/min [8]. Zhang et al. obtained 5 – 7 times higher separation efficiency of Co and Ni in a micro-scale coiled flow inverter compared to a batch reactor [9].

| S.N. | Metals extracted | Flow rate | Residence time | Reference |
|------|------------------|-----------|----------------|-----------|
| 1    | La               | 0.833 – 41.7 µL/s | 0.37 s max   | [3]       |
| 2    | Li               | 0.16 – 0.87 mL/min | 2 – 10 s     | [6]       |
| 3    | Cu               | 0.01 – 0.1 mL/min  | 0.392 – 3.915 s | [4]       |
| 4    | Cu               | 0.912 – 4.812 mL/min | 20 – 160 s  | [7]       |
| 5    | Fe               | 0.03 – 0.1 mL/min  | 45 – 60 s    | [8]       |
| 6    | Co & Ni          | 0.4 – 8 mL/min    | 15 – 300 s   | [9]       |
| 7    | Co & Ni          | 22 mL/min         | 20 – 100 s   | [10]      |
| 8    | Ce & Pr          | 0.56 µL/s         | 2 – 12 s     | [11]      |
| 9    | Cu & Ni          | 1.2 mL/s          | 1 – 5.5 s    | [12]      |
| 10   | Ag, Pd, and Pt   | 8.76 – 43.8 µL/s  | 2 – 10 s     | [13]      |
| 11   | Cu, Fe, and Zn   | 2 – 25 mL/min     | 0.016 s      | [14]      |
| 12   | Nd               | 7 – 26.5 mL/min   | 90 – 150 s   | [15]      |
| 13   | Cu               | 10 – 200 mL/min   | 0.03 – 4 s   | Present work |

Table 1. Metal extraction using microreactors.

There are several evidences to give credence to the advantage of using microreactors for solvent extraction. However, the flow rate of the microreactor is required to be higher for commercial applications since industrial solvent extraction of metals is operated in very high throughput compared to low volume industries such as fine chemicals or pharmaceuticals. Therefore, in the present work, the flow rate was maintained in the range of 10 – 200 mL/min to achieve higher throughputs. The flow pattern in high flow rate applications is usually dispersed flow since slug, annular, and parallel flow regimes require strict controls over the flow parameters [16]. This fact allows high flow rate applications to exploit chaotic mixing without having to tune the flow parameters for segregated or stratified flow.

Passive chaotic mixing can be improved by the placement of mixing structures such as cavities. Lin et al. used a 1 mm 3D cavity for 1 phase mixing [17]. The use of rotational flow in the cavity was found to improve the mixing efficiency from 74% to 90%. 2D circular cavities along the length of the microchannel, placed tangentially to the channel, have been found to obtain 85 – 90% mixing efficiency at Re of 50 [18]. Through numerical simulations and experiments on mixing index, Chung et al. found that hollow mixing chambers provided 50% better mixing efficiency than straight channels without cavities and 15 – 20% better mixing efficiency than chambers with mixing structures [19]. A water-ethanol system in the range of Re: 50
– 300 was employed. Viktorov et al. combined the designs of ‘H and Chain mixers’ onto a ‘Split & Recombine’ micromixer and obtained greater than 95% mixing efficiency in the range of Re: 1 – 100 through experimentation and numerical studies [20]. In two-phase immiscible flow, Zhao et al. found that dispersed flow can be induced for better mass transfer by varying the width of the inlet junction of T micromixer [21]. By inducing a cross-flow geometry in the T micromixer, volumetric mass transfer coefficient of as high as 16 s⁻¹ was obtained for water-n-butanol system. The inclusion of passive mixing structures, particularly cavities, is an expedient way of improving the mixing due to its simple fabrication. In the present work, a spherical mixing cavity is introduced at the junction of the inlets to enhance mixing. The advantage of a mixing cavity at the inlet junction of a Y micromixer in comparison to a conventional Y micromixer is emphasized.

2. Experimental

2.1 Materials

All the aqueous solutions contained 5 g/L Cu in CuSO₄ form. The pH of the aqueous solution was not changed from its initial value of 3.8. All the organic solutions contained 5% v/v LIX 84I in distilled kerosene. Copper sulphate was procured from Merck, India and LIX 84I was procured from BASF, India. Distilled water was used for all applications. The copper concentration in the aqueous solution was measured using Atomic Absorption Spectrophotometer (Perkin-Elmer, AAnalyst-200).

2.2 Apparatus

The micromixer was fabricated by CNC milling on the surface of polypropylene (PP) plates. On the surface of the bottom plate, square microchannel with and without hemispherical cavity of the desired size was milled. On the surface of the top plate, another hemispherical cavity was milled in the case of the cavity micromixer, and it was left flat in the case of Y micromixer (Figure 1). The top plates contained SS 316, 1/4” NPT connectors for inlet and outlet. Calibrated syringe pumps (New Era, Dual NE 4000) were used to pump both the aqueous and organic solutions. Polyurethane (PU) tubes were used to connect the syringe outlet to the micromixer inlets. Pressure drop was measured by connecting a digital manometer (Cole Parmer, Traceable-98767) at the inlet junction of the aqueous stream using a SS 316 T junction. SS clamps were used to fasten the PU tubes on the connectors to avoid leakage at high pressures. Teflon tapes were used between connecting surfaces to ensure leakproof fit. The batch experiments were carried out in a 1 L glass beaker, fitted with an overhead stirrer (IKA, RW 20). The total volume filled in the beaker was 500 mL comprising of equal ratios of organic and aqueous solutions. Samples were taken at regular intervals for kinetic analysis.
2.3 Methods

The length of the micromixer was varied between 50 mm to 400 mm to obtain kinetic data. All experiments, unless specified otherwise, were conducted at 1:1 organic: aqueous phase ratio, 150 mm channel length and ambient temperature. The overall flow rate was controlled between 10 mL/min and 200 mL/min. Table 2 lists the velocities, Reynolds number and residence times at various flow rates and channel sizes used in the current study. The performance of the microreactors was characterized through extraction efficiency (Equation 1) and the volumetric mass transfer coefficient (Equation 2). The volumetric mass transfer coefficient has been calculated using the mass transfer equation from film diffusion theory [7]. Equilibrium concentration was found out by equilibrating the two phases in a separating funnel for 30 min, and the equilibrium concentration of copper in aqueous was found to be 2.54 g/L.

\[ \eta = 100 \times \frac{C_i - C_f}{C_i - C_e} \]  
\[ k_{La} = \frac{1}{t} \ln \left( \frac{C_i - C_f}{C_f - C_e} \right) \]  

where \( \eta \) is extraction efficiency (%), \( C_i \) is the initial concentration of Cu in the aqueous phase (g/L), \( C_f \) is the final concentration of Cu in the aqueous phase (g/L), \( C_e \) is the equilibrium concentration of Cu in the aqueous phase (g/L), \( t \) is residence time (s), and \( k_{La} \) is the volumetric mass transfer coefficient (s\(^{-1}\)). In order to check the extraction that may occur during the collection and separation of the outlet, mixing with zero-length channel was carried out. It was found that the Cu extraction during collection and phase separation was negligible. Therefore it was assumed that the extraction values obtained at the outlet were solely a result of mass transfer inside the micromixer.

| Overall flow rate (mL/min) | Overall velocity (m/s) | Overall Reynolds number (Re) | Total residence time for 150 mm length (s) |
|---------------------------|------------------------|----------------------------|------------------------------------------|
|                           | 1 mm | 0.75 mm | 0.5 mm | 1 mm | 0.75 mm | 0.5 mm | 1 mm | 0.75 mm | 0.5 mm |
| 10                        | 0.17 | 0.30    | 0.67   | 111  | 148    | 222    | 0.9  | 0.506  | 0.225  |
| 40                        | 0.67 | 1.19    | 2.67   | 444  | 592    | 889    | 0.225| 0.127  | 0.056  |
| 80                        | 1.33 | 2.37    | 5.33   | 889  | 1185   | 1778   | 0.113| 0.063  | 0.028  |
| 160                       | 2.67 | 4.74    | 10.67  | 1778 | 2371   | 3557   | 0.056| 0.032  | 0.014  |
| 200                       | 3.33 | 5.93    | 13.33  | 2223 | 2964   | 4446   | 0.045| 0.025  | 0.011  |

3. Results and Discussion

3.1 Extraction efficiency

The inclusion of the mixing cavity significantly improved extraction efficiency. Figure 2 indicates the variation of extraction efficiency as a function of flow rate for microchannels of 0.5, 0.75, and 1 mm side of the square, which is also its hydraulic diameter. The ratio of cavity diameter to channel size was maintained at a value of 10 in all three cases.

The function of extraction efficiency and flow rate has a flattened U-shaped trend. The effect of increasing flow rate on extraction efficiency is contingent on the opposing effects of increasing interfacial area and decreasing residence time as described by Equation 3 [22].
where $k$ is the diffusion coefficient (m$^2$/s), $t$ is the residence time (s), $r$ is the droplet radius (m), $D$ is the distribution coefficient, and $V_{org}/V_{aq}$ is the ratio of organic phase volume to aqueous phase volume.

Increase in flow rate causes a reduction in residence time and an increase in the specific interfacial area due to reduced droplet diameters. With the increase in flow rate initially, a drop in efficiency was observed due to a reduction in residence time. But with further increase in flow rate, the increased interfacial area offsets the negative effects of the reduction in residence time. Jiang et al. reported a decreasing efficiency with flow rate for copper extraction in parallel flow [4]. A similar decreasing trend was reported by He et al. for Sm extraction in a slug flow pattern [22]. These clear decreasing trends occur due to reduced residence time without an appreciable increase in the interfacial area. However, in chaotic mixing profiles, U-shaped trend analogous to the present work has been reported in different extraction systems such as water-succinic acid [24] and Co-Cyanex 272 [25].

The same confluence of residence time and interfacial area effects resulted in the variation of efficiency with diameter. A decrease in diameter increases efficiency in the Y micromixer due to higher interfacial area by way of decreased droplet diameter [26]. In the cavity micromixer, however, a greater interfacial area is generated at the mixing cavity compared to the Y micromixer. A saturation of increase in interfacial area with a reduction in diameter, combined with the reduction in residence time, resulted in the 0.75 mm cavity micromixer generating maximum efficiency. Notably, in any particular channel size and flow rates, the cavity micromixer enhanced the extraction efficiency considerably. This indicates that the presence of the mixing cavity has improved the extraction by virtue of increases interfacial area or by the increased residence time due to the additional volume in the cavity. However, the effect of flow rate on extraction efficiency (Figure 2) indicates a sensitivity towards residence time that suggests that the actual residence time is comparable to the Y micromixer. Therefore, the enhancement in efficiency was predominantly a result of increased interfacial area.
3.2 Kinetics

The kinetics of Cu-LIX 84I system in the agitated batch vessel, cavity micromixer, and Y micromixer are shown in Figure 3. At the outset, the kinetics for the micromixers was much faster than the batch reactor; the residence time required in the batch reactor was around 200 s, while in the micromixers the reaction was completed within 0.35 s. The shape of the Y micromixer curve is similar to instances reported in the literature [4,9,14]. However, the cavity micromixer has a region of slow kinetics followed by a surge in the extraction rate. The residence time in this region of slow kinetics corresponds to the theoretical residence time of the mixing cavity. The flattened kinetic curve is evidence that the liquids do not undergo complete circulation in the mixing cavity. Instead, a mechanism of dispersion inside the cavity followed by flow into the linear microchannel is more likely.

![Kinetic plots for the batch reactor, Y micromixer, and cavity micromixer (1 mm channel size, 10 mm cavity size, total flow rate = 160 mL/min).](image)

Figure 3. Kinetic plots for the batch reactor, Y micromixer, and cavity micromixer (1 mm channel size, 10 mm cavity size, total flow rate = 160 mL/min).

Figure 4 illustrates the residence time and extraction efficiency obtained along the length of the channel. The mixing cavity has a calculated residence time of 0.196 s while the square channel has 0.15 s. However, only 16.5% of the extraction takes place in the cavity, and 79.5% extraction occurs in the mixing channel. This indicates that the actual residence time of the liquids in the mixing cavity was considerably lesser than the calculated residence time. The liquids in the mixing cavity are not thoroughly mixed; instead, they form a dispersion and flow into the outlet microchannel connected to the mixing cavity. This further substantiates the similarity of efficiency curves of Y and cavity micromixer (Figure 2) since their residence times are not significantly different.

The kinetic plot was redrawn by excluding the residence time from the mixing cavity. The re-plotted kinetic curve suggests that the cavity micromixer had superior kinetics over the Y micromixer (Figure 5) due to increased mass transfer through the interfacial area. Because of this reason, the operative residence time of the cavity micromixer is approximated to the residence time of the Y micromixer for calculations in further comparative parameters.
3.3 Mass transfer coefficient

Figure 6 depicts the effect of flow rate and diameter on the volumetric mass transfer coefficient ($k_{La}$). $k_{La}$ had positive correlations with flow rate and negative correlations with the hydraulic diameter of the channel. The generation of finer dispersion at higher flow rates and smaller diameters enhances the interfacial area and thereby, the volumetric mass transfer coefficient [27]. Similar trends for solvent extraction systems such as Sm [22], Cu [7] and La [3] have been reported. Unlike extraction efficiency, the volumetric mass transfer coefficient had a clear dependence on the flow rate and diameter. It also supports that the drop in efficiency with an increase in flow rate was a result of shortened residence time and not because of reduced mixing. Therefore, the volumetric mass transfer coefficient was a better measure to characterize the micromixer. The $k_{La}$ for the cavity micromixer is higher than the Y micromixer by a factor of 2 – 3 at all comparable flow rates and channel sizes. The distinct enhancement provided by the mixing cavity can be attributed to the creation of chaotic dispersion inside the mixing cavity as the two liquid phases contact [21].
Dispersed flow has been reported to have higher mass transfer coefficients due to higher interfacial area generated [28].

The range of values obtained for the volumetric mass transfer coefficient is higher than those reported in the literature. Most of the reported values for solvent extraction of metals are in the range of $10^1 – 10^2 \text{ s}^{-1}$ [7,9,29,30]. Jafari et al. obtained values between $1 – 7 \text{ s}^{-1}$ for a Cu-D2EHPA system in the flow rate range of 1-10 mL/min in twisted microchannels of 800 µm channel width [31]. Using a water–n-butanol system, Zhao et al. reported a maximum $k_{La}$ value of 17 $\text{s}^{-1}$ in a T micromixer at Reynolds number of 100 – 650 [27].

In the present work, since high extraction efficiency was obtained at lower residence times, the volumetric mass transfer coefficient was considerably higher, in the range of $1 – 93 \text{ s}^{-1}$.

3.4 Pressure drop

The volumetric mass transfer coefficient, and by extension the mixing, is higher at increasing flow rates and decreasing diameters. But the constraint on operating at higher flow rates and lower diameters is the energy expended during pumping as indicated by the pressure drop. The pressure drop at different channel size as a function of flow rate is shown in Figure 7. The pressure drop of cavity micromixer was higher than the Y micromixer in all channel sizes by a factor of 1.3 – 2. Correspondingly, the extraction efficiency was also higher by a similar factor. The pressure drop might be contributed by two factors, the flow resistance in the microchannel and the energy required for mixing. The additional pressure drop in the cavity micromixer was substantially contributed by the additional energy required for mixing the two liquids since the microchannel length was identical. In comparison with laminar pressure drop using the Darcy-Weisbach equation (Equation 4), the experimental pressure drop was significantly higher in all the channels. The pressure energy consumed for secondary flows and interface area generation rendered the experimental pressure drop significantly greater than the theoretical Darcy-Weisbach pressure drop [32]. In work attempting scale-up, 200 – 400 kPa pressure drop was reported in the range of 0.5 – 9 L/h total flow rate for 65 – 95% of Co extraction in a 3D re-entrance [24]. Darekar et al. reported a pressure drop of 100 – 1600
kPa for extraction of nitric acid with 30% tributyl phosphate in 800 – 300 micron channels in flow rates of 0.5 L/h [25].

\[
\Delta P = f \frac{\rho L v^2}{2D_h g}
\]

(4)

where, \(\Delta P\) is pressure drop (kPa), \(f\) is the friction factor (64/Re), \(\rho\) is the density of the liquid mixture (kg/m\(^3\)), \(L\) is the length of the channel (m), \(v\) is the velocity of the flow (m/s), \(D_h\) is the hydraulic diameter (m), and \(g\) is the acceleration due to gravity (m/s\(^2\)).

Figure 7. Pressure drop across the channels as a function of flow rate and diameter.

Figure 8. Correlation between \(k_{La}\) and energy dissipation rate among all flow rates and channel sizes.

The energy dissipation rate (Equation 5) [33], correlated with \(k_{La}\) obtained under various conditions is shown in Figure 8. A positive correlation between energy dissipation rate and \(k_{La}\) was observed at all flow rates and diameters. It was also observed that the cavity micromixer was more efficient by way of consuming
lesser energy than Y micromixer to obtain the same value of $k_{La}$. This indicated that the excess pressure energy in the cavity micromixer was used for mixing of the fluids through the generation of interfacial area [33].

$$\varepsilon = \frac{Q \Delta P}{\rho V}$$  \hspace{1cm} (4)

where $Q$ is the total flow rate ($m^3/s$), $\Delta P$ is the pressure drop (Pa), $\rho$ is the mean density ($kg/m^3$), and $V$ is the volume of the micromixer ($m^3$).

4. Conclusion

The presence of a macroscopic mixing cavity at the junction of the incoming streams in a Y micromixer improved mixing and thereby extraction efficiency for solvent extraction of copper. This enhancement of efficiency was exploited to operate the micromixer at high overall flow rates of 10-200 mL/min. The volumetric mass transfer coefficient, extraction efficiency, and the pressure drop were considerably higher for the cavity micromixer compared to the Y micromixer. Kinetic analysis revealed that the residence time of the entire volume of the cavity does not take part in the extraction process, thereby suggesting that the mixing cavity served only to create dispersion and a substantial portion of the reaction proceeds in the microchannel. The volumetric mass transfer coefficient was in the range of $1 – 93 \text{ s}^{-1}$ with a pressure drop of $10 – 500 \text{ kPa}$ for a microchannel length of $150 \text{ mm}$ and channel sizes of $0.5 – 1 \text{ mm}$. The energy consumed by cavity micromixer was lesser than the energy consumed by Y micromixer, for the same $k_{La}$ value. This device can be numbered up to obtain industrial scale throughputs required in solvent extraction circuits employed in the metals industry.

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