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

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A R T I C L E   I N F O

A B S T R A C T

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 (SO_2), 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 SO_2 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 thermo-gravimetry (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
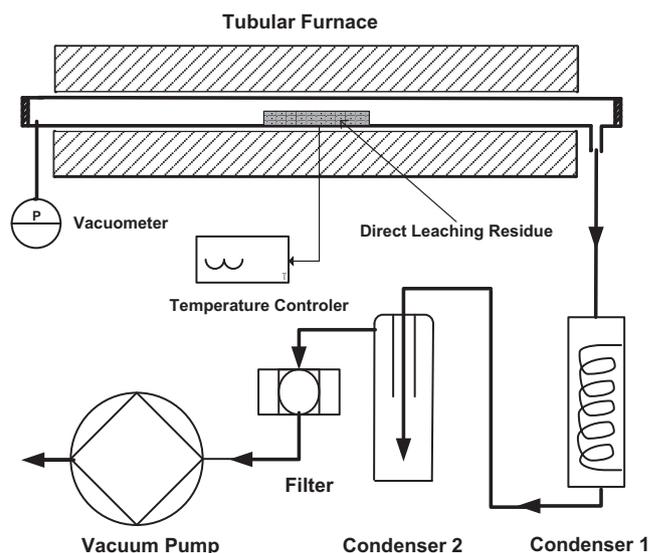

Fig. 1. Schematic diagram of the experimental system.

Table 1
Experimental conditions.

Experiments	Vacuum (MPa)	Temperature (°C)	Particle size (mm)	Distillation area (cm²/g)
Set I	−0.10	200	>0	2.97
Set II	−0.06, −0.07, −0.08, −0.09, −0.10	200	>0	2.97
Set III	−0.10	140, 180, 200, 220, 260, 300	>0	2.97
Set IV	−0.10	220	0–0.083, 0.083–0.147, 0.147–0.246, >0.246	2.97
Set V	−0.10	260	>0	1.51, 2.97

size of porcelain crucible in the Pyrex reactor. Experiment under each condition was triplicate.

At the beginning of each experiment, the DLR sample was balanced to obtain the precise feeding weight of DLR (W_{DLR}). The weight of elemental sulfur in the feeding DLR (F_{sul}) was obtained by deducting the weight of water (W_{wat}) from W_{DLR}, and then multiplying by the percentage composition of elemental sulfur. The DLR was then distilled under vacuum to separate sulfur from DC. After distillation and cooling down, solid matters in the crucible was weighed to get the weight of DC (W_{DC}). The difference between W_{DLR} and W_{DC} was considered to be the weight of water and elemental sulfur evaporated during the distillation process. Water in the DLR was assumed to be completely evaporated during the distillation, due to the fact that water is much more volatile than elemental sulfur. Assuming all elemental sulfur evaporated can be recovered in the subsequent processes, the elemental sulfur recovery rate (R_{sul}) was obtained according to the following equation:

$$R_{sul} = W_{DLR} - W_{DC} - W_{wat}/F_{sul} \times 100\% \qquad (1)$$

3. Results and discussion

3.1. Characterization of direct leaching residue and distillation concentrate

The DLR samples in this study were existing in black powder form with averaging of 9.75% moisture content. The XRF analysis results showed that silicon dioxide (SiO_2), iron oxide (Fe_2O_3), zinc oxide (ZnO), and sulfur trioxide (SO_3) were the primary components of the DLR sample [14]. Averaging elemental sulfur content of the dry DLR sample was 58.21%.

The XRD patterns of the DLR and DC samples are shown in Fig. 2. Crystalline phases were identified by comparison with the International Centre for Diffraction Data (ICDD) files. As shown in
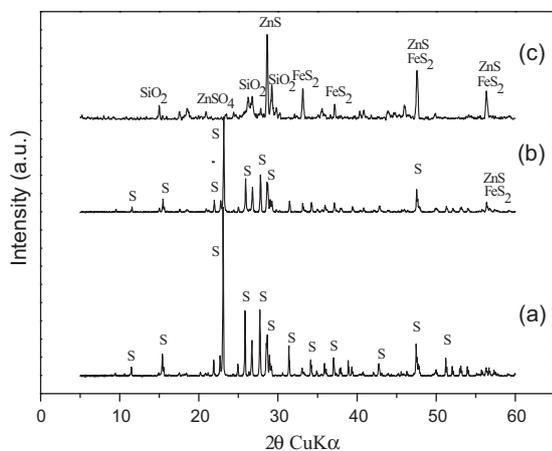
Fig. 2. XRD profiles of DLR and DC samples (a: DLR; b: DC after 30 min distillation at 140 °C; c: DC after 30 min distillation at 260 °C).

Fig. 2(a), only few weak peaks attributed to zinc sulfate ($ZnSO_4$), zinc sulfide (ZnS) and iron pyrite (FeS_2) were observed, crystalline sulfur was observed to be the dominating phase of the DLR, indicating that the DLR was enriched in elemental sulfur. The findings were in accordance with the chemical component analysis, which showed that elemental sulfur content of the DLR was higher than 50%. The results from XRD analysis of DC samples are shown in Fig. 2(b) and (c). As shown, the XRD profile of DC after distillation at 140 °C was similar to that of the DLR, i.e., peaks attributed to sulfur dominated. Most sulfur peaks disappeared for the DC sample after distillation at 260 °C, while peaks from $ZnSO_4$, ZnS, SiO_2, and FeS_2 dominated. The XRD results indicate that distillation temperature is a critical factor for vacuum distillation.

The FE-SEM imagines of DLR and DC samples are shown in Fig. 3. As observed in Fig. 3(a) and (b), most fine DLR particles joined together and existed as irregular particles with diameter larger than 10 μm. Most DLR particles were observed to be extremely smooth. DC particles with different structures varied from nanoscale to micron-scale, and most DC particles were smaller than 2 μm in diameter. Different from DLR particles, most DC particles had a rough surface and separated from others. This indicates that elemental sulfur scattered on particle surface before the distillation process and then evaporated during the distillation process. This is agreed with the XRD results of which most sulfur peaks disappeared after distillation.

The TG and the DTG curves are illustrated in Fig. 4 [14]. There were three distinct weight loss stages. The weight loss at temperature ranging from 90 to 280 °C was due to dehydration. The limited weight loss around 100 °C indicates that most moisture content in the DLR sample existed either in inner pores of the DLR or in the form of crystal water. The other two weight loss peaks distributed in the temperature range of 280–360 °C and 360–460 °C, respectively. Elemental sulfur on the DLR particles' surface evaporated easily, and hence resulted in the first weight loss stage raging from 280 to 360 °C. Besides sulfur on the particle surface, sulfur in inner part or pores of the DLR particles can also evaporate. However, it is difficult for sulfur vapor to transfer from particle interior to the gas phase. Moreover, capillary condensation could take place in mesopores [15] of the DLR particles, which would make sulfur evaporation more difficult. Therefore, higher temperature was needed for evaporation of sulfur in the inner part or pores of the DLR particles. The weight loss peak in the higher temperature range (360–460 °C) was attribute to the evaporation of sulfur in the inner part or pores of the DLR particles. It should be noted that the thermal analysis was conducted around atmospheric pressure. The TG curve clearly showed that almost no sulfur evaporation occurred under atmospheric pressure when temperature was below 280 °C. Vacuum was responsible for sulfur evaporation in the temperature range studied.

3.2. Effect of distillation duration

To investigate sulfur recovery rate during different period of vacuum distillation process, time-series experiment was conducted at 200 °C with a vacuum of −0.1 MPa. Sulfur recovery rates of vacuum distillation processes as a function of time are shown in
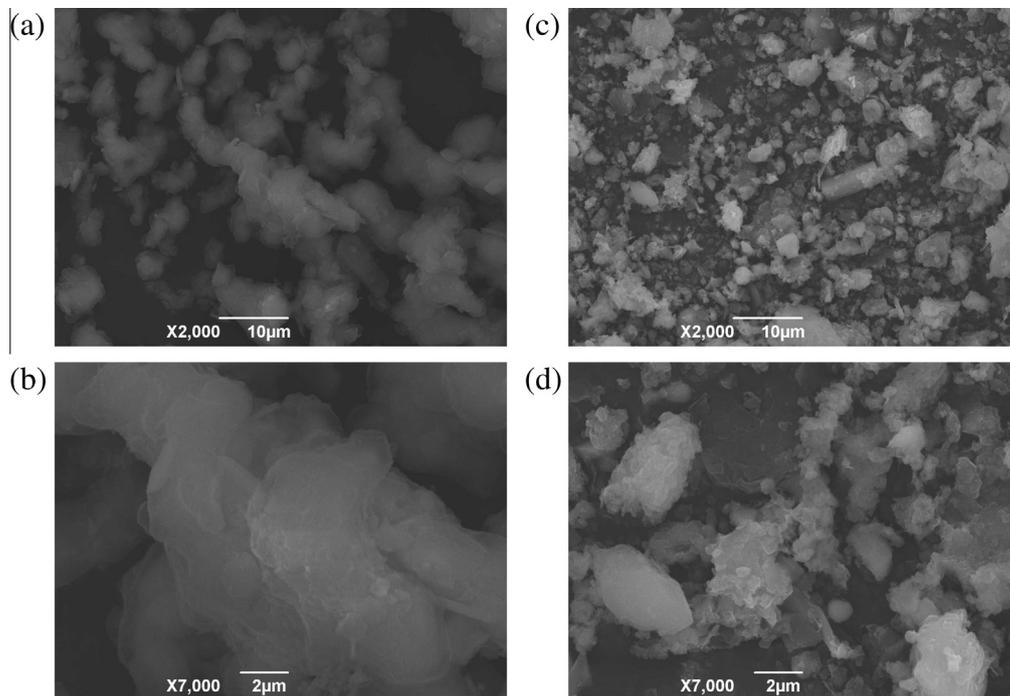
Fig. 3. SEM images of DLR and DC samples (a: DLR sample; b: partial enlarged view of image a; c: DC sample; d: partial enlarged view of image c).
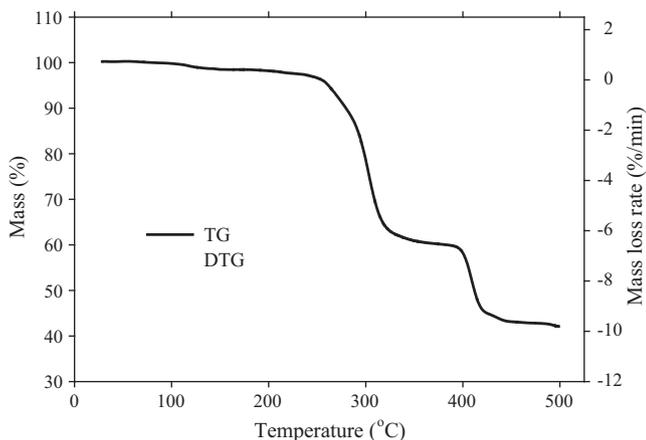

Fig. 4. Thermo-gravimetry (TG) and differential thermo-gravimetry (DTG) curves of the DLR sample [14].

Fig. 5. Effect of distillation duration on sulfur recovery rate.

Fig. 5. R_{sul} for the first 10 min was lower than 50%, while it increased sharply from 44.3% to 92.3% during 10 to 30 min period of distillation. Further extending of distillation duration to 40 min resulted in an R_{sul} of 97.9%, and no observable increasing after 40 min. The result indicates that almost all elemental sulfur in the DLR sample was separated in a distillation process shorter than 40 min under the experimental condition. The purity of sulfur produced under this condition was averaging 97.8%. In additional to the high recovery rate, the distillation temperature of 200 °C was much lower than the distillation temperature adopted for atmospheric distillation of sulfur which is above 400 °C [14]. Lower distillation temperature would probably result in less power consumption for the DLR disposal and sulfur production [16]. In sum, recovery of elemental sulfur from zinc concentrate DLR using vacuum distillation is technologically practicable and probably economical.

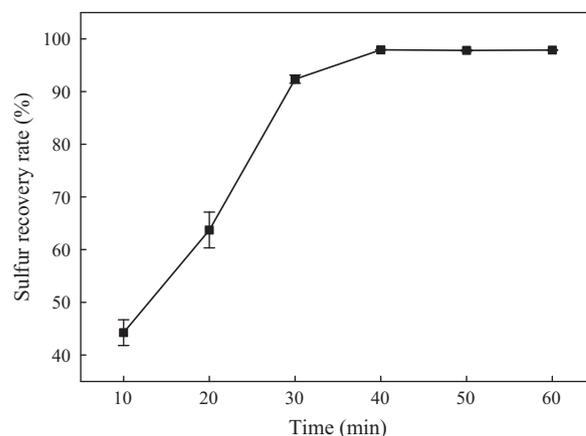The experiments showed a near 100% of saturated R_{sul} when distillation duration was longer than 30 min. Hence, the experimental length was fixed at 30 min as the optimal distillation duration, to study the effects from other factors (vacuum degree, temperature, and particle size).

3.3. Effect of vacuum degree

It is common sense that violent evaporation only occurs when temperature raises to boiling point. In contrast, violent evaporation can also happen far below boiling point with the presence of vacuum [13]. Effect of vacuum degree on sulfur recovery rate as a function of vacuum degree at 200 °C is shown in Fig. 6. The x-axis is the value of vacuum degree which is represented by relative pressure compared to atmospheric pressure. Sulfur recovery was negligible when the vacuum degree was above −0.06 MPa. Sulfur recovery rates slightly increased to about 20% when relative pressure reached −0.08 MPa, indicating that sulfur evaporation at 200 °C was insignificant when relative pressure was higher than −0.08 MPa. A notable increase of R_{sul} was observed when relative
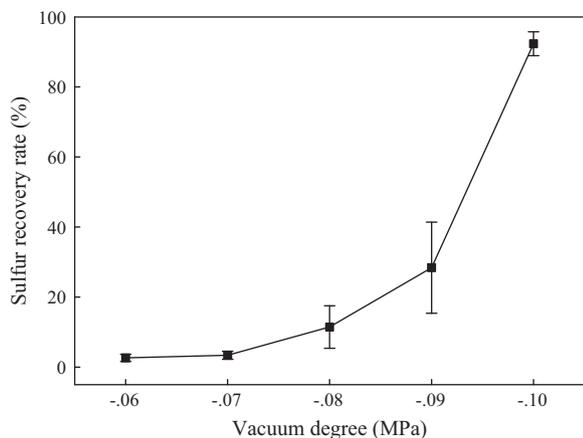
Fig. 6. Effect of vacuum degree on sulfur recovery rate.

Fig. 8. Effect of particle size on sulfur recovery rate.

pressure decreased from -0.08 to -0.10 MPa, with an R_{sul} of 92.4% at -0.10 MPa. This demonstrates that sulfur can evaporate violently at 200 °C under -0.10 MPa, led to more than 90% elemental sulfur separated from DLR within 30 min. Further decreasing the relative pressure was difficult for the experimental system in this study and certainly energy consuming. Therefore, -0.10 MPa was adopted for further experiments as an optimized vacuum degree.

3.4. Effect of distillation temperature

Distillation temperature is an influential parameter and can affect the sulfur evaporation below the boiling point under vacuum condition. Set III experiments were designed to study the effect of distillation temperature on sulfur recovery rate under vacuum degree of -0.10 MPa. As shown in Fig. 7, sulfur recovery rate higher than 90% was observed when temperature was higher than 200 °C. R_{sul} increased exponentially in the temperature range of 140–220 °C. Sulfur evaporation was promoted with higher temperature and led to more sulfur extraction from the DLR. The XRD profiles of DCs after distillation at 140 °C and 260 °C are presented in Fig. 2(b) and (c), respectively. For DC after distillation at 140 °C, the XRD profile was very similar to that of DLR shown in Fig. 2(a). The XRD results indicate that only few sulfur evaporated during the vacuum distillation at 140 °C, with a sulfur recovery rate of 10.7%. For DC after distillation at 260 °C, peaks attributed to $ZnSO_4$, ZnS, SiO_2, and FeS_2 dominated, while sulfur was absence. Most sulfur was effectively separated from other components of the DLR during the vacuum distillation at 260 °C. Elevation of temperature
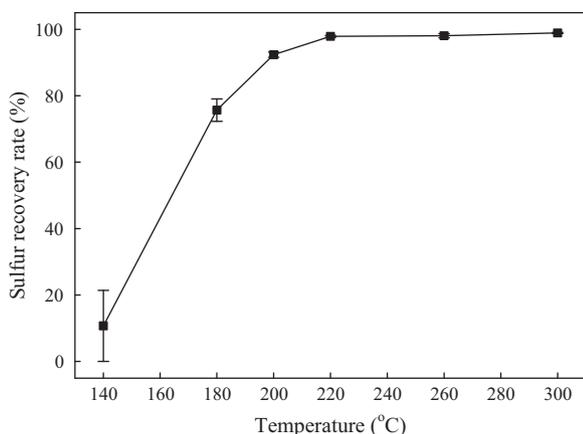
from 220 to 300 °C only slightly increased sulfur recovery, due to the fact that it was already above the sulfur boiling point at -0.10 MPa [13].

3.5. Effect of particle size

The DLR samples were sieved into four portions with different particle sizes, i.e., 0–0.083, 0.083–0.147, 0.147–0.246, and >0.246 mm. As shown in Fig. 8, R_{sul} of 80.4% was observed for DLR particles with diameter larger than 0.246 mm. R_{sul} values were inversely proportional to the particle sizes. Smaller DLR particle facilitated mass and heat transportation, and hence resulted in higher sulfur recovery rate. Over 98% sulfur evaporated from the DLR particles with size smaller than 0.147 mm, during the 30 min distillation at -0.10 MPa. Further decrease of particle size to smaller than 0.083 mm, negligible increase of R_{sul} was observed.

3.6. Effect of distillation area

Besides above-mentioned parameters, distillation area also impacts the performance of vacuum distillation process. Effect of distillation area on sulfur recovery by vacuum distillation at 260 °C is depicted in Fig. 9. R_{sul} for distillation area of 2.97 cm^2/g was higher than that for distillation area of 1.51 cm^2/g all the time. Low sulfur recovery rate (<40%) was observed for the distillation process with duration shorter than 10 min for both distillation area conditions. The result verified the time-series experiment conducted at 200 °C in Set I. During distillation from 5 to 15 min, small and large distillation area experiment had a different increasing
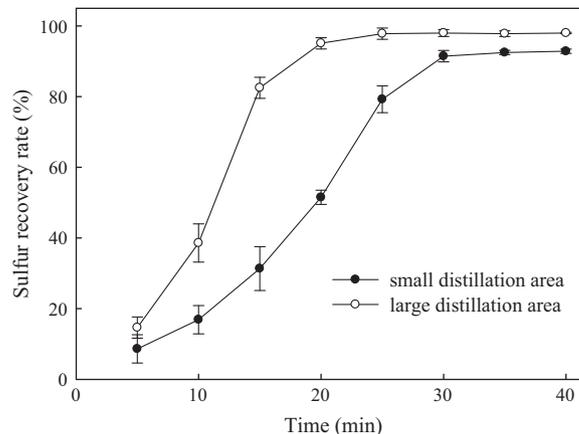

Fig. 7. Effect of distillation temperature on sulfur recovery rate.

Fig. 9. Effect of distillation area on sulfur recovery rate.

pattern of R_{sul}. At 15 min, sulfur recovery rates for two distillation area conditions exhibited the largest difference. R_{sul} for distillation area of 2.97 cm^2/g was 82.5%, while R_{sul} for distillation area of 1.51 cm^2/g was 31.3%. Smaller distillation area was corresponding to larger DLR thickness with same amount of DLR. This tends to inhibit mass and heat transportation. Difference in R_{sul} became smaller when distillation duration reached 30 min. This was due to the fact that the mass transfer dynamic for the larger distillation area condition became smaller, as most sulfur evaporated during the first 15 min. When distillation duration was longer than 30 min, R_{sul} maintained at the same level for both conditions (98% and 92.5% for distillation area of 2.97 cm^2/g and 1.51 cm^2/g, respectively).

4. Conclusion

Separation of elemental sulfur from zinc concentrate DLR using vacuum distillation was systematically studied for the first time. The results proved that efficient recovery of elemental sulfur from the DLR using vacuum distillation is achievable. Under optimal operation conditions, sulfur recovery rate and sulfur purity were higher than 98.0% and 97.8%, respectively.

Decrease of relative pressure from −0.06 to −0.10 MPa resulted in more elemental sulfur recovered, with more than 90% elemental sulfur separated from DLR at 200 °C, −0.10 MPa in 30 min. When temperature was lower than 220 °C, increase of temperature significantly facilitated sulfur evaporation and recovery. Further increase of temperature from 220 to 300 °C slightly promoted sulfur recovery. The temperature requirement (200–300 °C) for sulfur recovery using vacuum distillation was much lower than the traditional atmospheric distillation, which may significantly reduce the energy consumption and operation cost. Smaller DLR particle and larger distillation area facilitated mass and heat transportation, and hence resulted in higher evaporation rates and higher sulfur recovery rates for a limited distillation duration.

In sum, this study explored the possibility of elemental sulfur recovery from zinc concentrate DLR using vacuum distillation. The impact of operation parameters on sulfur recovery rate was also investigated. The results are of fundamental importance in developing separation and purification technologies for handling DLR. Future studies should be conducted on a large-scale system to investigate the feasibility and economics of this technology.

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