



Froth flotation separation of carbon from barium slag: Recycling of carbon and minimize the slag



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ARTICLE INFO

Article history:

Received 11 August 2020

Revised 31 October 2020

Accepted 18 November 2020

Available online 5 December 2020

Keywords:

Barium slag

Carbon recovery

Flotation

Minimize waste discharge

ABSTRACT

Barium slag is a kind of solid waste derived from the carbon reduction process of producing barium salt. Carbon is one of the main components in barium slag with a content of more than 10%. In this study, a barium slag was characterized using XRF, XRD and SEM-EDS, and froth flotation test was introduced to recover the carbon in the barium slag. In the process of froth flotation test, diesel was selected as a collector and terpenic oil was selected as a frother for carbon separation. The flotation influences of pulp pH, collector dosage, frother dosage and flotation time were investigated. The results showed that the obtained carbon concentrate had a carbon grade of 63.25% and its recovery was 82.70% under the conditions of pH 8.50, diesel 600 g/t, terpenic oil 500 g/t, and flotation time 5 min. SEM-EDS analysis results revealed that the flotation concentrate was coarse and its particle sizes had a homogeneous distribution. The findings of this research provide a new pathway for barium slag utilization. The carbon obtained from the flotation test in this study can be recycled as raw materials, while the reduction of the remaining tailings can be utilized as building material additives.

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1. Introduction

Barium slag (BS) refers to the solid waste generated in the process of producing barium salt from barite by carbon reduction (Jamshidi and Salem, 2010; Guzmán et al., 2012; Gu et al., 2019). In the industrial, the most critical reaction is thermal reduction of barium sulfate, in which process coal/coke is added to a rotary kiln or fluidized bed reactor to react with barite to produce barium sulfide at 950–1100 °C (Salem and Osgouei, 2009). After the reduction reaction, the obtained production of barium sulfide solution will be separated from the insoluble precipitate by water leaching and filtration (Jamshidi and Ebrahim, 2008). The insoluble precipitate is mainly comprised of unreacted raw materials (including residues of barite ore and coal/coke) and some new generated phases. Thus, carbon remains one of the main components of BS due to the fact that the amount of reducing reagent (coal/coke) added usually accounts for about 20% of the weight of barite (Jamshidi and Ebrahim, 2008). Meanwhile, BS contains high content of soluble barium which is highly toxic when liberating to the surrounding water (Lamb et al., 2013). As thus the stockpiling

of BS occupies lands and poses considerable negative environmental threats (Guo et al., 2020). Therefore, it is important to propose new methods to recycle or reuse of BS.

The acid-soluble barium salts in BS can be used to prepare barium chloride and barium nitrate (Dong et al., 2003). BS has also been reported to produce non-load-bearing bricks (Yang et al., 2008) or as an additive in cement clinker (Jiang, 2007). However, there are few studies on the carbon recovery and utilization of BS. The application of slag or ash to building materials is affected by unreacted carbon included (Mohebbi et al., 2015; Xu et al., 2017; Wierzchowski et al., 2020). Thus, the carbon contained in BS may limit the industrial utilization of BS. In addition, carbon in BS can be considered as a kind of potential resource. Separation and recovery of carbon from BS are capable of reducing the waste slag volume, obtaining useable carbon products, and prompting the residual slag more applicable.

Froth flotation is a separation method based on the different surface properties of minerals (Harvey et al., 2002; Sun et al., 2009; Yalcin and Kelebek, 2011; Xia et al., 2019a). The flotation process is mainly the collision and attachment of hydrophobic particles and bubbles (Harvey et al., 2002; Nikolaev, 2019). Most of the carbon in BS comes from coal/coke, and this kind of material belongs to non-polar compounds, which have the characteristics of good natural floatability and strong hydrophobicity (Yu et al., 2012). Therefore, the carbon can be effectively separated by

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flotation. To improve the floatability of carbon, non-polar hydrocarbon oils such as diesel and kerosene are widely used as collectors (Polat et al., 2003; Taha et al., 2017; Xing et al., 2017; Xia et al., 2019b; Xu et al., 2019; Zhu et al., 2019a). Surfactant as a frother can enhance the stability of bubbles (Cho and Laskowski, 2002). Terpenic oil or methyl isobutyl methanol is often used as foaming agents for coal/coke flotation (Harvey et al., 2002). There are many literatures on carbon flotation, but most of the carbon flotation materials are from fly ash (Altun et al., 2009; McCarthy et al., 2013; Uçurum et al., 2011; Walker and Wheelock, 2006; Zhang and Honaker, 2015). However, there are few researches on carbon recovery from BS by flotation.

The present study was undertaken to recover carbon from BS through froth flotation using diesel and terpenic oil. After characterizing BS samples, the effects of slurry pH, collector dosage, frother dosage, and flotation time on flotation performance were investigated to determine the optimal conditions for carbon flotation from BS.

2. Materials and methods

2.1. Samples and reagents

The BS material used in this experiment was collected from a barium salt factory, Guizhou, China. The chunk BS sample was roughly crushed using a jaw crusher until the particle size was less than 2 mm. BS samples of 300 g crushed less than 2 mm were taken for particle size distribution analysis. Particle size distribution of the BS was analyzed by manual screening using the following sieve sizes: 80 mesh (175 μm), 160 mesh (97 μm), 200 mesh (74 μm), 400 mesh (38 μm). The results of particle size distribution with carbon contents are shown in Table 1.

From the results of particle size distribution analysis, proper particle sizes were selected for flotation experiments. Specifically, a ball mill was then used to grind the BS to less than 0.074 mm accounting for about 65%. The obtained powders were stored in a sealed bag after drying at 105 °C for 24 h for the flotation experiments. The diesel and terpenic oil used in the experiment are industrial grade reagents. The original pH value of BS solution was approximately 11.60, and the solution prepared by analytical sulfuric acid (98.0%, H₂SO₄) was used as the pulp pH regulator. Deionized water was used in the experimental process.

2.2. Characterization methods

The main chemical compositions of raw BS sample were determined using X-ray fluorescence spectroscopy (XRF, PANalytical PW2424, Netherlands). The sample was fused with weight loss in the XRF determination process, and a loss-on-ignition at 1000 °C was separately determined to produce a “total”. The carbon contents of the BS and the flotation products were analyzed using an elemental analyzer (vario MACRO cube, Germany). To ensure the accuracy of the analysis results, hydrochloric acid was used to

remove carbonate minerals in BS or flotation products before carbon contents were measured.

Powder X-ray diffractometer (XRD, PANalytical Empyrean, Netherlands) with Cu Kα radiation was conducted to measure the main mineral phases composition of BS. Sample was prepared by compaction into a silicon sample holder, and a 2θ range between 5 and 70 degree was scanned. Samples for scanning electron microscopy (SEM) observation were viewed in a JSM-6460LV and a FEI Scios scanning electron microscope with an energy dispersive spectroscopy (EDS).

2.3. Flotation tests

Single-stage tests were conducted for flotation to obtain the optimal parameters such as pulp pH, collector dosage, frother dosage and flotation time. All flotation tests were performed using a mechanical agitation flotation machine (RK/FDIII0.75L, Wuhan, China).

In each test, 100 ± 0.2 g of BS sample (65% passing 200 mesh) was placed into a 750 mL flotation cell filled with 700 mL water. Each feeding BS sample was first prewetted in the flotation cell for 3 min to prepare flotation pulps by controlling the impeller at a constant speed of 2000 rpm. The impeller speed was used during the whole process of conditioning and flotation stages. The pulp pH was adjusted using aqueous solutions of H₂SO₄ with agitating for 2 min, after which diesel was added as a collector with agitating for 5 min. Then, terpenic oil was added to the cell with agitating for 3 min. Subsequently, air flow was introduced into the cell at a rate of 1.6 L/min, in the process of which bubbles occurred with sizes of 5–10 mm. After a certain flotation time, the froth products were automatically collected by a scraper blade with a speed of 60 times per min. The products and the tailings were filtered using 40 μm pore-size filter paper, dried at 105 °C for 24 h, and weighed for characterization.

According to the previous literature (Nie et al., 2015) and our preliminary experiments, the parameters of pulp pH, collector dosage, frother dosage and flotation time in the flotation process were set as a range of 11.60–6.50, 200–1000 g/t, 300–700 g/t and 1–7 min, respectively. The flowsheet of the flotation process is shown in Fig. 1.

The yield and recovery of flotation products were calculated using the following Eqs. (1) and (2) (Huang et al., 2012; Zhang and Liu, 2019):

$$W_p (\%) = (M_p / M_f) \times 100\% \tag{1}$$

$$\varepsilon (\%) = (W_p \times \beta_p) / \alpha_f \tag{2}$$

where M_p represents weight of dry flotation products (g), M_f is weight of dry feeds of BS (g), W_p means the yield of flotation products (%), β_p represents the carbon content of flotation products (%), α_f is the carbon content of BS (%), and ε means the carbon recovery of flotation products (%).

Table 1
The particle size distribution analysis of BS.

Size range (μm)	Yield (%)	Carbon (%)	Cumulative yield (%)	
			Overscreen	Underscreen
>175	60.10	25.48	60.10	100.00
97–175	14.97	9.13	75.08	39.90
74–97	6.54	6.24	81.62	24.92
38–74	3.18	4.21	84.80	18.38
<38	15.20	2.57	100.00	15.20
Total	100.00	17.61		

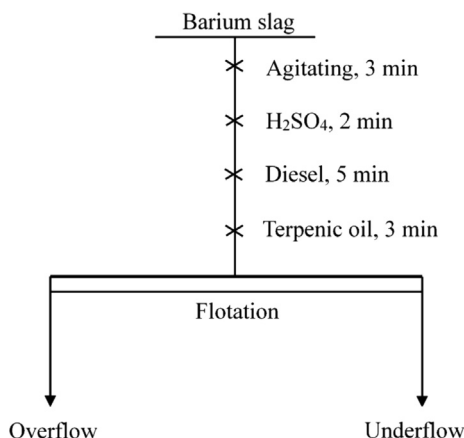


Fig. 1. Flowsheet of flotation test for BS.

3. Results and discussion

3.1. Characterization of BS sample

The chemical compositions of BS sample are important to subsequent carbon separation. The BS carbon content measured by the elemental analyzer in this study was 13.48%, which has the value of recycling. The XRF results of BS are given in Table S1, and elements in BS are represented by oxides. In addition to carbon, the main components of BS were BaO (37.60%), SiO₂ (15.52%), CaO (10.10%), Al₂O₃ (4.23%), Fe₂O₃ (3.29%), MgO (1.44%), etc. The LOI of BS reached 14.51%, a bit higher than carbon content. This was ascribed to other volatile components, such as absorbed water.

The main mineral phase composition results of BS are shown in Fig. 2. According to Fig. 2, the main mineral phases of BS were barite, barium carbonate, quartz, carbon, and silicates. Barite and carbon were raw materials and their presence in BS indicated that the carbothermal reaction was incomplete. Quartz and the silicates were from the raw barite ore (Han et al., 2015) and they can be observed in the XRD pattern, thus it can be concluded that they did not react completely during the carbothermal reaction process. Barium carbonate identified from the pattern was newly generated as byproducts. The small particles of barium carbonate and the residual minerals, such as barite and quartz, usually had associated relationships with carbon, which adhered to the surface of carbon particles. Therefore, these hydrophilic phases could increase difficulty in separating of carbon during the flotation process.

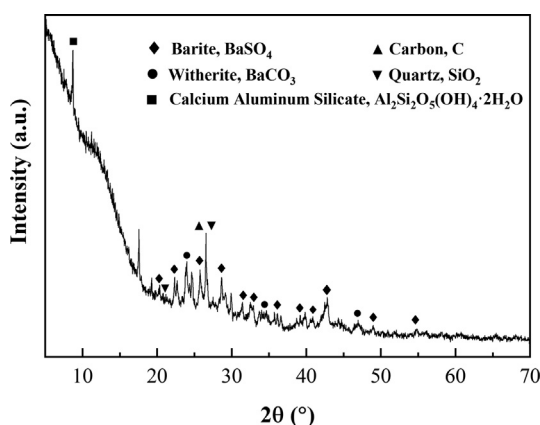


Fig. 2. X-ray diffraction pattern of BS.

3.2. Size distribution of BS

The particle size has great influence on flotation separation of target minerals (Zhang et al., 2013). With increasing of particles size (seen in Table 1), carbon content in BS indicated an increase trend. The particle size more than 175 μm accounted for approximately 60% in weight and the carbon content reached 25.48%. The yield of fine section (less than 38 μm) was as large as 15.20% with a carbon content of only 2.57%, and the non-carbon substances in this size range are easy to be carried into the flotation concentrate by machinery in the flotation process. To improve the efficiency of carbon separation, it is not necessary to grind particles too fine for flotation feed to reduce the entrainment phenomenon.

3.3. The effect of pulp pH

In the flotation process, mineral floatability, ionic composition in pulp and the activity of flotation agents are directly affected by pulp pH value (Ma et al., 2009; Wang et al., 2018). To obtain an optimal pH range in flotation process, the effect of pulp pH on carbon grade and recovery of flotation was studied under the conditions of diesel dosage 600 g/t, terpenic oil dosage 500 g/t, and flotation time 5 min, as shown in Fig. 3. The pulps pH was adjusted to five different pH levels, from natural pH 11.60 to 6.50. It is apparent that the carbon recovery increased from 28.33 to 82.70% when pH value decreased from 11.60 to 8.50, and the carbon recovery remained unchanged when pH decreases from 8.50 to 6.50. The graphical results can be explained by two reasons. On one hand, the interaction between negatively charged diesel and carbon particles is hydrophobic (Naik et al., 2005), which was probably controlled by the system pH value. Under higher pH, excessive OH⁻ can compete with the diesel to adsorb on the carbon surface (Huang et al., 2003), resulting in a poor flotation effect. At a suitable pH value, an appropriate amount of H⁺ or OH⁻ reacts with the functional polar groups that occur on the carbon surfaces such as -COOH, -OH, -CO, and makes them non-polar (Huang et al., 2003). The non-polar collector diesel can better adsorb on the surface of the non-polar carbon particles to improve the recovery of carbon particles.

On the other hand, soluble components in the pulp will interact with the flotation agent and mineral surface and affect the flotation effect (Qin and Hu, 1999). BS was reported containing soluble barium salt (Gu et al., 2019), and it also contains Ca, Mg, Fe, and Al species according to the XRF results of this study. With the change of pH, precipitation or adsorption phenomenon of these metals occurred (Liu et al., 1994), and this process may affect the

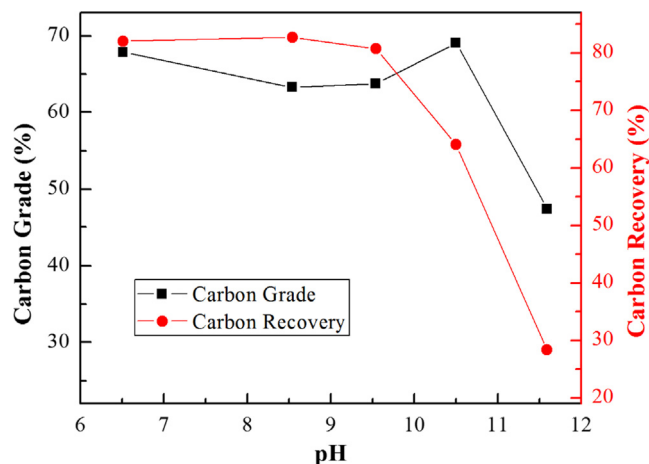


Fig. 3. The effect of pulp pH on carbon grade and recovery of flotation.

hydrophilicity of carbon particles. With the decrease of pH, Ca and Mg species dissolved into the solution as shown in Table S2, in which process the carbon surface became clean and could interact better with diesel, leading to increasing carbon recovery (Fig. 3). In addition, H₂SO₄ solution was used as pH regulator, and it could also obviously reduce the concentration of barium ion in pulp (Table S2). Thus, the optimal pulp pH was suggested at around 8.50, and the slurry was environmental non-hazardous for soluble barium ions (Gu et al., 2019; Guo et al., 2020).

3.4. The effect of collector dosage

The collector dosage is one of the key parameters affecting flotation performance in the flotation test (Uçurum, 2009). The addition of collector can improve the surface hydrophobicity of particles and affect the recovery of the target (Zhu et al., 2019b). The effect of diesel addition on the carbon properties of flotation was investigated under the conditions of pulp pH 8.50, terpenic oil dosage 500 g/t, and flotation time 5 min, as shown in Fig. 4. As diesel dosage increased from 200 to 600 g/t, the carbon recovery increased from 75.82 to 82.70%. It can be explained that the addition of diesel collector can cover the surface of carbon particles, reduce the surface tension of carbon, increase the hydrophobicity of carbon particles, and improve the recovery of carbon (Huang et al., 2003). The carbon recovery was reduced when the diesel exceeded 600 g/t. This may be due to the fact that adding too much diesel collector will counteract the effect of frother (Lynch et al., 1981), resulting in poor foam fluidities, longer foam residence time, and the transfer of carbon particles from the foam to the slurry. Based on the analysis of the results, the optimal collector dosage was 600 g/t.

3.5. The effect of frother dosage

The frother dosage also has an important influence on the grade and recovery of flotation (Tao et al., 2002). The effect of frother dosage on carbon flotation of BS is plotted in Fig. 5. With the increase of frother dosage from 300 to 700 g/t, the carbon recovery showed a tendency of ascending in first and then descending. The recovery of carbon increased indicated that more carbon particles were trapped on bubbles (Naik et al., 2005), and the amount of bubbles depended on the dosage of frother added. However, excessive terpenic oil added as a frother could promote its hydrophobic interaction (Fuerstenau and Pradip, 1982) with diesel (collector), which caused the hydrophobic part of terpenic oil to collect the

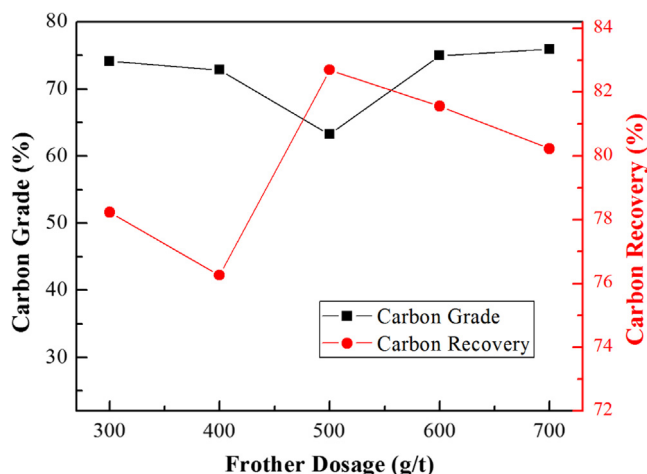


Fig. 5. The effect of terpenic oil dosage on carbon grade and recovery of flotation (pH 8.50, diesel 600 g/t, flotation time 5 min).

diesel that has been adsorbed on carbon particles. Meanwhile, the hydrophilic part (—OH group) of terpenic oil could form hydrogen bonding with the hydrated mineral matters in the pulp (Aston et al., 1989; Naik et al., 2005). The above process resulted in a decrease of carbon particles hydrophobicity. From the experimental results, the optimal frother dosage was selected 500 g/t.

3.6. The effect of flotation time

The objective of this section is to find the optimal flotation time. Fig. 6 shows the influence of flotation time under the conditions of the pulp pH 8.50, diesel dosage 600 g/t, and terpenic oil dosage 500 g/t. When the flotation proceeded only for 1 min, the carbon recovery could reach 60.23% with a carbon grade of 78.42%. As the flotation time increased from 1 min to 5 min, the carbon recovery increased from 60.23 to 82.70%. The results indicated that more carbon particles in pulp can be captured with the flotation time increased. After 5 min, the carbon recovery and grade remained almost unchanged. This could be assigned to the fact that the carbon particles that could be captured were in a certain amount when the collector and frother used were quantitative. To prolong the flotation time will lead to entrapments of other substances and the reduction of carbon grade as shown in Fig. 6. Considering the grade and recovery of carbon products, the optimal flotation time was suggested as 5 min.

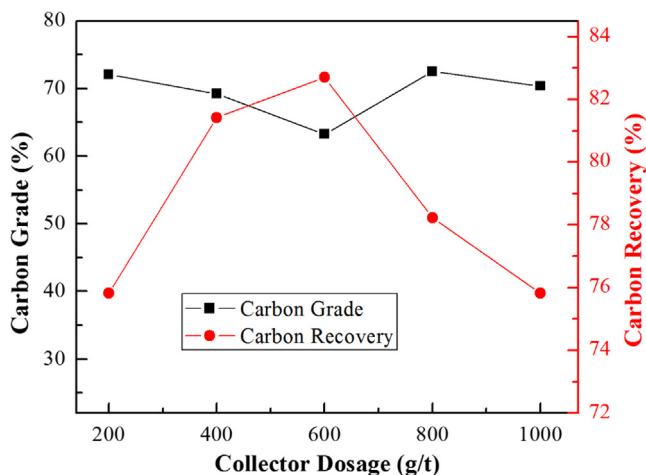


Fig. 4. The effect of diesel dosage on carbon grade and recovery of flotation.

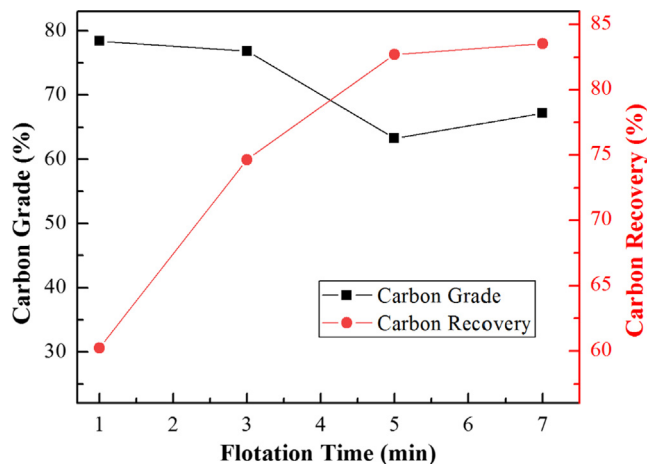


Fig. 6. The effect of flotation time on carbon grade and recovery of flotation.

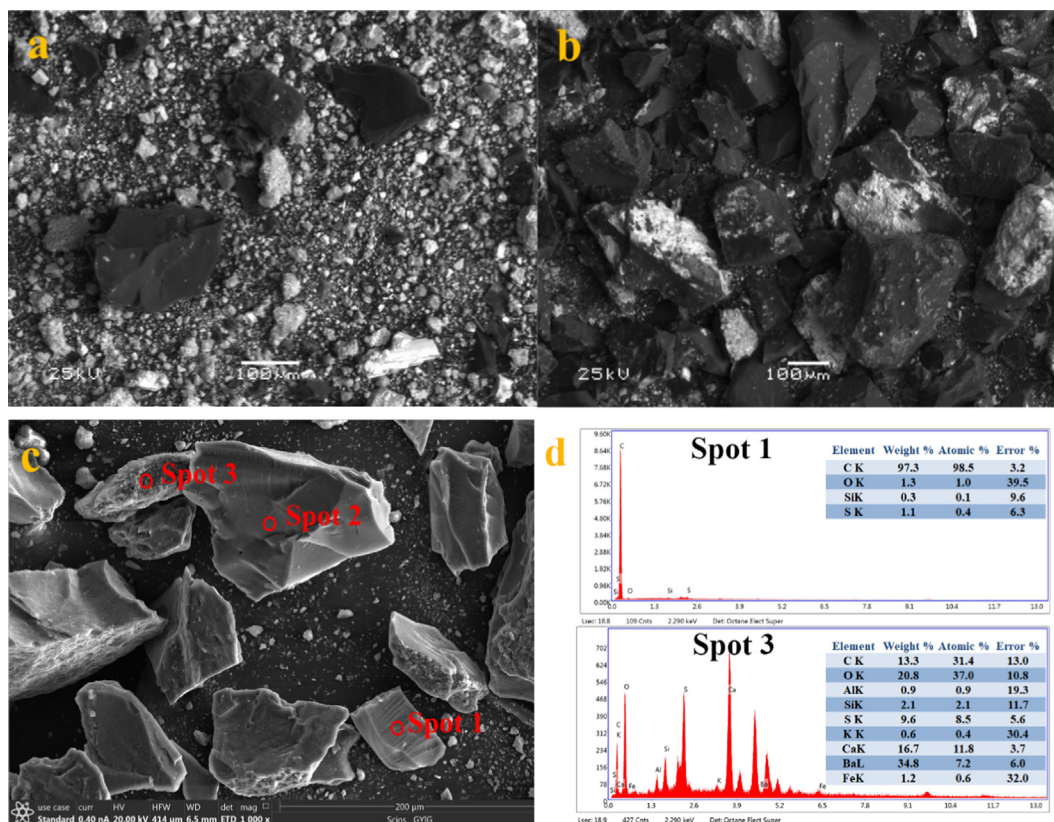


Fig. 7. SEM-EDS images of BS and the flotation concentrate.

3.7. Microstructure of BS and the flotation concentrate

The surface morphology and element distribution of BS and its concentrates were observed by SEM-EDS. The SEM images of BS and flotation concentrates are shown in Fig. 7. The raw BS particles in Fig. 7a were mainly composed of large black particles, some medium bright particles, and fine gray particles. The flotation concentrate as shown in Fig. 7b consisted of black particles, which were angular and irregular in shape. Most of the black particles had sizes greater than 100 μm (Fig. S1) with a few impurities adhered to their surfaces. The results revealed that some of the carbon particles were not completely separated from other substances in the BS, affecting the floatability of carbon.

A higher resolution SEM with energy spectrum analysis was used to further amplify the concentrate compositions (Fig. 7c and d). The results showed that the main elements at spots 1 and 2 (Fig. S2) were carbon, while the main elements at spot 3 were compounds of Ba, Ca, S, and other impurities. The results confirmed that the flotation concentrate was composed of carbon particles with uniformly sizes. From the comparison of their photos (Fig. S3), the flotation concentrate was obviously darker than the raw BS sample due to the high carbon content. Since the flotation concentrate was mainly carbon, it can be recycled as raw material in the carbothermal reaction process of barite.

4. Conclusions

Recovery of remnant carbon in barium slag was investigated to recycle the carbon and to minimize the discharge of the slag. In this study, a barium slag was characterized, and the results showed that the barium slag contained 13.48% of carbon. In addition, the barium slag used had chemical components of BaO (37.60%), SiO₂

(15.52%), CaO (10.10%), Al₂O₃ (4.23%), Fe₂O₃ (3.29%), etc., and had mineral composition of barite, barium carbonate, carbon, quartz and silicates.

The flotation test was conducted to obtain the carbon concentrates, and the parameters including pulp pH, collector dosage (diesel), frother dosage (terpenic oil), and flotation time, were investigated. The optimum froth flotation conditions for the barium slag were identified as pulp pH 8.50, diesel 600 g/t, terpenic oil 500 g/t, and flotation time 5 min. Under the conditions, carbon recovery achieved 82.70% with a grade of 63.25%. The results demonstrated that froth flotation using diesel as collector dosage was effective for carbon recovery from barium slag. The flotation carbon concentrate can be recycled, and flotation tailings with low carbon content can be easily used as building material additives eliminating influence of carbon.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

This work was supported by the National Natural Science Foundation of China financial support [U1812402; 41972048], and The Youth Innovation Promotion Association CAS (Hannian Gu).

Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.wasman.2020.11.022>.

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