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Experimental study on separation of oil shale and semi-coke by fluidization principle

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Abstract. *With the progress of technology, the development and utilization of oil shale are becoming more and more extensive. Oil shale is composed of organic matter and inorganic matter. The latter refers to minerals that cannot be utilized in the production process. On the one hand, due to the uneven distribution of inorganic material in oil shale, the density and composition of organic matter of different oil shales are different. On the other hand, due to the limited mining technology, oil shale gets often mixed with various impurities, which makes its purification more difficult and increases the respective cost. Based on the characteristics of uneven distribution of inorganic substances and high impurity density in oil shale, its separation experiments were carried out using a self-made separator. The results show that the separation efficiency of oil shale and semi-coke can reach 56.74% and 73.17%, respectively.*

Keywords: *fluidization, oil shale separation, total sulfur content, inorganic density.*

1. Introduction

As an emerging energy source, oil shale has become a focus of research and development in recent years. Oil shale is composed of organic matter and inorganic matter. The remaining inorganic matter is impurities. Because of the restricted exploitation conditions, oil shale gets mixed with various impurities. The presence of impurities makes the purification of oil shale more difficult and increases the respective cost. Therefore, it is of great significance to remove impurities from oil shale in the course of processing [1–5]. The distribution of inorganic substances in oil shale is not uniform. Some inorganic substances can be separated from oil shale through crushing, together with small particles. The relative density of organic minerals in oil shale is 1.20–2.00 g/cm^3, the density of inorganic minerals is higher than 2.20 g/cm^3, and impurities are mostly inorganic minerals involved in the process of exploitation. The separated organic minerals can be directly

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subjected to purification, which greatly eases the whole process of oil shale refining and reduces its cost [6–9].

If the semi-coke produced during the purification of oil shale is just discarded, it will mean a huge waste of energy and cause serious environmental pollution. Semi-coke is separated by the experimental platform, and the preliminary processing of oil shale is completed to facilitate its subsequent use. In recent years, dry separation technology has become more and more mature, in which a complete fluidized separation system is created on the basis of the gas-solid fluidization technology, including factors and conditions affecting the separation process of the gas-solid fluidized bed [10].

2. Experiment

2.1. Experimental material

The main materials in this experiment are oil shale and semi-coke. Oil shale is produced in Lulong County, Hebei Province, China. The experimental results show that the oil content of Lulong oil shale is 8.54%, the water content is 2.19%, the gas loss rate is 3.03%, and the semi-coke yield is 86%. Judged by its high oil content, 8.54%, Lulong oil shale is fit for refining oil by low temperature retorting technology and direct combustion. The other material used in this experiment is semi-coke, which is the final solid product of Lulong oil shale heated in a distillation furnace at 450–600 °C in the absence of air. Although gas and shale oil are obtained from pyrolysis of oil shale, some combustible substances remain in semi-coke, so semi-coke has the value of secondary utilization.

2.2. Experimental device

The experimental device design idea of this study is to install a separation device in the coarse powder separator in the original milling system of the pulverized coal boiler, and make as small changes to the equipment as possible. The experimental device is shown in Figure 1. The main body of the experimental platform is a completely symmetrical cylinder that gradually shrinks from top to bottom. The barrel is composed of two parts. The organic matter will fall from the large tube in the middle of the barrel, and the remaining heavy material will fall from the small tube below the feed port. The exhaust vent is mounted above the cylinder to ensure a slight positive pressure inside the cylinder. The centrifugal fan in the air supply system can provide 0.1 MPa air pressure, and the butterfly valve in the pipeline can control the experimental air flow. A total of six pressure measuring points were set on the side of the test bench body, and the data collector was connected to the computer to collect the pressure signal during the experiment.

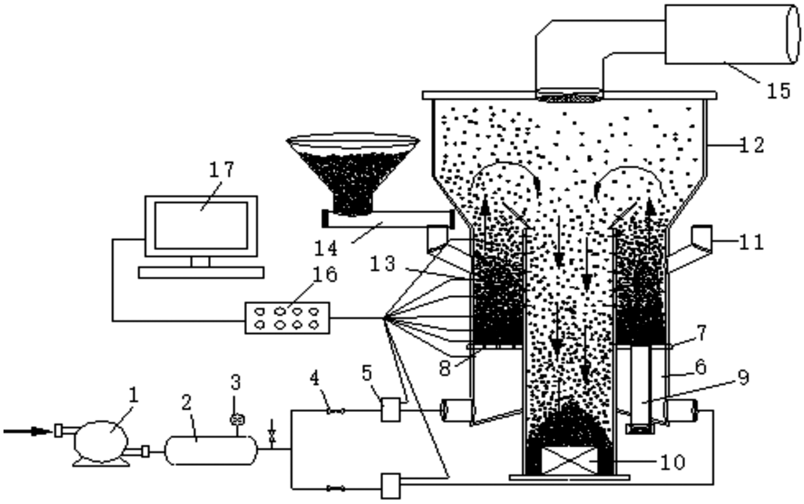


Fig. 1. Schematic diagram of the fluidized bed separation test system: 1 – high pressure centrifugal fan; 2 – ventilation pipeline; 3 – pressure gauge; 4 – butterfly valve; 5 – vortex flowmeter; 6 – equal pressure chamber; 7 – winding panel; 8 – wind cap; 9 – inorganic substances product discharge port; 10 – organic substances product discharge port; 11 – feed port; 12 – fluidized bed separation body; 13 – fluidized bed pressure measuring point; 14 – screw feeder; 15 – dust bag; 16 – data collector; 17 – computer.

2.3. Experimental principle

According to the measured equivalent diameter range of coarse particles in the return pipe of the crude powder separator, the particle size of oil shale is about 0.6 mm. The relative density of oil shale is about 1.6 g/cm^3, that of organic minerals in oil shale is about 1.2 g/cm^3, and the density of inorganic minerals is higher than 2.2 g/cm^3. The density difference between organic and inorganic substances of oil shale helps achieve the maximum separation effect. The fluidization performance of the particles in the fluidized bed will change with changing fluidization wind speed.

3. Experimental results and analysis

3.1. Analysis of separation efficiency

Separation efficiency is an important parameter reflecting the fluidization effect of the experiment. It refers to the ratio between the mass of the heavy experimental material and the total amount of the experimental feed after the separation experiment. It is expressed by Equation (1):

$$\eta = \frac{My - Mz}{My} \times 100\%, \tag{1}$$

where η is the separation efficiency, %; M_y is the original mass of heavy experimental material, kg; M_z is the mass of heavy material after experiments, kg.

The reason why light samples are not used in this study is that a small part of such samples will inevitably be discharged from the upper exhaust outlet of the test bench. In order to minimize the experimental error, we choose the mass of heavy samples to define the separation efficiency.

In this work, three feed mass amounts were used in five experiments. Five maximum wind speeds were recorded. The separation efficiencies at these wind speeds were compared, and the mechanism and optimal wind speed of separation were determined. Figure 2 shows the relationship between the efficiency and wind speed of separation of oil shale and semi-coke.

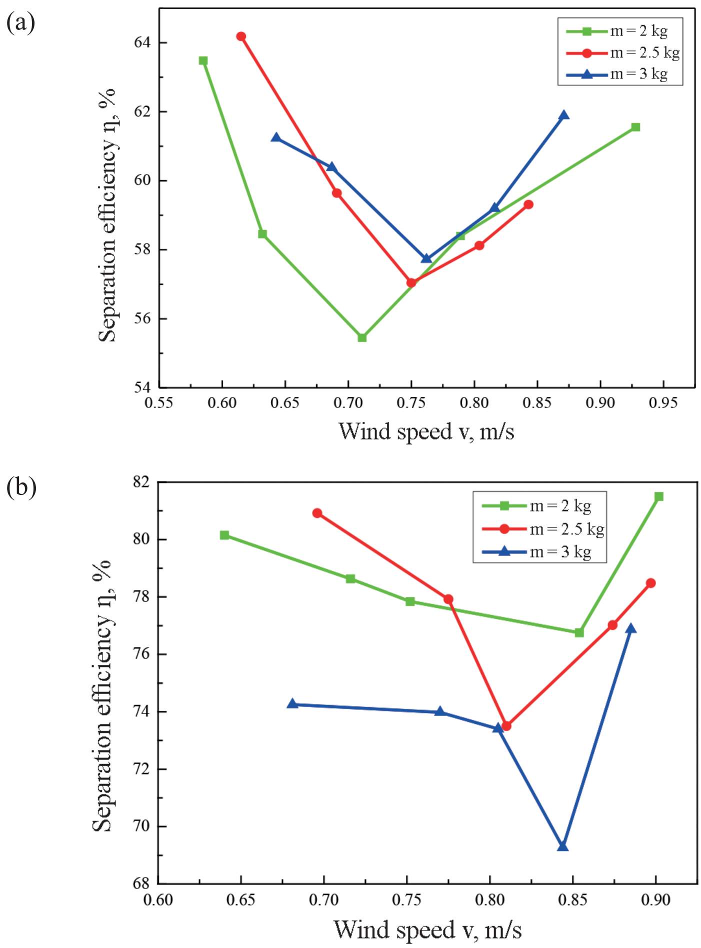


Fig. 2. The relationship between the efficiency and wind speed of separation of material samples with different masses: (a) oil shale and (b) semi-coke.

The broken line in Figure 2a indicates that under the condition that the inlet mass of oil shale remains unchanged, with the gradual increase of wind speed, the separation efficiency first decreases and then increases. When the experimental material mass is 2 kg and the wind speed is about 0.711 m/s, the optimum separation efficiency is 55.45%. Under these conditions, the separation effect is the highest and the amount of the light material separated from the heavy material is the largest. When the wind speed exceeds 0.711 m/s, the separation efficiency increases. This is because oil shale is of flaky structure and difficult to fluidize. When the wind speed exceeds the optimum value, the flaky oil shale will hinder the fluidization process. Eventually, even if the wind speed is high, lump flaky oil shale will prevent fine oil shale from separating. With the increase of separation efficiency, a lot of light material will remain in the heavy material, which makes the experimental results unreliable. With the material mass of 2.5 kg, the optimum separation efficiency is 57.04% and the wind speed is 0.750 m/s. When the material mass is 3 kg, the optimum separation efficiency is 57.72%, and the wind speed is 0.762 m/s. The analysis of experimental results shows that the separation efficiency is dependent only on the fluidization wind speed and not on the feed mass. When the optimum fluidization wind speed is reached, the separation efficiency is optimal. The optimum separation wind speed is about 0.741 m/s, and the optimum separation efficiency is about 56.74%.

The broken line in Figure 2b shows that the separation efficiency of semi-coke first decreases and then increases when the feed mass remains unchanged. The reason behind this is that semi-coke is flaky after crushing, and the excessive wind speed leads to its incomplete fluidization. Lump semi-coke hinders the fluidization of fine semi-coke, resulting in poor separation effect. When the experimental material mass is 2 kg, the wind speed reaches 0.854 m/s and the optimum separation efficiency is 76.75%. With the experimental mass of 2.5 kg, the wind speed reaches 0.810 m/s and the optimum separation efficiency is 73.50%. When the feed mass is 3 kg, the wind speed reaches 0.844 m/s and the optimum separation efficiency is 69.27%. It can be seen that within the allowable range of error, the efficiency of semi-coke separation is optimum. Regardless of the experimental material mass amount, when the wind speed reaches about 0.836 m/s, the optimum separation rate is about 73.17%.

3.2. Ash content analysis

3.2.1. Analysis of ash content in oil shale

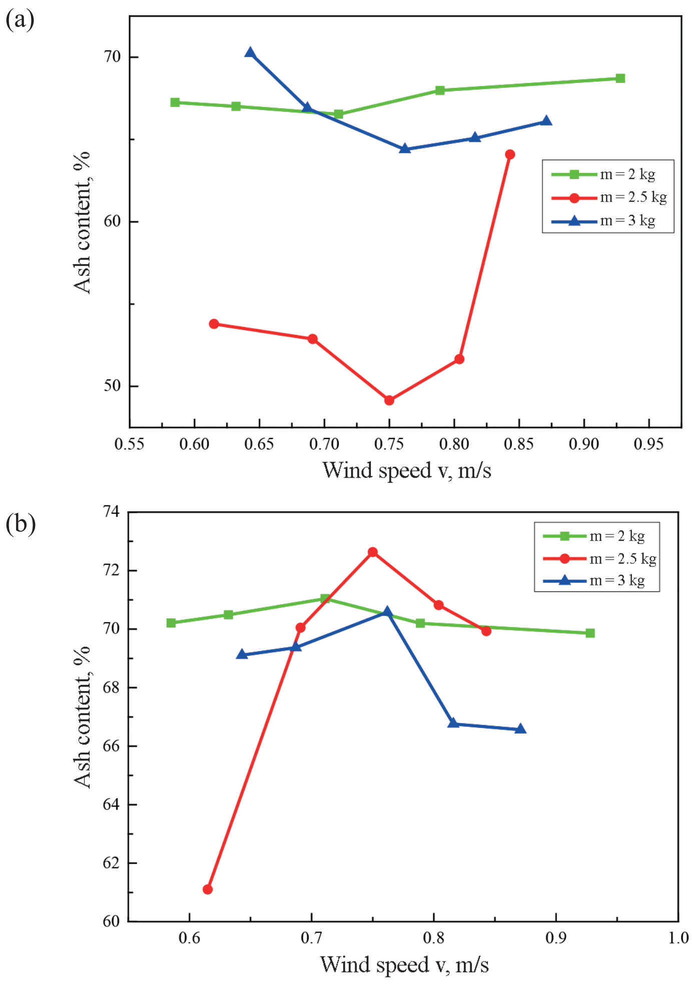


Fig. 3. The relationship between the ash content in and separation wind speed of oil shale samples with different masses: (a) light samples; (b) heavy samples.

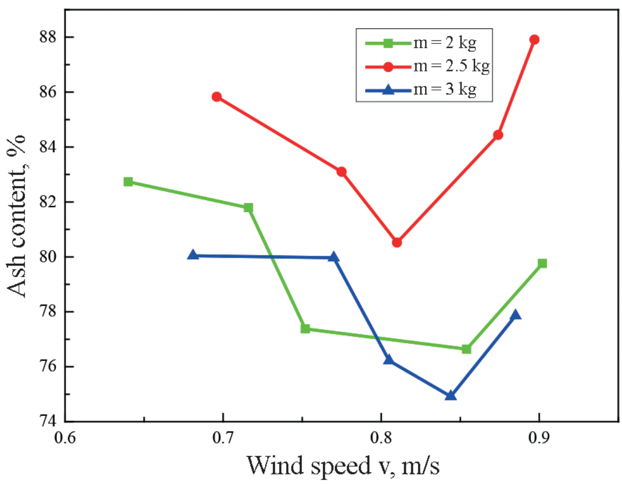
As shown in Figure 3a, when the feed mass remains unchanged, the ash content in the light sample first decreases and then increases with the wind speed. When the experimental material mass is 2 kg, the wind speed reaches 0.711 m/s and the ash content of the sample is 66.53%, while with the material mass of 2.5 kg, the respective figures are 0.750 m/s and 49.14%. The wind speed reaches 0.762 m/s and the sample ash content is 64.39% with the experimental material mass of 3 kg. The average ash content is 60.02%. After the optimum wind speed is exceeded, lump oil shale will be separated due to the high wind speed, and the separation of light samples will be hindered by lump shale and they remain in the heavy sample. Because of their high density, inorganic minerals are difficult to crush, so the ash content in lump oil shale is high. As a result, the separation effect weakens and the ash content in light samples increases gradually.

As seen from Figure 3b, when the feed mass remains unchanged, the ash content in the heavy sample first increases and then decreases. When the experimental material mass is 2 kg, the wind speed is 0.711 m/s and the ash content is 71.04%. When the material mass is 2.5 kg, the wind speed is 0.750 m/s and the ash content is 72.63%. With the experimental sample mass of 3 kg, the wind speed is 0.762 m/s and the ash content is 70.58%. The average ash content is 71.42%. When the optimal separation rate is reached, the inorganic minerals content in the heavy sample is the highest. After the optimum wind speed is exceeded, part of lump oil shale is blown into the light material, and some portion of it floats to the upper part of the fluidized bed. As a result, the ash content in the heavy sample gradually decreases and the ash content in the light sample gradually increases.

The ash content of light oil shale samples determined in the three experiments remained constant within the tolerances allowed, and this was also the case with heavy samples. Therefore, the feed mass of the experiment does not affect the change in ash content, which proves that the experimental results are widely applicable.

3.2.2. Analysis of ash content in semi-coke

(a)



(b)

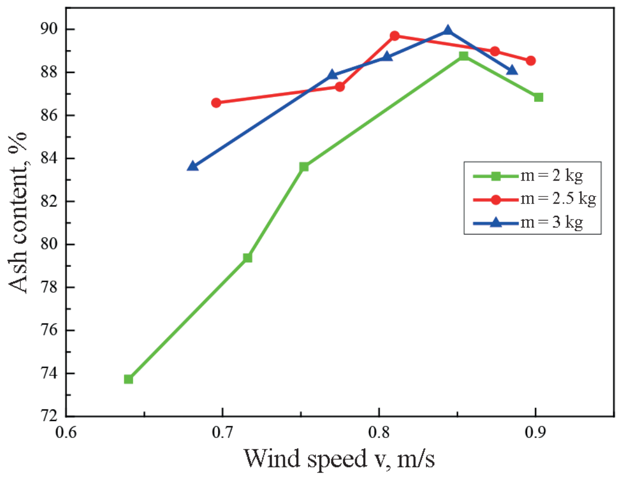


Fig. 4. The relationship between the ash content in and separation wind speed of semi-coke samples with different masses: (a) light samples; (b) heavy samples.

It can be seen from Figure 4a that with the same feed mass, with gradually increasing wind speed, the ash content of the light semi-coke sample first decreases and then increases. When the experimental sample mass is 2 kg, the wind speed reaches 0.854 m/s and the ash content is 76.64%. With the experimental sample mass of 2.5 kg, the wind speed reaches 0.810 m/s and the ash content is 80.52%. When the experimental sample mass is 3 kg, the wind speed reaches 0.844 m/s, at which time the ash content is 74.92%. The average ash content is 77.36%. When the optimum wind speed is exceeded, the separation effect weakens, the portion of lump flaky semi-coke in the light sample increases and the density of sample inorganic minerals is higher than that of organic matter, so the ash content in lump semi-coke is higher.

Figure 4b reveals that when the same feed mass is used, the ash content in the heavy samples increases as the wind speed first increases and then decreases. When the experimental sample mass is 2 kg, the wind speed reaches 0.854 m/s and the ash content is 88.76%. With the experimental sample mass of 2.5 kg, the wind speed reaches 0.810 m/s and the ash content is 89.70%. When the experimental sample mass is 3 kg, the wind speed reaches 0.844 m/s, at which time the ash content is 89.92%. The average ash content is 89.46%. After the optimum wind speed is exceeded as the wind speed increases, a large piece of semi-coke in the upper part of the fluidized bed is blown into the light material. A large piece of semi-coke in the middle of the fluidized bed blocks the separation of its small pieces, eventually causing most of the inorganic minerals in the heavy sample to be blown and the ash content is reduced.

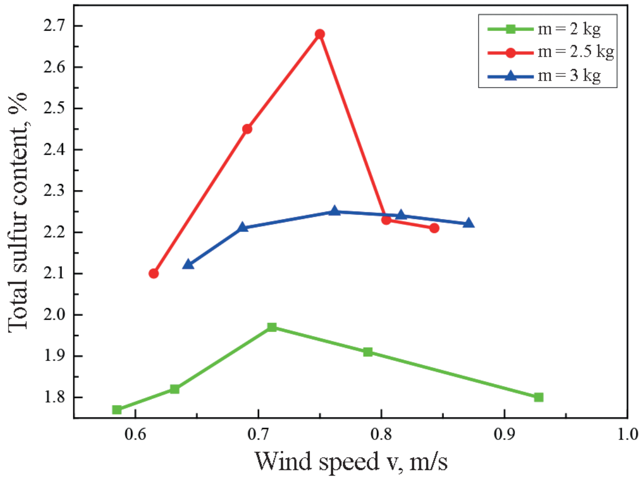
Figures 4a and 4b indicate that the semi-coke separation experiment results are not influenced by the size of the feed mass used, so these experimental results are universal and allow semi-coke to be effectively demineralized.

3.3. Analysis of total sulfur content

3.3.1. Analysis of total sulfur content in oil shale

The sulfur in oil shale and semi-coke is mainly composed of organic sulfur and inorganic sulfur. Inorganic sulfur mostly comes from pyrite and its content is relatively low, while organic sulfur of higher content originates from organic matter. Therefore, in the experiments the total sulfur is determined, and since organic matter and organic sulfur coexist, it is possible to establish whether organic matter in oil shale is separated.

(a)



(b)

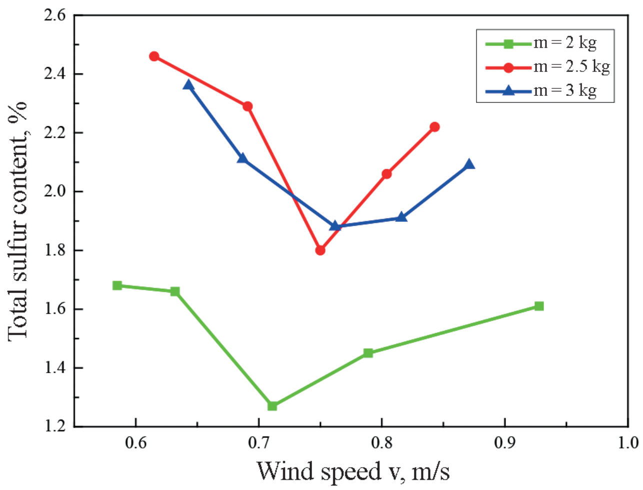


Fig. 5. The relationship between the total sulfur content in and separation wind speed of oil shale samples with different masses: (a) light samples; (b) heavy samples.

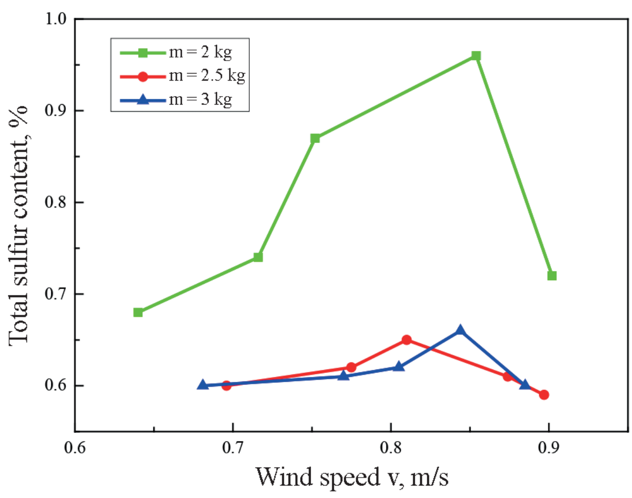
Figure 5a shows that with the increase of wind speed, the total sulfur content in light oil shale samples first increases and then decreases. When the experimental sample mass is 2 kg, the wind speed reaches 0.711 m/s and the total sulfur content is 1.97%. When the experimental sample mass is 2.5 kg, the wind speed reaches 0.750 m/s and the total sulfur content is 2.68%. With the experimental sample mass of 3 kg, the wind speed reaches 0.762 m/s, at which time the total sulfur content is 2.25%. The average total sulfur content is 2.3%. Since organic sulfur and organic minerals coexist, it is proved that when the optimum wind speed is reached, the organic matter content of the light sample is the highest.

Figure 5b displays that the total sulfur content in the heavy sample first decreases and then increases as the wind speed increases. When the experimental sample mass is 2 kg, the wind speed reaches 0.711 m/s and the total sulfur content is 1.27%. With the experimental sample mass of 2.5 kg, the wind speed reaches 0.750 m/s and the total sulfur content is 1.8%. When the experimental sample mass is 3 kg, the wind speed reaches 0.762 m/s, at which time the total sulfur content is 1.88%. The average total sulfur content is 1.65%. This is because the organic sulfur content in oil shale is the highest. When the optimum wind speed is reached, the organic matter content of the heavy sample is the lowest, so the total sulfur content of the heavy sample is the lowest.

Within the allowable range of error, the total sulfur content of the light sample determined in the three experiments remains unchanged, and similar is the case with the heavy sample. Therefore it can be concluded that the sample mass used in the experiment will not affect the experimental results, and these are universal.

3.3.2. Analysis of total sulfur content in semi-coke

(a)



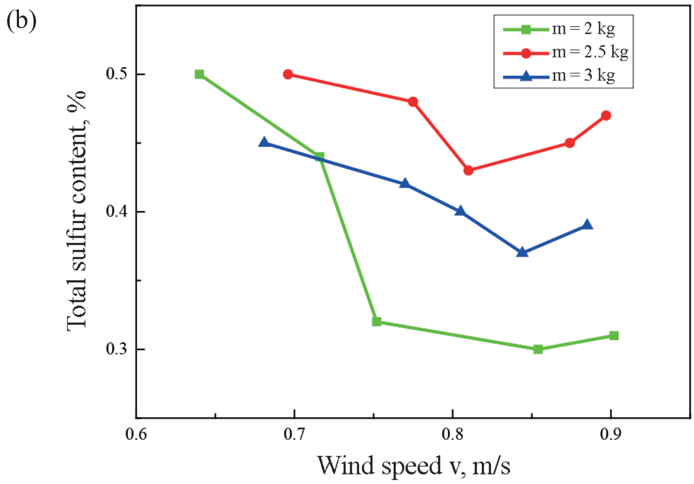


Fig. 6. The relationship between the total sulfur content in and separation wind speed of semi-coke samples with different masses: (a) light samples; (b) heavy samples.

It can be seen from Figure 6a that with the same feed mass, the total sulfur content in the light sample first increases and then decreases with increasing wind speed. When the experimental sample mass is 2 kg, the wind speed reaches 0.854 m/s, at which time the total sulfur content is 0.96%. With the experimental sample mass of 2.5 kg, the wind speed reaches 0.810 m/s and the total sulfur content is 0.65%. When the experimental sample mass is 3 kg, the wind speed reaches 0.844 m/s, at which time the total sulfur content is 0.66%. The average total sulfur content is 0.76%. When the separation efficiency is the highest, the light and heavy samples are separated to the maximum extent, while the total sulfur content in the light sample reaches the maximum. Therefore it can be concluded that the organic matter content in the light sample is also the highest. After exceeding the optimal wind speed, the fluidization effect becomes weaker and the organic matter content in the light sample decreases.

Figure 6b shows that the total sulfur content in heavy samples first decreases and then increases with the increase of wind speed under the same feed mass condition. When the experimental sample mass is 2 kg, the wind speed reaches 0.854 m/s, at which time the total sulfur content is 0.3%. With the experimental sample mass of 2.5 kg, the wind speed reaches 0.810 m/s and the total sulfur content is 0.43%. When the experimental sample mass is 3 kg, the wind speed reaches 0.844 m/s, at which time the total sulfur content is 0.37%. The average total sulfur content is 0.37%. From the above it is clear that when the separation efficiency reaches the optimum value, the organic matter content in the semi-coke heavy samples is the lowest, and the fluidization effect weakens when the wind speed exceeds 0.836 m/s. According to the experimental data analysis, within the allowable range of error, the total sulfur content of the light sample obtained from the three experiments remains unchanged, and the heavy sample also follows this model. The results of the semi-coke separation experiment are not dependent on the size of the feed mass used.

4. Conclusions

Analysis of the experimental results shows that the separation efficiency is only related to the change of wind speed. The light sample ash content and total sulfur content remain unchanged within the allowable range of error, and the heavy sample follows this pattern. Therefore, the quality of the experiment will not have any effect on the experimental results.

The results of experiments demonstrate that when the optimum wind speed is reached, the separation effect is the highest, and organic matter can be separated from inorganic matter to the greatest extent. For oil shale, the optimum wind speed is 0.741 m/s and the optimum separation efficiency is 56.74%. For semi-coke, the optimum wind speed is 0.836 m/s and the optimum separation efficiency is 73.17%.

In the case of oil shale separation, when the optimum separation efficiency is reached, the ash content of light samples is 60.02% and that of heavy samples is 71.42%, whereas the total sulfur content of light samples is 2.3% and that of heavy samples is 1.65%. In the case of semi-coke separation, when the optimum separation efficiency is reached, the ash content of light samples is 77.36% and that of heavy samples is 89.46%, whereas the total sulfur content of light samples is 0.76% and that of heavy samples is 0.37%.

If it comes to total sulfur content, further research should focus on the removal of sulfur from light samples, but also on the removal of ash from heavy samples during separation of oil shale and semi-coke.

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