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# Fast separation of $Cu^{2+}$ and $Ni^{2+}$ in sulfate solution by Lix984N extraction using a microchannel chip

DOI 10.1515/gps-2016-0224
Received December 7, 2016; accepted May 10, 2017;

**Abstract:** A comparison study about the extraction and separation of $Cu^{2+}$ and $Ni^{2+}$ with Lix984N in a microchannel reactor and separating funnel has been conducted. The results showed that, in the microchannel reactor, the overall volumetric mass transfer coefficient of copper was 20 times that of nickel, whereas in the separating funnel, it was only 2 times that of nickel. In addition, the separation coefficient of copper and nickel in the microchannel reactor was 5 times that of the conventional one. Typically, at initial pH = 2.5, contact time 1.95 s, volume fraction of extractant Lix984N 15% and within 1.9 g $l^{-1}$ nickel ion concentration, the extraction rate of copper was higher than 95%, but the nickel was hardly extracted. In comparison, it needed almost 50 s to reach a Cu extraction of 95% in the separation funnel, with more than 5% Ni co-extraction rate. Although the microfluid extraction showed excellent extraction performance, there exists a need to further improve its processing capacity to apply it to industrial production. Furthermore, scaling up the microreactor has become increasingly promising under the fast developing 3D printing technology.

**Keywords:** copper; Lix984N; microreactor; nickel; solvent extraction.

## Abbreviations

| | |
|---|---|
| $C$ | mass concentration of metal ions (g/l) |
| $t$ | residence time (s) |
| $V$ | volume of microchannel (ml) |
| $Q$ | volumetric flow rate (ml $s^{-1}$) |
| $J$ | mass transfer flux (g $m^{-2}$ $s^{-1}$) |
| $\Delta C_{ln}$ | logarithmic mean concentration difference (g/l) |
| $\alpha$ | interfacial area ($m^2/m^3$) |
| $K_L$ | overall mass transfer coefficient (m $s^{-1}$) |
| $K_L\alpha$ | overall volumetric mass transfer coefficient ($s^{-1}$) |

## Subscripts

| | |
|---|---|
| aq | aqueous phase |
| Cu | copper |
| eq | at equilibrium |
| in | at inlet |
| Ni | nickel |
| org | organic phase |
| out | at outlet |

## 1 Introduction

In the traditional hydrometallurgy process of copper extraction, the presence of Ni is undesirable, because nickel ions could be extracted by some copper extraction agent during the solvent extraction (SX) process [1]. Thus, efficiently separating copper and nickel has become a necessity, and a great deal of studies have been carried out using different extracting agents, such as ACORGA M5640 [1], ACORGA PT5050 [2], LK-C2 [3], LIX973N [4], and LIX87QN [5].

Although the higher extraction rate of copper was achieved in the literature, the procedures usually required very long treatment times and multiples steps. For example, Bidari et al. [6] found that the copper extraction percentage could run up to 98% when mixing the 3% oxime extractant in D2EHPA for 20 min. In addition, in the process of separation and purification, multi-stage extraction was usually needed to obtain a better separation effect [4, 7–9].

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In recent years, microfluidic extraction technology has attracted much attention, especially in the fields of medicine, biology, chemistry, and analytical testing [10–15]. In the microfluidic extraction process, the main devices are microreactors with microchannel structures, whose internal diameters are in range of 10–1000 μm [16]. Such microreactors have the following advantages: larger interface area to volume ratio and shorter diffusion distance [17, 18], safer processing [19], and no emulsification or formation of a third phase due to its laminar flow state [20, 21].

The superiority of the microreactor used in the solvent extraction of metal ions has also been confirmed by numerous research. For example, Yin et al. [22] found that the extraction rate of La(III) reached 99.5% at the following conditions: pH = 4.0, O/A phase ratio = 1:1, saponification rate 40% of P507, and volumetric flow rate $\nu_{aq} = \nu_{org} = 8.33\times10^{-10}$ m$^3$ s$^{-1}$. Furthermore, the time needed was just 0.37 s. Yang et al. [23] investigated the copper extraction characteristics in a T-Junction microchannel, and reported a 96% maximum copper extraction rate. Priest et al. [21] studied the extraction of copper from a copper sulphate solution containing silica nanoparticles. They found that emulsification appeared in the case of the conventional extraction experiments, but did not occur when the copper was extracted within a microchip.

In the current work, the extraction and separation of $Cu^{2+}$and $Ni^{2+}$ in the sulfate solution by Lix984N in a microchannel reactor and separating funnel was carried out. The effects of various parameters, such as initial pH, volume flow rate, and extractant concentration, on the extraction rate of copper and nickel were investigated, after which the extraction rate and separation factors were obtained under these conditions. The advantages of the microchannel reactor were described by comparing the separation performance with the separating funnel. Finally, an applicable approach (i.e. 3D printing technology) for scaling up the microchip system into industry field was demonstrated in detail.

## 2 Materials and methods

### 2.1 Materials

In this study, the aqueous phase used was obtained by dissolving a certain amount of $CuSO_4 \cdot 5H_2O$ and $NiSO_4 \cdot 6H_2O$ (Tianjin Zhiyuan Chemical Reagent Co., Ltd, Tianjin, China) in deionized water. The initial concentrations of $Cu^{2+}$ and $Ni^{2+}$ were detected as 2.92 g l$^{-1}$ and 1 g l$^{-1}$, respectively. Two types of dilute solution, sulfuric acid and sodium hydroxide, were used to adjust the initial pH of the aqueous solution, which was determined by a pH meter (pHs-3C, Leici, Shanghai, China).

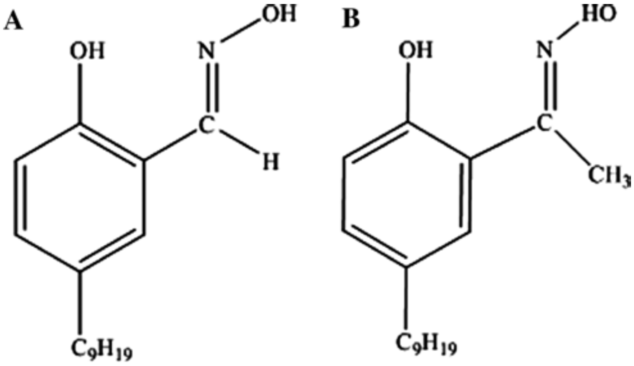

Figure 1: The structural formula of the main chemical composition of LIX984N: (A) 5-nonyl salicylaldoxime, (B) 2-hydroxy-5-nonyl acetophenone.

The organic phase was obtained by dissolving Lix984N (Zhengzhou Dezhong Chemical Reagent Factory, Zhengzhou, China) into sulfonated kerosene (Hallochem Group Co., Ltd). The main extracter, Lix984N, is actually a mixture of 5-nonyl salicylaldoxime with 2-hydroxy-5-nonyl acetophenone at the same volume, and their structural formulas are shown in Figure 1. The product obtained after extraction is a chelate, as shown in Figure 2.

### 2.2 Microfluidic device

The microfluidic experimental apparatus employed in this work is shown in Figure 3A. The microchip consisted of two pieces of Plexiglas bonded together by thermal compression. A set of 2 “Y” type microchannels, illustrated in Figure 3B, were made previously at the side of the chip by machining. Two microchannels converged at a Y-junction and then merged into a single microchannel where the extraction reaction took place on one piece of Plexiglas. The structure size of the microreactor is shown in Figure 3C and Table 1.

### 2.3 Microchannel reaction extraction

Organic phase and aqueous solution were pumped into the microchannel reactor via two inlets at the same flow rate by a programmable

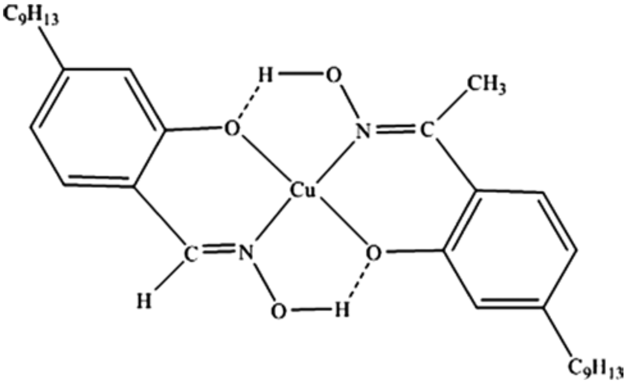

Figure 2: The structural formula of the product after chelating extraction.

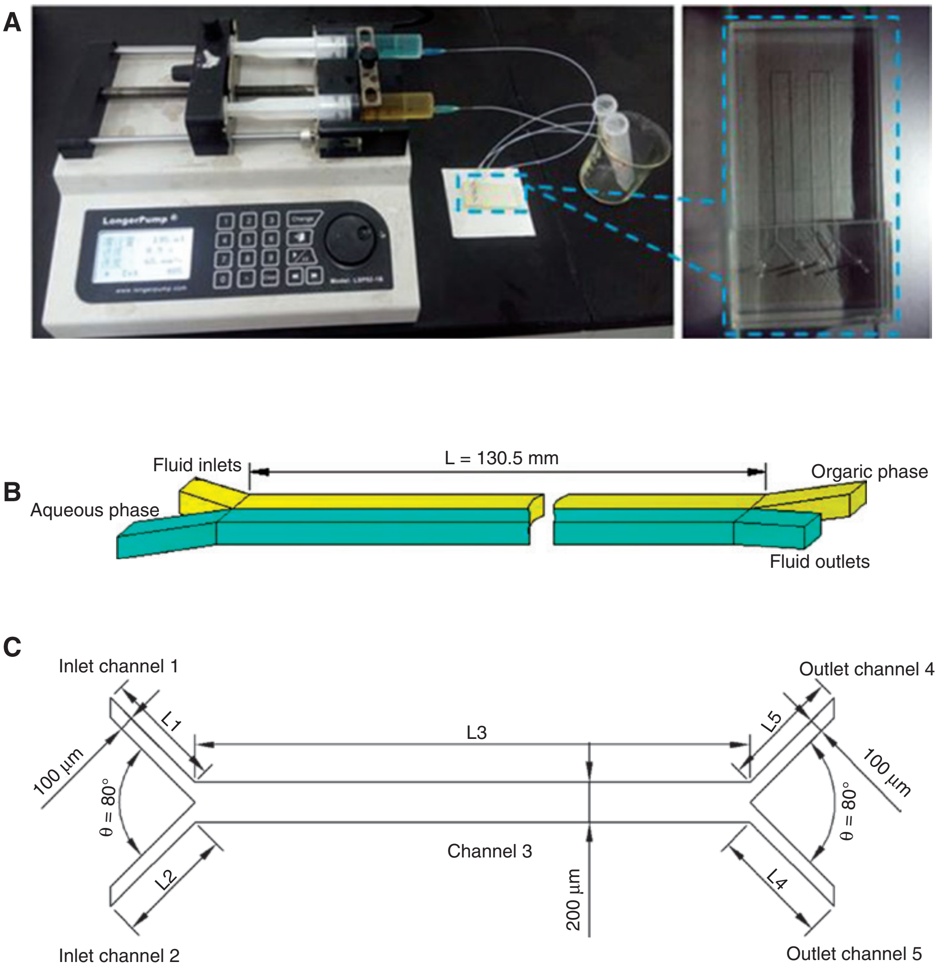

**Figure 3:** Schematic illustration of the experimental apparatus: (A) diagram of experimental apparatus; (B) and (C) two phases microchannel schematic.

**Table 1:** Specifications of the microreactor used in the study.

| **Channel length (mm)** | | | **Channel cross section (μm × μm)** | | | **Angle between inlet channels 1 and 2 or 4 and 5, θ/(°)** |
|---|---|---|---|---|---|---|
| **L1 and L4** | **L2 and L5** | **L3** | **Inlet channels 1 and 2** | **Channel 3** | **Outlet channels 4 and 5** | |
| 5 | 5 | 130.5 | 100 × 50 | 200 × 50 | 100 × 50 | 80 |

syringe pump (Harvard, PHD 2000-M). The two phases came into contact at the first Y-junction and then flowed into the laminar flow. At the same time, the extraction reaction began in this flow state (Figure 4). Finally, the resulting aqueous and loaded organic phases were respectively excreted from the two exits.

## 2.4 Funnel extraction

In the extraction experiments, a separating funnel (60 ml) was used as extraction reactor. The oscillation frequency of separating funnel was 300 rpm in all conventional extraction experiments. In these experiments, the aqueous solution and extractant were successively poured into the separating funnel and then shocked in an oscillator (Jite, Jintan Company, China) for mixing and extraction. After that, the organic phase and aqueous phases were separated directly. The organic solutions were collected and stripped with $H_2SO_4$, and the resulting aqueous samples were sent to the Kunming Metallurgical Research Institute to detect the content of copper and nickel ions.

After analyzing copper and nickel content in the raffinate, some key parameters, such as extraction efficiency $E$ [%], distribution ratio $D$, and separation factor $\beta$, were respectively calculated by the equations

$$E = \frac{C_{\text{aq,in}} - C_{\text{aq,out}}}{C_{\text{aq,in}}} \times 100\%, \tag{1}$$

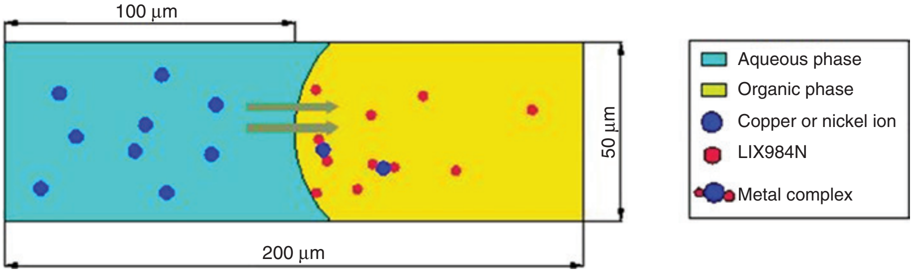

**Figure 4:** Schematic of the cross-section of the main channel.

$$D=\frac{C_{\mathrm{aq,in}}-C_{\mathrm{aq,out}}}{C_{\mathrm{aq,out}}}, \tag{2}$$

$$\beta=\frac{D_{\mathrm{Cu}}}{D_{\mathrm{Ni}}}. \tag{3}$$

For the microchannel experiments, the contact time was adjusted by a two-phase volume flow rate, which was the same in all experiments. The contact time was calculated by the formula

$$t=\frac{V}{2Q_{\mathrm{aq}}}. \tag{4}$$

# 3 Results and discussion

## 3.1 Effect of contact time on extraction efficiency

In this section, the effect of contact time on the extraction rate of copper and nickel was investigated. The extraction results are shown as Figure 5.

As shown in Figures 5A and B, the equilibrium extraction rate of copper in the microchannel was nearly 100% after contacting for 3.9 s, whereas that in the separating funnel was just nearly 97% after contacting for 50 s. The reason why the mass transfer speed was so fast in the microfluidic device can be attributed to the thickness of the aqueous phase in the microchip and the target ions could contact the extraction agent sufficiently and efficiently [24]. Hence, it just needed little time to accomplish ion diffusion from the aqueous phase to the organic–aqueous interface and a nearly complete mixture can be achieved within milliseconds. Furthermore, as shown in Figures 5C and D, the separation factor of copper and nickel in the microfluidic extraction was much greater, thereby demonstrating better separation performance of copper and nickel.

In order to obtain higher copper extraction rate and separation effect during the following microfluidic experiments, the contacting time was set as 1.95 s in the microchannel reactor, which was achieved by setting the volume flow rate of each phase at 0.02 ml min$^{-1}$ and at 60 s in the separating funnel.

## 3.2 Effect of the extractant concentration on extraction efficiency

Figure 6 shows the effect of volume fraction of extracting agent on copper and nickel extraction rate.

The extraction rate of copper, both in the separating funnel and the microchannel reactor, reached 95% when volume fraction of extracting agent increased to 15%, and as the volume fraction continued to increase, the extraction rate of copper slightly increased and then became constant in the microchannel, whereas it slightly decreased in the separation funnel.

Another phenomenon worth mentioning was that the co-extraction rate of nickel became smaller and was hardly affected by the extractant concentration in the microchannel when the volume fraction of extracting agent was not less than 15%. Hence, the volume fraction of the extracting agent was set as 15% in the following experiments.

## 3.3 Effect of the initial pH of aqueous phase on extraction efficiency

The solvent extraction of copper with LIX984N can be expressed as

$$2\mathrm{RH}_{(\mathrm{org})}+\mathrm{Cu}^{2+}_{(\mathrm{aq})}\rightleftharpoons \mathrm{R}_2\mathrm{Cu}_{(\mathrm{org})}+2\mathrm{H}^{+}_{(\mathrm{aq})}. \tag{5}$$

As LIX984N is a chelating extractant with acidic group OH, when generating stable neutral compounds with $\mathrm{Cu}^{2+}$ (Figure 2), the hydrogen ions are released. Thus, the extraction of copper with LIX984N is affected by acidity.

As shown in Figure 7A, when the pH value was lower than 2.5, the extraction rate of copper in microchannel reactor was influenced obviously by acidity and became

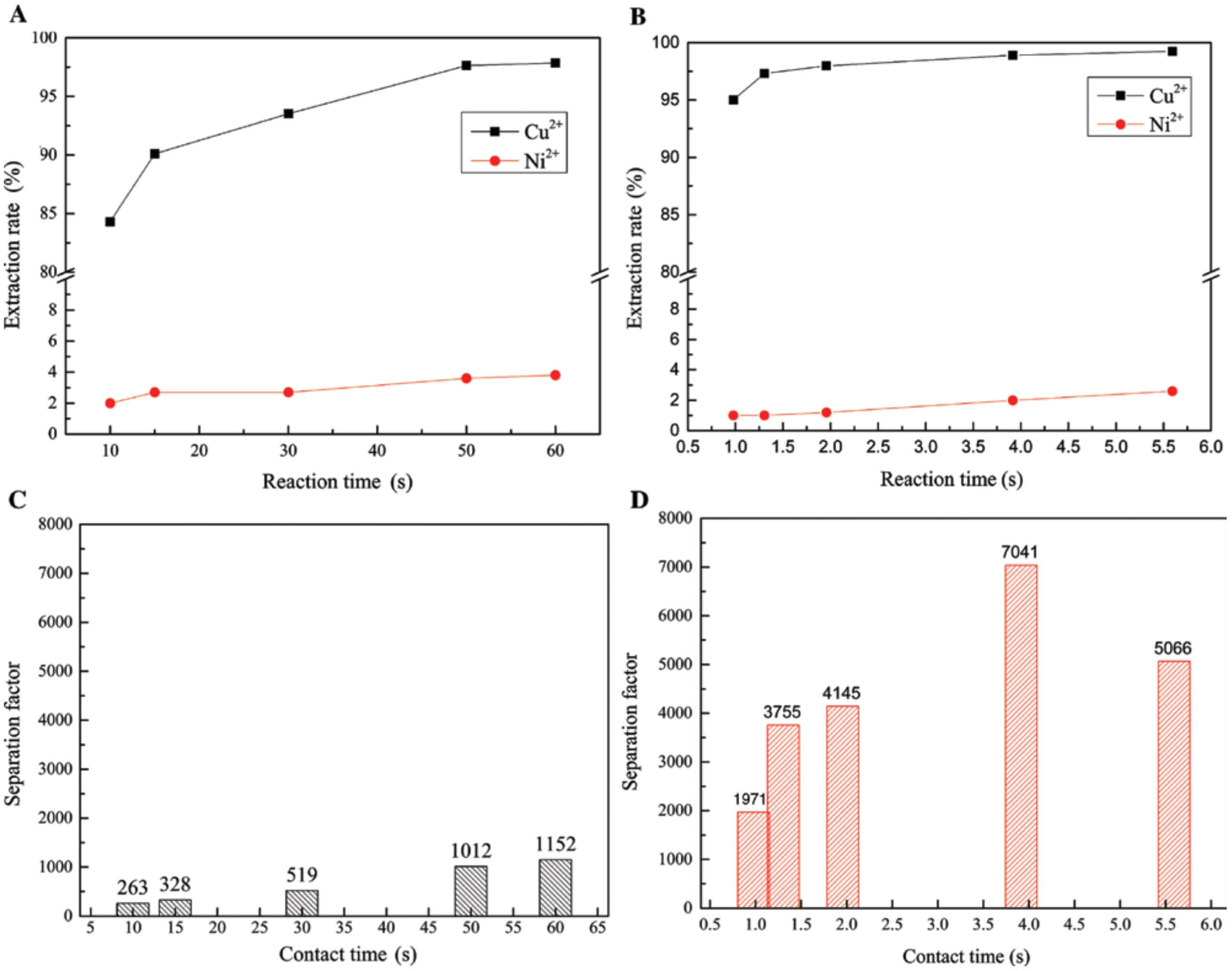

**Figure 5:** Effect of contact time on the extraction efficiency.
(A) Extraction rate and (C) separation factor in separating funnel: initial pH = 2.5, oscillation frequency 300 rpm, volume fraction of extractant 15%; (B) extraction rate and (D) separation factor in microreactor: initial pH = 2.5, volume fraction of extractant 15%.

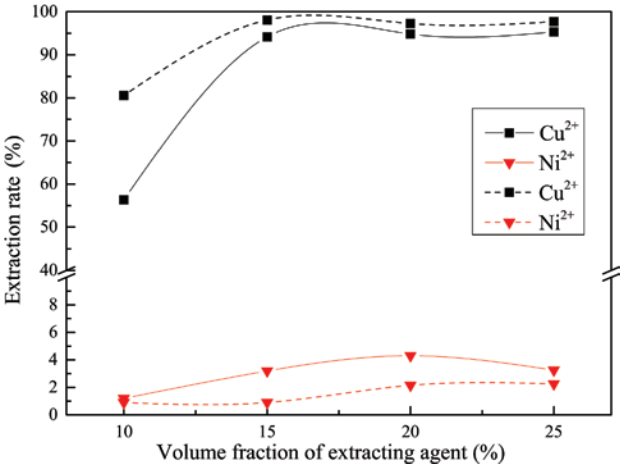

**Figure 6:** Effect of volume fraction of extracting agent on copper and nickel extraction rate. (----) Microfluidic extraction: initial pH = 2.5, reaction time 1.95 s; (—) Conventional extraction: initial pH = 2.5, oscillation frequency 300 rpm, contact time 60 s.

higher than that in separating funnel, whereas the extraction rate of copper was nearly 99% in the both reactors when the value of pH more than 2.5. This is mainly because LIX984N is a mixture of two chelating agents and the copper complex generated is a stable chelate. Although acidity also affected the extent of extraction reaction, the reaction equilibrium was not controlled by the pH value in the chelating extraction reaction.

Moreover, the selectivity of chelating extraction agent for ions is determined by the structure stability of chelate, and it is not controlled by the acidity. Hence, the effect of initial pH value on the extraction rate of nickel was relatively small (Figure 7A) in both the microchannel reactor and the separation funnel. However, it was lower in the microchannel reactor. In addition, as shown in Figure 7B, the separation factor further indicated that the microchannel reactor had an obvious advantage in the separation of copper and nickel. Hence, in order to obtain the

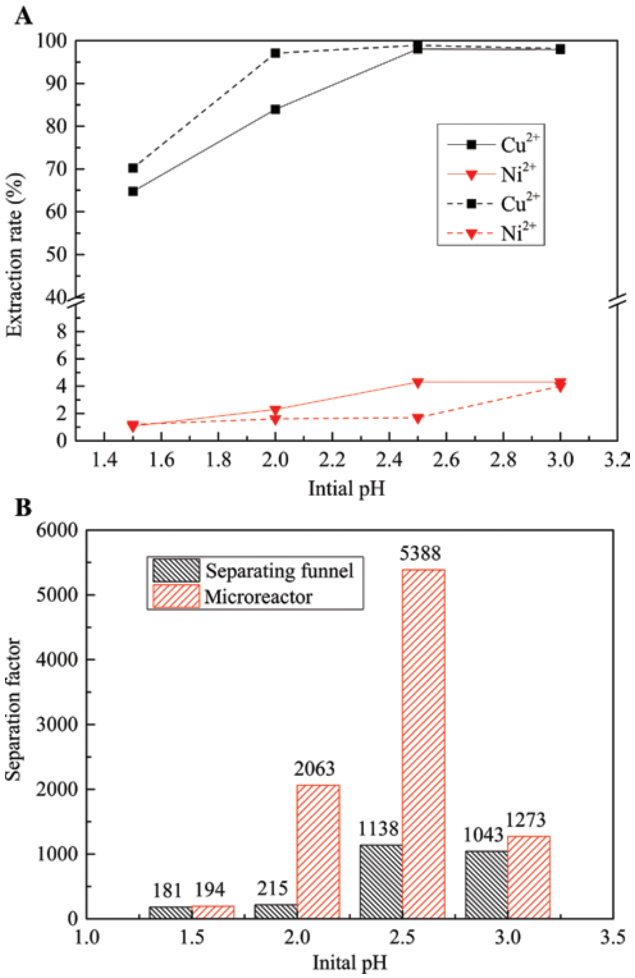

**Figure 7:** Effect of initial pH on the extraction efficiency. (A) Extraction rate, and (B) separation factor between Cu and Ni. (----) Microfluidic extraction, (—) Conventional extraction.

best copper extraction rate and copper-nickel separation effect, the optimum initial pH should be set at 2.5.

## 3.4 Effect of temperature on extraction efficiency

In the microchannel reactor, reaction temperature could be conveniently controlled by a water bath and the solvent could not be volatilized due to the closed passage. In comparison, conventional extraction systems need to decrease the temperature to avoid the loss of organic solvents. Thus, we just studied the effect of temperature on extraction efficiency in the ingenious microreactor.

In this section, a mercury thermometer was used to detect the current water temperature to monitor the error between the actual value and set value. The microchannel reactor and most of the capillary connected to the chip was subjected to a constant-temperature water bath based on the experimental apparatus mentioned above (Figure 3A). When water temperature rose to the set value, we waited for 10 min and began to collect samples. Under the condition without nickel ions, the extraction rate of copper was above 98.5% and was not affected by temperature, as shown in Figure 8. Meanwhile, the extraction rate of copper decreased by 1% when there were nickel ions. Furthermore, with the increase of temperature, the nickel extraction rate improved but the copper extraction rate was almost unchanged at below to 98.5%.

The reaction temperature should be set to room temperature if considering energy consumption and copper-nickel separation. The effect of temperature on the extraction efficiency was not obvious, which indicated that the copper ion diffusion was very adequate in the microchannel reactor. This can be attributed to the higher concentration gradient and the ratio of interface to volume. As temperature did not affect the copper extraction rate and the copper extraction rate was more than 95% at room temperature, the copper ion diffusion in the microchannel was very adequate at room temperature without other strengthening measures, such as stirring. Thus, we could initially determine that the copper extraction was controlled by the interfacial chemical reaction.

## 3.5 Effect of copper ions concentration on extraction efficiency

Figure 9 shows the effect of copper ion concentration on copper and nickel extraction rate. As can be seen,

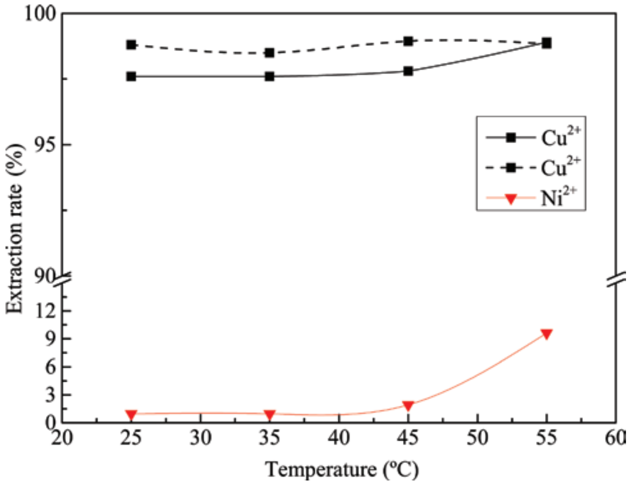

**Figure 8:** Effect of temperature on copper-nickel extraction efficiency. pH = 2.5, contact time 1.95 s, volume fraction of extractant 15%: (----) without nickel ions; (—) nickel ions $1\ \text{g l}^{-1}$.

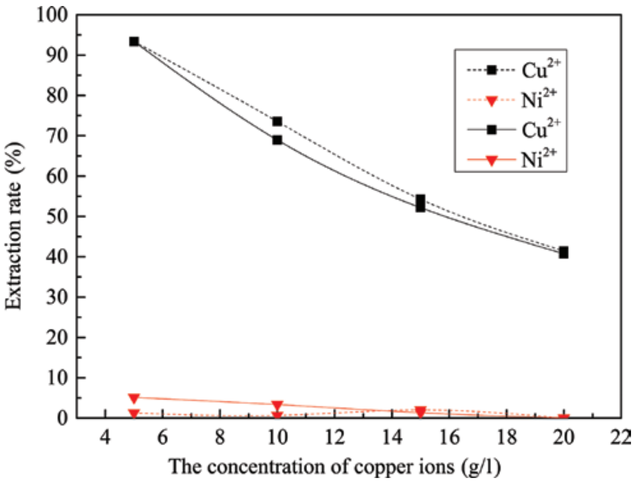

**Figure 9:** Effect of copper ions concentration on extraction efficiency. (----) Microfluidic extraction; (—) conventional extraction.

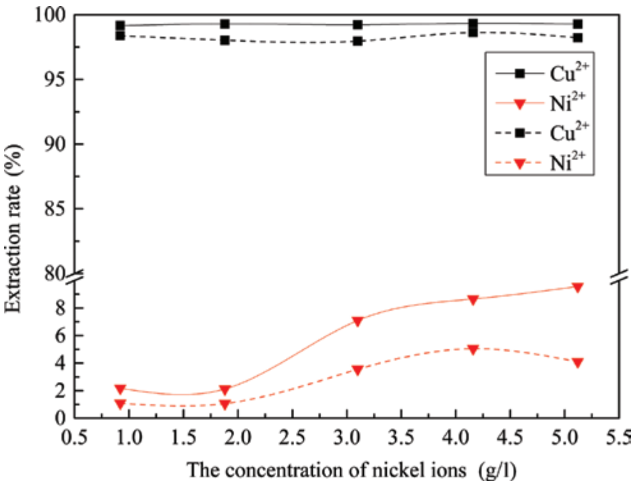

**Figure 10:** Effect of nickel ions concentration on extraction efficiency. (----) Microfluidic extraction; (—) conventional extraction.

the higher the copper ion concentration, the smaller the copper extraction rate in both the microchip and separating funnel. This can be attributed to the fact that, when the amount of extractant did not change, the amount of metal ions that can be extracted in the extraction reaction was also constant. As shown in Equation (1), with the increase of copper ion concentration in the aqueous phase, the value of $C_{\mathrm{aq,in}}$ also increased in this equation, whereas the value of $C_{\mathrm{aq,in}} - C_{\mathrm{aq,out}}$ increased slowly and finally reached a certain value. Thus, we can say that the extraction rate of copper decreased with the increase of the concentration of copper ions.

In addition, the higher extraction rate of copper and the lower extraction rate of nickel were obtained in the microchip. On the one hand, the microchip has a high surface area to volume ratio, making the reaction more thorough and fast. On the other hand, the mixing of two-phase fluid in the microchip was more uniform, and the aqueous phase was not wrapped in a large amount of oil phase, so the extraction rate of nickel was lower than that in the separating funnel.

## 3.6 Effect of nickel ion concentration on extraction efficiency

The effects of nickel ion concentration on copper extraction in the separating funnel and the microchannel were both studied. The results are shown in Figure 10.

As shown in Figure 10, the extraction rate of copper, which was very high (nearly 99%), did not change with the increase of nickel ion concentration in both reactors. In addition, it was obvious that the nickel ions were extracted easily in the separating funnel than in the microchannel, and the extraction rate increased with the increase of nickel ion concentration. This phenomenon might be due to the two different flow patterns. From the experimental results, the extraction agent Lix984N showed some nickel extraction ability. In conventional extraction, nickel ions might be extracted into the organic phase due to strong stirring and mixing. In comparison, in the microfluidic laminar flow conditions, the extraction was always carried out under equilibrium conditions; nickel ions were hardly extracted, unless the copper ions were extracted entirely.

## 3.7 Mass transfer characteristic

In the multi-phase system, mass transfer characteristic could be investigated by overall volumetric mass transfer coefficient ($K_L\alpha$). For mass transfer, the driving force is characterized by a logarithmic mean concentration difference ($\Delta C_{\ln}$) defined as Equation (6) [25]

$$\Delta C_{\ln} = \frac{(C_{\mathrm{aq,eq}} - C_{\mathrm{aq,in}}) - (C_{\mathrm{aq,eq}} - C_{\mathrm{aq,out}})}{\ln[(C_{\mathrm{aq,eq}} - C_{\mathrm{aq,in}})/(C_{\mathrm{aq,eq}} - C_{\mathrm{aq,out}})]}. \quad (6)$$

The mean mass transfer flux ($J$) can be expressed as

$$J = K_L \Delta C_{\ln}, \quad (7)$$

and the mean mass transfer flux ($J$) is equal to

$$J = \frac{Q_{aq}(C_{\mathrm{aq,out}} - C_{\mathrm{aq,in}})}{V\alpha}. \quad (8)$$

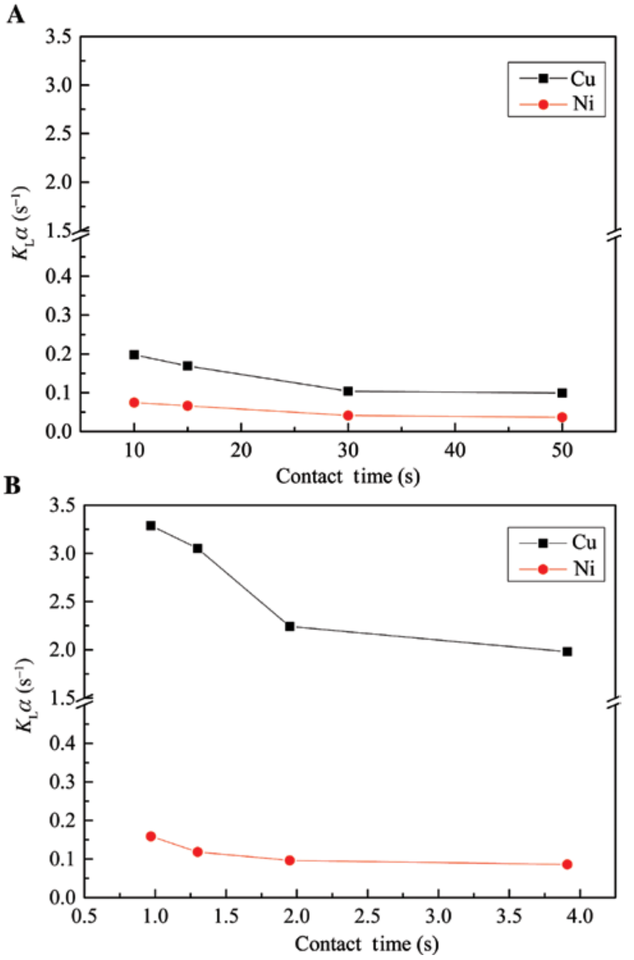

**Figure 11:** Overall volumetric mass transfer coefficient versus contact time in (A) separating funnel and (B) microreactor.

By combing the above equations, the overall volumetric mass transfer coefficient can be obtained

$$K_L\alpha = \frac{1}{t}\ln\left(\frac{C_{\text{aq,eq}} - C_{\text{aq,in}}}{C_{\text{aq,eq}} - C_{\text{aq,out}}}\right). \tag{9}$$

The overall volumetric mass transfer coefficients values of copper and nickel extraction versus residence time are shown in Figure 11. As shown in the figure, $K_L\alpha$ of Cu and Ni reduced with the increase of residence time in both the microchannel reactor and separating funnel, and gradually became stable. A much higher value of $K_L\alpha$ of Cu (3.28–1.98 s$^{-1}$) than that in the separating funnel (0.19–0.09 s$^{-1}$) was obtained. Meanwhile, $K_L\alpha$ value of Ni were relatively smaller in both reactors. The overall volumetric mass transfer coefficient of Cu and Ni illustrated indirectly that the separation of copper form nickel in the microreactor was efficient and fast.

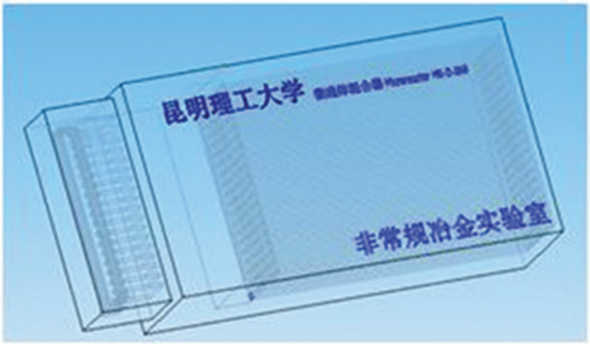

**Figure 12:** A microfluidic model with 200 microchannels.

## 4 Possibility of scaling up the microfluidic device

Nowadays, two methods can be used to increase the handling capacity of the microreactor. The first one is the “reaction chamber enlargement,” and its essence is to expand the channel size from micron to the millimeter or even up to the centimeter level. Wang et al. [26] successfully developed an amplifying device with treatment capacity of 0.6 m$^3$ h$^{-1}$, whose internal channel size was expended from micron to millimeter level (4 × 2 mm cross-section).

Another method is the “Numbering up” mode. Due to the limitation of conventional manufacture technology, it is difficult to produce thousands of microchannels in a small facility. However, 3D printing technology has recently shown the possibility of solving the problem. Therriault et al. [27] used 3D printing techniques to fabrication a complex microvascular network, whose internal diameters were in the range of 100–300 μm. Moreover, Anderson et al. [28] used 3D design and 3D printing techniques to complete the preparation of a micro-reactor containing eight parallel channels, 3 mm in width and 1.5 mm in depth. Vogel et al. [29] combined computer-aided tools (CAD, CMFD, CMPD, and FEA) with 3D printing, and produced a device with a complex structure: a state-of-the-art commercial slurry phase reactor.

We also designed a microreactor with 200 microchannels and optimized it with fluidics simulation, as shown in Figure 12. Therefore, as a consequence of the rapid progress of 3D printing technology, the problem of scaling up can be solved in the near future.

## 5 Conclusion

In the microchannel reactor, the flow state of the fluid was laminar, whereas the fluidic state was turbulent flow in the separating funnel. The results showed that microfluidic extraction had better advantages in the separation of copper and nickel. The ratio of Cu mean mass transfer rate in the microchannel reactor to that of Ni could be as high as 20, whereas that in the separating funnel was just 2. Moreover, the ratio of the separation factors of Cu and Ni in the microchannel reactor to that in separating funnel was up to 5.

Typically, at initial pH = 2.5, contact time of 1.95 s, volume fraction of extractant Lix984N 15% and within 1.9 g $l^{-1}$ nickel ion concentration, the copper extraction rate was higher than 95%, but nickel was hardly extracted. In comparison, almost 50 s was needed to reach a Cu extraction of 95% in the separation funnel, with more than 5% Ni co-extraction rate. Overall, with the rapid development of 3D printing technology, the scaling up of microfluidic solvent extraction has become more promising.

**Acknowledgments:** Financial aid from the following programs is gratefully acknowledged: the National Natural Science Foundation of China (Grant Nos. U1302271, 51504116, and 51404116), and the Kunming University of Science and Technology Personnel Training Fund (KKSY201452088).

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