

Three-step vacuum separation for treating arsenic sulphide residue

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ABSTRACT

Up to now, most of arsenic sulphide residue can't be properly disposed. The traditional processes for treating arsenic sulphide residue have some widespread drawbacks such as the complex process, the high cost in operation, and the less than satisfactory removal effect and environmental protection. In view of this, a process of three-step vacuum separation was proposed for treating arsenic sulphide residue in this work. During vacuum separation, elemental sulphide and arsenic trioxide could be recovered effectively in primary distillation at temperature of 180 °C, distillation time of 2.0 h, corresponding to the residual gas pressure of 15 Pa. In secondary distillation, arsenic sulfide was obtained under the condition of 450 °C for 30 min, with a residual gas pressure of 15 Pa. When the temperature increased to 1000 °C for 2 h, lead sulfide was evaporated out in third distillation. Through the three-step vacuum separation, calcium fluoride was left behind in the third distilland, in which the content of arsenic was 5.65×10^{-4} wt.%. Correspondingly, the removal rate of arsenic was almost 100%. The vacuum process is expected to be effectively employed for recycling valuable components from arsenic sulphide residue, and meanwhile eliminating its pollution.

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1. Introduction

As a common element existing in ores, arsenic is widely distributed in multiple procedures of mineral processing processes, which result in the problem of residual arsenic in the intermediate and final products of the processes, and the trouble in recovery of arsenic. In the case of filling these compounds containing arsenic, a significant contamination problem is likely to appear, due to the acute toxicity of these substances. The landfills will release toxins into the groundwater. It is worth pointing out that the treating of the hazardous materials contained arsenic has serious occupational and environmental implications, especially when the treating process would not like to take the necessary precautions to protect the environment and employees' health [1]. As is known, the arsenic pollution has become a global problem, which is reported frequently in many countries such as America, Japan, India, Mexico and China [2]. In the pollution of environment report by WHO in 1968, arsenic was ranked first. Therefore, much more special attention has been drawn to arsenic problem.

There are billions tons of waste acid liquid containing arsenic produced by the factories in where arsenic-bearing minerals act as

the raw materials all over the world annually. The existing methods for treating waste acid liquid containing arsenic fall into several categories: ion exchange, adsorption (activated alumina and activated carbon); ultrafiltration, reverse osmosis, and precipitation or adsorption by metals (predominately ferric chloride) followed by coagulation [1,3–5]. At present, one of the most widely used methods is vulcanizing agent leaching, in which arsenic from waste acid liquid reacts with the vulcanizing agent, such as sodium sulfide. This method is simple, low in cost and high in separation rate of arsenic (about 99.97%) [6]. However, significant quantities of arsenic sulfide byproduct, are produced through this process, which brings about a series of challenges for the disposal of them. Arsenic sulphide residue, which is a dreadful catastrophe to human health, may result in serious environmental pollution without reasonable disposal. On the other hand, as is well-known, arsenic sulfide has high application value in field of medicine [7–11] and the metallurgical industry [12]. For instance, arsenic sulfide has antitumor effects by the way of inhibiting the growth of K562-cells and SMMC-7721-cells. In addition to this, it is also a necessary raw material in the production of herbicides and wood preservative. Therefore, it is of great interest to turn the hazardous arsenic sulphide residue into good one. What's more, the recovery of valuable components from arsenic sulphide residue is one of way to enlarge utilization of arsenic and sulphide resources.

So far, the publicly reported researches about treating arsenic sulfide residue mainly focus on curing, landfill, or recycling in the

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form of arsenic trioxide, arsenate, arsenic trichloride and so on [1,13]. One of the most attractive options is a Japanese technology, which is detailed as follows: arsenic sulfide is repulped and heated to 70 °C in CuSO₄ solution, under which arsenic sulfide reacts with the CuSO₄ to form CuS. Meanwhile, the arsenic is dissolved as arsenious acid. Subsequently, As₂O₃ is precipitated from arsenious acid solution by cooling at 25 °C [14]. Obviously, the separation of solid–liquid is necessary for each steps, and the emission of arsenic trioxide in the process of crystallization should not be ignored either. As things now stand, the existing methods, including the Japanese technology, are less than satisfactory due to the drawbacks such as complex and time-consuming process flow, low recovery efficiency, high consumption in chemical reagents, high cost in operation. Above all, these processes are inevitably produce toxic gas and large quantities of waste acid liquid, which are potential to form secondary environmental pollution.

Enlightened by above thought, a method of vacuum separation, which shows many advantages such as simple technological flow sheet, low consumption of raw material and energy, no secondary off-gas or wastewater and so on [15–17], is proposed for treating arsenic sulfide residue in this work, with the purpose of recovering valuable components without negative impact on the environment. During vacuum separation, a method of three-step distillation was employed. Through three-step distillation, elemental sulphide/arsenic trioxide, arsenic sulfide, lead sulfide were evaporated into distillate in primary distillation, secondary distillation and third distillation, respectively. Yet the calcium fluoride is left behind as the third distilland.

2. Experimental section

2.1. Experimental materials

The arsenic sulphide residue used in the experiments was acquired from lead plant in which arsenic was recycled from waste water through sodium sulfide leaching. A small amount of arsenic trioxide was formed, due to oxidation of the waste residue after pile-up for a period of time. A homogenized sample was characterized by titrimetric analysis. Correspondingly, the contents of As, S, Pb and Ca are shown in Table 1.

2.2. Apparatus

The experiments were carried out in the self-made distillation apparatus, which was mainly comprised of quartz tube, quartz crucible, water condenser, resistance furnace, intelligent temperature controller and vacuum pump, as shown in Fig. 1. The quartz tube loaded with a quartz crucible, which is used for holding the experimental sample, was placed in the self-design resistance furnace. The water condenser was inserted into middle of quartz tube and exactly right above the quartz crucible. The furnace temperature was controlled by the temperature controller with a thermocouple connected. The outlet of the quartz tube was connected with a vacuum pump to maintain constant pressure of the whole distillation system.

2.3. Processes

The processes of the experiments were shown in Fig. 2. The quartz crucible loaded with about 4 g of arsenic sulphide residue

Table 1
Chemical composition of arsenic sulphide residue.

Element	As	S	Pb	Ca
Content (wt.%)	34.69	36.82	1.15	9.29

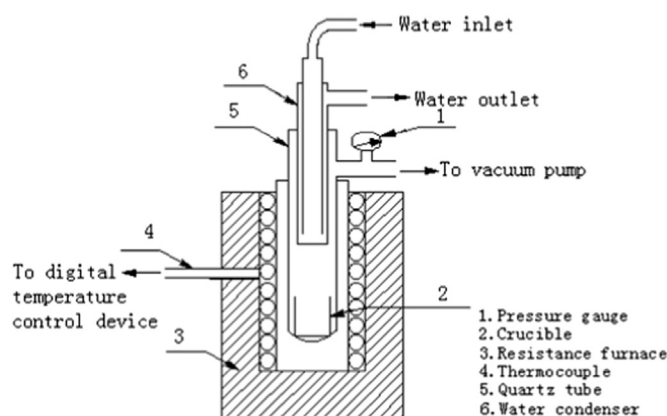


Fig. 1. Distillation apparatus.

was placed at the bottom of the quartz tube and then heated to preset temperature for a given period of time after the whole system was evacuated to a certain pressure.

During the experiment, the volatile components were evaporated out of the arsenic sulphide residue into gas phase, and then condensed in the surface of the water condenser as distillate. Meanwhile, the other components were left in the crucible as distilland.

After the experiment, the distilland left in the quartz crucible was taken out of the furnace, weighed. The distillate attached to the condenser was also cleaned out, collected and sampled. Subsequently, the residual distilland was evaporated again at a higher temperature.

As can be seen from Fig. 2, three different kinds of distillate could be obtained after three-step distillation, which elemental sulphide/arsenic trioxide, arsenic sulfide and lead sulfide, respectively. Correspondingly, the calcium fluoride was left in the crucible as third distilland.

The removal percent (*R*) of arsenic were calculated by the following formula:

$$R = \frac{m_1 \times w_1 - m_4 \times w_4}{m_1 \times w_1} \times 100\% \quad (1)$$

Where *m*₁ and *m*₄ are the mass of the arsenic sulphide residue and the third distilland (g), respectively; *w*₁ and *w*₄ are the content of arsenic (wt.%) of the arsenic sulphide residue and the third distilland respectively.

3. Principle analysis

It should be noted that, the difference in saturated vapor pressure of different components in arsenic sulphide residue at the same temperature is the basic principle for separating them from each other under vacuum. At the same temperature, the greater the saturation vapor pressure of substance, the easier it evaporates.

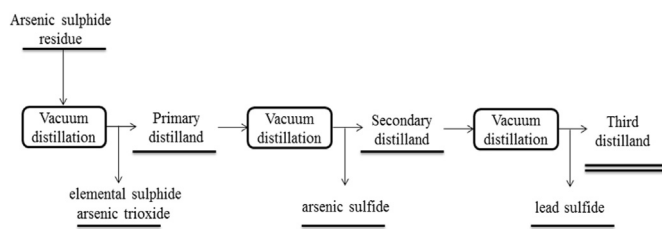


Fig. 2. Flowsheet for treating arsenic sulphide residue.

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