Separation and Purification Technology 138 (2014) 41-46

Contents lists available at ScienceDirect



Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

Separation of elemental sulfur from zinc concentrate direct leaching residue by vacuum distillation



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ARTICLE INFO

Article history: Received 29 January 2014 Received in revised form 24 September 2014 Accepted 27 September 2014 Available online 18 October 2014

Keywords: Elemental sulfur Direct leaching residue Vacuum distillation

ABSTRACT

Properly disposal and reutilization of direct leaching residue (DLR) from metal production industries can reduce environmental pollution as well as better conserve resources. In this study, recovery of elemental sulfur from zinc concentrate DLR using vacuum distillation was thoroughly investigated. The results show that elemental sulfur recovery using vacuum distillation was over 98% with a high purity under optimized conditions. The low temperature requirement (200–300 °C) of vacuum distillation may reduce the energy consumption comparing to traditional distillation under ambient pressure. Effects of parameters (distillation time, vacuum, temperature, particle size and distillation area) on sulfur recovery rate were presented. High sulfur recovery rate was observed with relative pressure lower than –0.09 megapascal (MPa). Sulfur recovery rate increased sharply with temperature in the range of 140–220 °C, while temperature above 220 °C only slightly promoted sulfur recovery. Smaller DLR particle size and larger distillation area facilitated mass and heat transportation, and resulted in higher sulfur recovery rates. The vacuum distillation separation technology reveals an alternative method to efficiently and economically separate elemental sulfur from zinc concentrate DLR. The impacts of operation parameters on the sulfur recovery rate were well documented in this study, and they are important for further optimizing the process and strategically applying the technology in a larger scale scenario.

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1. Introduction

Pyrometallurgical methods for producing zinc metal have always been plagued with environmental pollution issues, such as toxic emissions of sulfur dioxide (SO₂), arsenic, and mercury [1]. Therefore, a hydrometallurgical direct leaching process was developed and commercialized to avoid the air pollution concerns related to the high temperature pyrometallurgical methods [2]. During the entire direct leaching process, sulfur in the zinc sulfide ores transfers into the zinc concentrate direct leaching residue (DLR). During the leaching process, sulfur stayed in the readily storable and saleable elemental phase, and the SO₂ emission was eliminated. In addition, the direct leaching process has a lower operation cost and higher zinc recovery rate comparing to the traditional pyrometallurgical methods [3]. Due to these advantages, direct leaching process for zinc metal production attracted a lot of attention from refinery industries.

The direct leaching process for zinc metal production was first commercialized by the Cominco Limited at Trail Zinc Refinery (BC, Canada) in 1981 [2]. At least four zinc pressure leaching plants were commissioned in the last decades of 20th century [4,5]. In recent years, direct leaching process was successfully implemented by many other metal refineries [6]. As a consequence, more DLRs were produced with increasing metal production from the direct leaching process. The DLR was characterized as acid-producing and harmful to the environment, due to the fact that sulfur component reacts with oxygen and water to form thiosalts and sulfuric acid [7]. Other hazardous metals existing in the DLR also posed massive disposal challenges. From the recycling perspective, sulfur and metal components in the DLR are thought to be precious resources rather than wastes. Recovery of them from the DLR was economically sound and safer than just storing the DLR alone. Currently, recovery of elemental sulfur from the DLR was only practicing in a few refineries, e.g., the Trail Refinery employed a hot filtration method to recover elemental sulfur from the zinc concentrate DLR [2]. It is difficult to obtain a fluid melt in the melting and filtration steps, when elemental sulfur content in DLR is less than 70% [7]. Therefore, floatation of DLR was usually

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integrated with the hot filtration process to obtain a satisfactory sulfur recovery performance [7]. This raised the cost to recover sulfur and dispose DLR. To reduce the operation cost, DLRs were generally treated as solid wastes and discarded in most zinc refineries, especially those in developing countries like China. It is urgent to develop a cost-effective technology to properly handle the DLR and recover elemental sulfur.

The boiling point of sulfur (444.6 °C) is much lower than other major components of the DLR [8]. Thus, elemental sulfur in the DLR could be separated from other components by distillation method. Mackiw et al. [9] studied sulfur recovery from chalcopyrite leaching residue with elemental sulfur content of 38.02%. The experiment at 427 °C under ambient pressure showed a sulfur recovery rate of 100% and the sulfur purity can be higher than 99%. Lu et al. [10] conducted a small-scale (20 g per batch) experiment at 440–480 °C to separate sulfur from DLR. Sulfur with purity higher than 97% was obtained after distillation of the DLR under atmospheric pressure. Our previous study revealed the possibility of separation of elemental sulfur from zinc concentrate DLR using atmospheric distillation [14]. However, large energy consumption was needed for the atmospheric distillation process, especially when moisture content of the DLR was high and operation was continuous. The relative high temperature (around or higher than the boiling point of sulfur) for atmospheric distillation was responsible for the enormous power consumption [14]. Therefore, vacuum distillation which can be operated at lower temperatures could be an energy-saving method to separate elemental sulfur from the residues. Huang and He [11] adopted vacuum distillation (30-600 Pa) to separated sulfur from anode mud which contained more than 82% elemental sulfur. The sulfur recovery rate was higher than 97% and sulfur purity was higher than 99%. However, there were only a few studies on the separation of sulfur from sulfur containing residues using vacuum distillation, and no study was yet carried on the zinc concentrate DLR.

In this study, vacuum distillation was employed to separate elemental sulfur from the zinc concentrate DLR for the first time. Effects of distillation duration, vacuum, temperature, particle size, and distillation area on elemental sulfur recovery were systematically investigated. The ultimate goal is to develop an effective and economically feasible technology to recover elemental sulfur from the zinc concentrate DLR, which would facilitate an extensive application of the direct leaching process in metal production industries.

2. Experimental

2.1. Characterization of direct leaching residue, distillation concentrate and sulfur product

DLR samples were collected from a zinc refinery where direct oxygen-rich leaching process was adopted to produce the zinc metal. Moisture content of the DLR samples was examined by the atmospheric drying method using a drying oven. Mineral components of the DLR samples and distillation concentrate (DC) samples were characterized by X-ray diffraction (XRD) using a diffractometer (PANalytical X'Pert PRO) equipped with a graphite diffracted-beam monochromator. The XRD patterns were recorded over a 2θ interval of 5–60°, with a step size of 0.02° and a counting time of 10 s per step. The morphology of the DLR samples and DC samples were investigated by a field emission scanning electron microscope (FE-SEM, FEI Sirion200). Thermal analysis was conducted on a thermo-balance (NETZSCH STA 409C), which composed of a furnace with silicon carbide heater and a sample holder (HIGH RT2). 10.0 mg sieved DLR sample was heated in pure nitrogen (flow rate of 100 ml/min) under atmospheric pressure, with a heating rate of 10 °C/min from room temperature to 500 °C. The sample was then kept at 500 °C for 1 h to get the thermogravimetry (TG) and differential thermo-gravimetry (DTG) curves. Elemental sulfur contents of DLR, DC, and final recovered sulfur were measured using a gravimetric method according to China National Standards (GB/T 2449-2006) [12]. All chemical reagents used in this study were at analytical grade.

2.2. Distillation experiments

As shown in Fig. 1, a vacuum distillation experimental system was built to separate elemental sulfur from zinc concentrate DLR. In each test, a certain amount of DLR was loaded to a porcelain crucible placed in a Pyrex reactor. The reactor was placed in a tubular furnace to precisely control the distillation temperature. A vacuometer was inserted into the reactor to measure the vacuum degree for each run. Two condensers were placed downstream to the distiller to condense sulfur vapor. In the first condenser, water was used as the coolant to lower the temperature of distillation gas. The large volume of the second condenser warranted enough residence time which is crucial for heat transfer and gas-particles separation. Solid or liquid particles were commonly found at the outlet of the second condenser. These particles were filtered before proceeding to the vacuum pump to prevent clogging or corrosion. The sulfur recovery rate was calculated based on the mass difference of the samples in the crucible before and after each run.

The parameters and conditions of the five sets experiments are summarized in Table 1. In Set I, 3.0 g DLR was used to investigate sulfur recovery rates during an entire vacuum distillation process. Six distillation experiments were conducted at 200 °C, -0.1 megapascal (MPa), with distillation duration of 10, 20, 30, 40, 50 and 60 min, respectively. In Set II, vacuum distillation was under different vacuum degree at 200 °C. Set III experiments were designed to identify the effect of distillation temperature under pressure of -0.1 MPa, which was identified to be the optimal vacuum degree in Set II. In Set IV, experiments were conducted to study the effect of DLR particle size on sulfur recovery rate. Sieves with 60, 100, and 180 meshes were used to separate the DLR samples into four portions with different particle sizes. For Set II, III, and IV experiments, the duration for each distillation process was 30 min. Set V experiments studied the effect of distillation area on the distillation performance. Distillation area was adjusted by changing the



Fig. 1. Schematic diagram of the experimental system.

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