

## Reduction and deposition of arsenic in copper electrolyte

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**Abstract:** The influences of temperature, H<sub>2</sub>SO<sub>4</sub> concentration, CuSO<sub>4</sub> concentration, reaction time and SO<sub>2</sub> flow rate on the reduction of arsenic(V) with SO<sub>2</sub> were studied and the deposition behavior of arsenic (III) under the effect of concentration and co-crystallization was investigated in copper electrolyte. The results indicate that reduction rate of arsenic (V) decreases with increasing temperature and H<sub>2</sub>SO<sub>4</sub> concentration, but increases with increasing SO<sub>2</sub> flow rate and reaction time, and it can reach 92% under appropriate conditions that reaction temperature is 65 °C, H<sub>2</sub>SO<sub>4</sub> concentration is 203 g/L, CuSO<sub>4</sub> concentration is 80 g/L, reaction time is 2 h and SO<sub>2</sub> gas flow rate is 200 mL/min. To remove arsenic in the copper electrolyte, arsenic (V) is reduced to trivalence under the appropriate conditions, the copper electrolyte is concentrated till H<sub>2</sub>SO<sub>4</sub> concentration reaches 645 g/L, and then the removal rates of As, Cu, Sb and Bi reach 83.9%, 87.1%, 21.0% and 84.7%. The XRD analysis shows that crystallized product obtained contains As<sub>2</sub>O<sub>3</sub> and CuSO<sub>4</sub>·5H<sub>2</sub>O.

**Key words:** copper electrolyte; arsenic (V); reduction; sulfur dioxide; concentration; arsenic trioxide

### 1 Introduction

Arsenic acid can be reduced to arsenious acid. The reaction is widely applied to preparing arsenious acid, arsenic trioxide and arsenite from waste acid in chemical industry. For example, LINDROOS and VIRTANEN [1] have found the method for the removal of arsenic by sulfur dioxide reduction. ZHENG et al [2] have used the technology of sulfur dioxide reduction to prepare arsenic trioxide from arsenic sulfide slag. REDMON et al [3] have separated arsenic acid from an aqueous mixture comprising sulfuric and arsenic acids by sulfur dioxide reduction. This technology has resolved the problem of generating massive waste for storage with the conventional methods [4, 5]. Moreover, it can realize the recycle and non-hazardous treatment of arsenic. Nevertheless, very little published data exist concerning the factors affecting the reduction of arsenic (V) with sulfur dioxide and the extent of the reaction.

This investigation was conducted for two main aims: 1) to obtain the appropriate conditions of reducing arsenic (V) with sulfur dioxide in copper electrolyte by means of probing the roles of temperature, H<sub>2</sub>SO<sub>4</sub> concentration, CuSO<sub>4</sub> concentration, reaction time and SO<sub>2</sub> flow rates on the reduction process, and 2) to utilize

it to remove arsenic in copper electrolyte [6]. Firstly, arsenic (V) was reduced to trivalence in copper electrolyte by the aid of sulfur dioxide. Then the copper electrolyte was concentrated, and arsenic (III) in it would transform into arsenic trioxide and deposit efficiently under the effect of copper sulphate co-crystallization. Antimony and bismuth in electrolyte could be removed due to the function of reduction and inclusion. The technology does not produce toxic arsine gas and has special merit that it is easy to separate arsenic from crystallized product in comparison with electrowinning method [7]. It can be expected to be extensively applied in copper electrorefining.

### 2 Experimental

#### 2.1 Reduction of arsenic (V) in copper electrolyte with sulfur dioxide

300 mL copper electrolyte whose composition is shown in Table 1 was introduced into a 0.5 L flask. The flask contained three openings for admission of a stirring rod, a gas dispersion tube, a gas outlet tube and a thermocouple. The temperature was controlled accurately, the stirring rate was kept at 300 r/min and SO<sub>2</sub> gas was bubbled into the flask at a predetermined

rate. When the reaction time was up, SO<sub>2</sub> gas bubbling was stopped and copper electrolyte was heated to boil to end the experiment. The arsenic (III) concentration in the copper electrolyte was analyzed by ceric sulfate-potassium bromate titration.

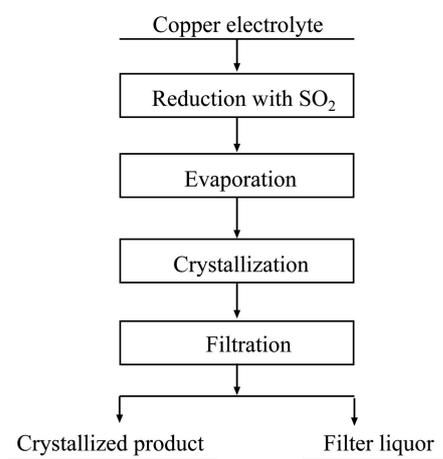
**Table 1** Compositions of copper electrolyte

$\rho(\text{Cu})/$ (g·L <sup>-1</sup> )	$\rho(\text{As}_T)/$ (g·L <sup>-1</sup> )	$\rho(\text{As(V)})/$ (g·L <sup>-1</sup> )	$\rho(\text{Sb})/$ (g·L <sup>-1</sup> )	$\rho(\text{Bi})/$ (g·L <sup>-1</sup> )	$\rho(\text{H}_2\text{SO}_4)/$ (g·L <sup>-1</sup> )
32	6.0	5.7	0.75	0.42	203

### 2.2 Deposition of arsenic (III) in copper electrolyte

500 mL copper electrolyte that had been reduced was introduced into a 1 L beaker with accurate scale and evaporated. The evaporated volume of copper electrolyte was controlled precisely. Then the copper electrolyte was cooled to 10 °C to crystallize, and the crystallization process was kept at a stirring rate of 15 r/min for 7 h. The treated solution was filtered to separate crystallized product. The concentrations of arsenic, antimony, bismuth and copper in copper electrolyte were determined by ICP emission spectroscope (IRIS Intrepid II XSP, Thermo Elemental Corporation, America). Crystallized product was examined by X-ray diffraction apparatus (D/max-rA, Rigaku Corporation, Japan). Compositions of crystallized product were obtained by X-ray fluorescence analysis (Philip24).

All experiments were conducted according to the flow sheet of reduction and deposition of arsenic in copper electrolyte which is shown in Fig. 1.



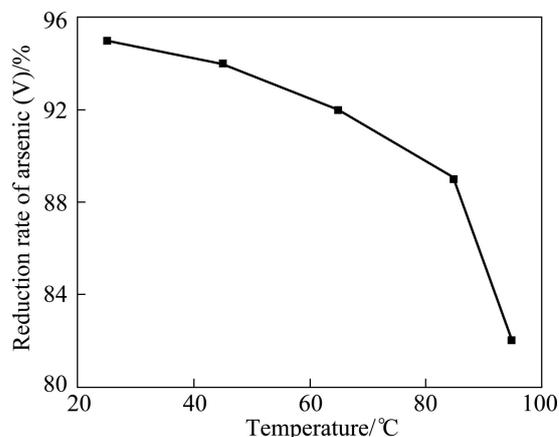
**Fig. 1** Flow sheet of reduction and deposition of arsenic in copper electrolyte

## 3 Results and discussion

### 3.1 Reduction of arsenic (V) in copper electrolyte with sulfur dioxide

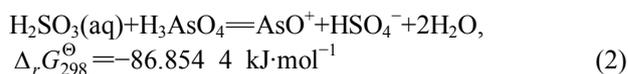
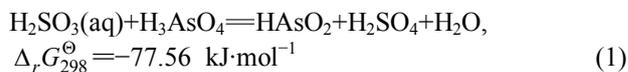
#### 3.1.1 Influence of reaction temperature on reduction rate of arsenic (V)

The influence of reaction temperature on reduction rate of arsenic (V) is shown in Fig. 2 under the conditions that H<sub>2</sub>SO<sub>4</sub> concentration and CuSO<sub>4</sub> concentration in copper electrolyte are 203 g/L and 80 g/L, respectively, reaction time is 4 h, SO<sub>2</sub> gas flow rate is 200 mL/min. It can be seen from Fig. 2 that reduction rate of arsenic(V) decreases with increasing temperature. When temperature rises from 25 °C to 95 °C, reduction rate of arsenic (V) decreases from 95% to 82%.



**Fig. 2** Influence of reaction temperature on reduction rate of arsenic (V)

Copper electrolyte is the H<sub>2</sub>SO<sub>4</sub>-CuSO<sub>4</sub>-H<sub>2</sub>O system comprising sulphuric acid, copper sulfate, water and other impurities. When H<sub>2</sub>SO<sub>4</sub> concentration is 203 g/L and CuSO<sub>4</sub> concentration is 80 g/L in the system, its pH value is about -0.32 which can be calculated according to H<sup>+</sup> concentration in the electrolyte. And the equation of reduction of arsenic (V) with sulfur dioxide can be proposed as follows [8–10]:



As the standard Gibbs free energies for the reactions are both large and negative, their equilibrium constants are large. Hence the chemical reactions have tremendous thermodynamic impetus and can go completion.

But the study of kinetics of the reduction process indicates that two sequential steps can be identified to control the reaction rate. One is the absorption of SO<sub>2</sub> gas into the liquid phase, and the other is chemical reaction between arsenic (V) and sulfur dioxide. As the experiment is conducted in an open system, the relationship between SO<sub>2</sub> solubility and temperature, SO<sub>2</sub> (g) partial pressure can be expressed as



where  $H$  is the Henry coefficient and can be fitted by the semi-empirical equation [11]:

$$1/H = \exp(3\,114.9/T - 10.324)$$

where  $p(\text{SO}_2)$  is the partial pressure of  $\text{SO}_2$  in the gas phase.

The influence of temperature on Henry coefficient is shown in Table 2. It is obvious that the reciprocal of Henry coefficient value decreases quickly as temperature rises from 298 to 373 K. The solubility of sulfur dioxide diminishes fast with the increase of temperature according to Eq. (3). The absorption of  $\text{SO}_2$  into the liquid phase turns to the controlling step of the reduction reaction. The reduction rate of arsenic (V) decreases with the increase of temperature.

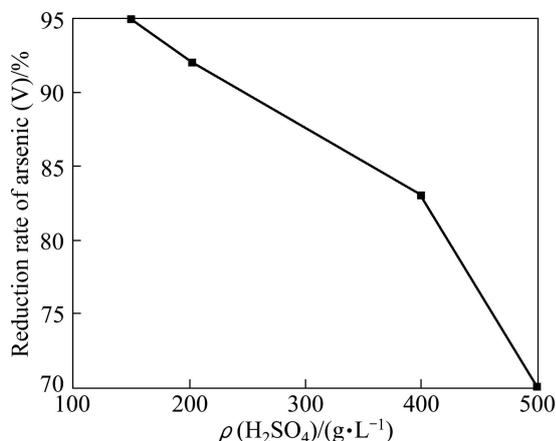
**Table 2** Influence of temperature on Henry coefficient

Temperature/K	298	318	338	358	373
$1/H$	1.137	0.589	0.330	0.197	0.139

To obtain high reduction rate of arsenic (V), the reduction reaction should be conducted under low temperature.

### 3.1.2 Influence of $\text{H}_2\text{SO}_4$ concentration on reduction rate of arsenic (V)

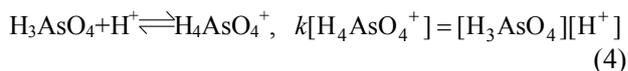
As shown in Fig. 3, the reduction rate of arsenic (V) drops dramatically with the increase of  $\text{H}_2\text{SO}_4$  concentration as reaction temperature is 65 °C and the other experimental conditions are constant. Reduction rate of arsenic (V) decreases to 70% when  $\text{H}_2\text{SO}_4$  concentration increases to 500 g/L.



**Fig. 3** Influence of  $\text{H}_2\text{SO}_4$  concentration on reduction rate of arsenic (V)

The increase of  $\text{H}_2\text{SO}_4$  concentration in the  $\text{H}_2\text{SO}_4\text{-CuSO}_4\text{-H}_2\text{O}$  system can restrain solubility of sulfur dioxide obviously [12]. However, it is not the only reason for the decrease of reduction rate of arsenic (V). Some investigations reveal that  $\text{H}^+$  can protonate arsenic acid to form  $\text{H}_4\text{AsO}_4^+$  which is less reactive than  $\text{H}_3\text{AsO}_4$ .

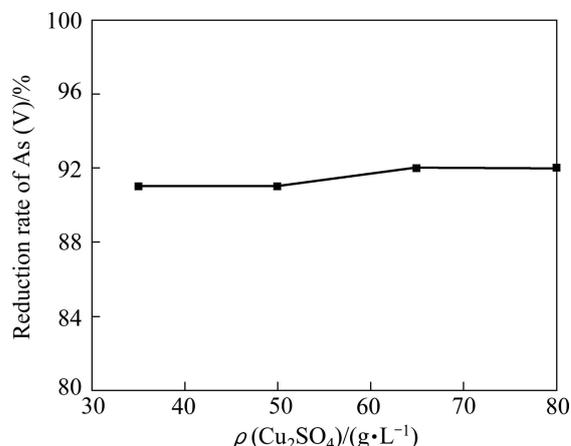
The formation of  $\text{H}_4\text{AsO}_4^+$  makes arsenic acid less active and so that it is difficult to reduce arsenic (V) with  $\text{SO}_2$  [13].



where  $k$  is the thermodynamic equilibrium constant for the reaction.

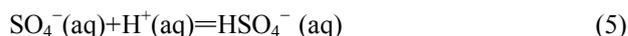
### 3.1.3 Influence of $\text{CuSO}_4$ concentration on reduction rate of arsenic (V)

The influence of  $\text{CuSO}_4$  concentration on reduction rate of arsenic (V) is shown in Fig. 4 under the conditions that  $\text{H}_2\text{SO}_4$  concentration is 203 g/L and the other factors remain constant. It can conclude that  $\text{Cu}$  concentration has little effect on reduction rate of arsenic (V). Reduction rate of arsenic (V) is near or equal to 92% when  $\text{CuSO}_4$  concentration varies in the range of 35–80 g/L.



**Fig. 4** Influence of  $\text{CuSO}_4$  concentration on reduction rate of arsenic (V)

As the concentration of  $\text{CuSO}_4$  in electrolyte rises,  $\text{SO}_4^{2-}$  concentration increases.  $\text{SO}_4^{2-}$  can react with  $\text{H}^+$  to form  $\text{HSO}_4^-$ , and the reaction can be proposed as Eq. (5). So, the increases of  $\text{CuSO}_4$  concentration will bring down the acidity of electrolyte and favor the reducing process.



In fact, there is little change of reduction rate of arsenic (V) when  $\text{Cu}$  concentration in copper electrolyte varies from 35 to 80 g/L. It is on account to not only the fact that the range of variation is relatively little in the strong acid solution, but also the bisulfate acid formed due to the reaction restricting the reduction reaction of arsenic (V), which can be found from Eqs. (1) and (2). The influence of  $\text{CuSO}_4$  concentration on reduction rate of arsenic (V) is complex, and  $\text{CuSO}_4$  concentration does not affect the reduction rate of arsenic (V) significantly.

3.1.4 Influence of reaction time on reduction rate of arsenic (V)

Figure 5 shows the influence of reaction time on reduction rate of arsenic (V) when Cu concentration is 32 g/L and other factors remain constant.

It can be seen that reduction rate of arsenic (V) reaches 92% when the reaction proceeds for 2 h. The reduction rate of arsenic (V) almost remains constant when the reaction time exceeds 2 h.

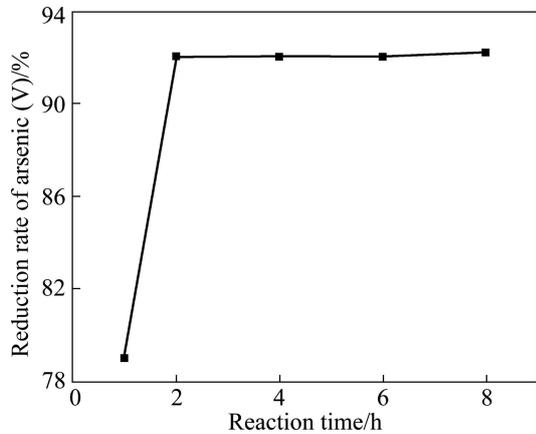


Fig. 5 Influence of reaction time on reduction rate of arsenic (V)

3.1.5 Influence of SO<sub>2</sub> flow rate on reduction rate of arsenic (V)

The influence of SO<sub>2</sub> flow rate on reduction rate of arsenic (V) is shown in Fig. 6 when reaction time is 2 h. It can be found from Fig. 6 that reduction rate of arsenic (V) increases quickly with the increasing SO<sub>2</sub> flow rate until it exceeds 200 mL/min.

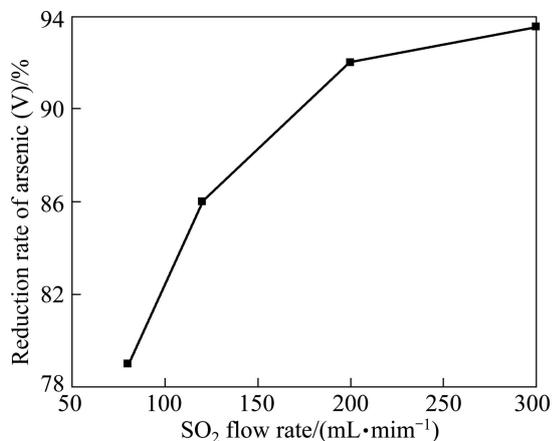


Fig. 6 Influence of SO<sub>2</sub> flow rate on reduction rate of arsenic (V)

The reduction process is controlled by the sulfur dioxide dissolution under the circumstance that the SO<sub>2</sub> flow rate is large enough. In fact, the increase of SO<sub>2</sub> flow rate can increase the concentration of sulfurous acid in copper electrolyte according to Eq. (3), and will

increase the reducing reaction rate and even the extent of reaction.

In order to get a high reduction rate of arsenic (V) in the finite time, SO<sub>2</sub> flow rate should be maintained at least 200 mL/min according to the result and then the reduction rate of arsenic (V) reaches 92%.

3.2 Deposition of arsenic (III) in copper electrolyte

To purify the copper electrolyte, the deposition of arsenic reduced in copper electrolyte was carried out according to the flow sheet as shown in Fig. 1. After arsenic(V) in copper electrolyte was reduced to trivalence under the appropriate conditions, the copper electrolyte was condensed till the H<sub>2</sub>SO<sub>4</sub> concentration reached 645 g/L, then the copper electrolyte was cooled to crystallize and filtered. The composition of copper electrolyte has been reduced under the appropriate conditions before and after concentration and crystallization can be seen from Table 3, from which the removal rates of arsenic, copper, antimony and bismuth can be calculated to be 83.9%, 87.1%, 21.0% and 84.7%. The XRD analysis result of the crystallized product obtained can be seen from Fig. 7, and it shows that the crystallized product includes arsenic trioxide and copper sulphate. The compositions of crystallized product is shown in Table 4 which are obtained by X-ray fluorescence analysis, and it can be seen that the crystallized product includes 4.4%As, 32.6%Cu, 0.15%Sb, 0.22%Bi.

Table 3 Chemical compositions of electrolyte before and after concentration and crystallization

Electrolyte	$\rho(\text{Cu})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{As}_T)/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{As(III)})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{Sb})/(\text{g}\cdot\text{L}^{-1})$
Before	32.0	6.0	5.57	0.75
After	12.8	3.0	2.40	1.84
Electrolyte	$\rho(\text{Bi})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{H}_2\text{SO}_4)/(\text{g}\cdot\text{L}^{-1})$	Volume/mL	
Before	0.42	210	500	
After	0.20	645	161	

Table 4 Compositions of crystallized product (mass fraction, %)

Cu	As	Sb	Bi	Ni	S	O
32.6	4.4	0.15	0.22	1.5	18.3	41

H<sub>2</sub>SO<sub>4</sub> concentration in copper electrolyte increases in the process of concentration. When H<sub>2</sub>SO<sub>4</sub> concentration increases from 0 to 700 g/L, the solubility of trivalent arsenic decreases to a rock bottom gradually [14], and trivalent arsenic will deposit in the form of arsenic trioxide. So, arsenic in the electrolyte is removed.

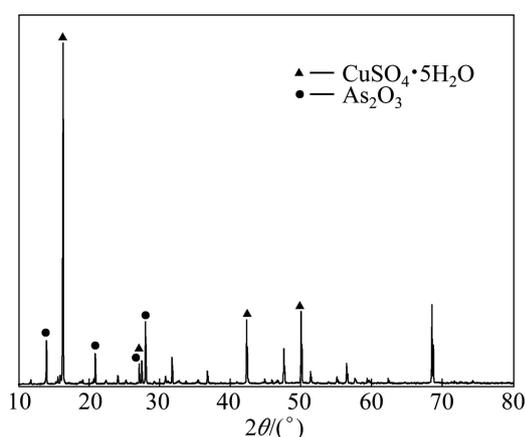


Fig. 7 XRD pattern of crystallized product obtained

The crystallization course of arsenic trioxide includes two steps, the formation and growth of crystal nucleus, and the formation of crystal nucleus is the control step. WANG and HENG [15] pointed out that the crystallization process of arsenic trioxide is considerably slow. But the formation of the copper sulfate crystal in copper electrolyte can promote heterogeneous nucleation of arsenic trioxide, which brings down its surface energy of crystallization.

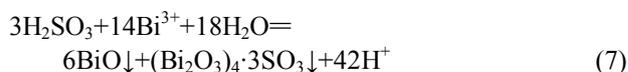
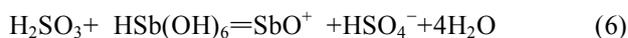
In order to investigate the influence of copper sulfate co-crystallization on the deposition behavior of arsenic (III) in copper electrolyte, one contrast experiment was carried out according to the flow sheet as shown in Fig. 1. The contrast experiment was conducted with the same experiment method, except that  $\text{CuSO}_4$  concentration in the copper electrolyte was 4 g/L and copper sulfate crystal would not form in the crystallization course. The chemical compositions of electrolyte of the contrast experiment before and after concentration and crystallization are shown in Table 5. It can be calculated that the removal rate of arsenic in the contrast experiment is 75.3%, which is lower in comparison with the experiment which has the copper sulfate crystallization.

Antimony (V) was reduced to antimony (III) in the presence of the reducing agent sulfur dioxide [16], and diantimony trioxide formed due to the co-crystallization

Table 5 Compositions of electrolyte of contrast experiment before and after concentration and crystallization

Electrolyte	$\rho(\text{Cu})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{As}_T)/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{As(III)})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{Sb})/(\text{g}\cdot\text{L}^{-1})$
Before	4.0	6.0	5.57	0.75
After	12.4	4.6	4.40	1.79
Electrolyte	$\rho(\text{Bi})/(\text{g}\cdot\text{L}^{-1})$	$\rho(\text{H}_2\text{SO}_4)/(\text{g}\cdot\text{L}^{-1})$	Volume/mL	
Before	0.42	210	500	
After	0.24	642	161	

function of arsenic trioxide. So, antimony is removed from the electrolyte. Bismuth is the impurity which is easy to be removed from copper electrolyte and can be reduced to  $\text{BiO}$  and  $(\text{Bi}_2\text{O}_3)_4\cdot 3\text{SO}_3$ , which are both precipitation [17–18].



## 4 Conclusions

1) Reduction rate of arsenic (V) decreases with increasing reaction temperature and  $\text{H}_2\text{SO}_4$  concentration, but increases with increasing reaction time and  $\text{SO}_2$  flow rate. When As (V) concentration is 5.7 g/L in the copper electrolyte, reduction rate of arsenic (V) can reach 92% under the appropriate conditions that reaction temperature is 65 °C,  $\text{H}_2\text{SO}_4$  concentration is 203 g/L, Cu concentration is 80 g/L, reaction time is 2 h and  $\text{SO}_2$  gas flow rate is 200 mL/min.

2) The experiment results of reduction and deposition of arsenic in copper electrolyte show that when the concentrations of As, Cu, Sb and Bi in the electrolyte are 6, 32, 0.75 and 0.42 g/L, the removal rates of As, Cu, Sb and Bi can reach 83.9%, 87.1%, 21.0% and 84.7% after the copper electrolyte was reduced and  $\text{H}_2\text{SO}_4$  concentration reached 645 g/L.

3) XRD analysis shows that the crystallized product obtained contains  $\text{As}_2\text{O}_3$  and  $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$ . The formation of copper sulfate crystal has promoted the crystallization course of arsenic trioxide.

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## 铜电解液中砷的还原及脱除

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**摘要:** 探讨了反应温度、H<sub>2</sub>SO<sub>4</sub>浓度、CuSO<sub>4</sub>浓度、反应时间、SO<sub>2</sub>气流量等因素对SO<sub>2</sub>还原铜电解液中As(V)的影响, 并对浓缩共晶作用下铜电解液中As(III)的脱除行为进行了研究。研究表明: As(V)还原率随着反应温度和H<sub>2</sub>SO<sub>4</sub>浓度的升高而降低, 随着SO<sub>2</sub>气流量的增大及反应时间的延长而升高。当反应温度为65℃, H<sub>2</sub>SO<sub>4</sub>浓度为203 g/L, CuSO<sub>4</sub>浓度为80 g/L, SO<sub>2</sub>流量为200 mL/min, 反应时间为2 h时, 铜电解液中As(V)还原率为92%; 铜电解液中的As(V)还原后, 将铜电解液浓缩至H<sub>2</sub>SO<sub>4</sub>浓度为645 g/L时, As、Cu、Sb、Bi脱除率分别达到83.9%, 87.1%, 21%, 84.7%。XRD分析结果表明: 结晶产物中含有As<sub>2</sub>O<sub>3</sub>和CuSO<sub>4</sub>·5H<sub>2</sub>O等物相。

**关键词:** 铜电解液; 五价砷; 还原; 二氧化硫; 浓缩; 三氧化二砷

(Edited by LI Xiang-qun)