

Received June 04, 2020; reviewed; accepted October 13, 2020

The influence of micro-morphology and micro-structure on fly ash triboelectrostatic beneficiation

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Abstract: Fly ash is a complex system with a variety of fine particles. The complex relationship between unburned carbon and ash particles has an important influence on the efficiency of fly ash triboelectrostatic beneficiation. The particles adhered to the two electrode plates are collected through the triboelectrostatic beneficiation experiment. The scanning electron microscopy and X-ray fluorescence are used to detect the microscopic differences between the particles of positive and negative plates. The results show that the flaky carbon particles in the raw ash and the ash particles larger than 4 μ m are more easily separated, while it is converse for the ash particles with particle size less than 4 μ m. With the particle size less than 4 μ m, it is gradually more obvious for the influence of adhesion caused by the roughness surface of spherical unburned carbon particles, and the surface pores structure of porous carbon particles. The binding structure between unburned carbon and ash particles is complex and changeable. It is not beneficial to improve the separation efficiency. Therefore, the micro-structure and micro-morphology have an important effect on fly ash triboelectrostatic beneficiation. Some suggestions were proposed from the microscopic point to improve the efficiency of fly ash triboelectrostatic beneficiation.

Keywords: fly ash, triboelectrostatic beneficiation, micro-structure, micro-morphology, separation efficiency

1. Introduction

Coal is an important non-renewable energy source and plays an important role in the fields of power generation and steel manufacturing (Yao et al., 2015). China is a large coal-producing country and a large coal-consuming country. As of 2019, there are already more than 300 thermal power plants in China (Yao et al., 2014; Han et al., 2018). Every ton of coal burned will produce 250 ~ 300kg of fly ash. The world will produce about 800 million tons of fly ash every year (Huang et al., 2003). Fly ash has become one of the largest sources of industrial solid waste, which seriously threatens the ecological environment and causes a series of problems such as water pollution, air pollution, land pollution and health effects (Li et al., 2015; Gao et al., 2012; Daniel et al., 2009).

As a useful resource, fly ash is widely used in construction engineering, road construction, coal mine backfilling and soil improvement, and can be made into various raw materials (Li et al., 2019; Ahmaruzzaman, 2010). It is very important to carry out resource utilization of fly ash and remove unburned carbon. The triboelectrostatic beneficiation process can recover a large amount of renewable energy, improve the quality of fly ash products, and meet the technical requirements of industrial fly ash materials (Iyer et al., 2001). Triboelectric separation has the advantages of simple process, low cost and no secondary pollution (LI et al., 2013).

Some research work has been carried out on the triboelectrostatic beneficiation of fly ash. Ban et al. (1997) studied the triboelectrostatic beneficiation of fly ash by using particle size analysis and carbon

content characterization. The results showed that triboelectrostatic beneficiation is suitable for separating unburned carbon from fly ash. Kim et al. (2000) evaluated the separation efficiency and charge density of fly ash at different air flow rates, air relative humidity and temperature. Relative humidity and particle size have a greater impact on the charge density and separation efficiency of fly ash particles. Zhang et al. (2018) studied the effect of rotating frictional electrostatic separator on the decarbonization efficiency of fly ash under different electrode voltages by comparing experiment with simulation. The numerical simulation results are consistent with the experimental results, which indicates the effectiveness of the simulation in predicting fly ash separation. Kim et al. (2001) studied the effects of air flow, feed speed and electric field strength on charge density and decarbonization efficiency using aerodynamic friction electrostatic beneficiation system. Zhang et al. (2018) recovered unburned carbon using a rotary friction electrostatic separator, and studied the influence of structural parameters and operating parameters on the recovery efficiency of unburned carbon. Cangialosi et al. (2008) studied the process of air friction electrostatic separation of unburned carbon. The results showed that the separation efficiency of fly ash decreased with the increase of particle concentration, and the fly ash with high fever loss was greatly affected by the particle concentration. Cangialosia et al. (2008) separated fly ash with high carbon content using beneficiation technology. By comparing the decarbonization efficiency of fly ash before and after drying, it was found that the fly ash exposed to the air for a long time had high humidity, which seriously reduced the decarbonization efficiency of fly ash. Baltrus et al. (2002) used buffer solutions with different PH values to treat fly ash and found that alkaline conditions can inhibit charge reversal under wet conditions. Adding calcium, sodium and borate ions to fly ash can effectively prevent the charge reversal and improve the separation efficiency of fly ash. Menghua et al. (2003) separated fly ash particles size less than 74 μm , and pointed out that the decarbonization efficiency of fly ash could be improved by extending the plate length or increasing the electric field strength. Haisheng et al. (2016) found that microwave heating caused changes in moisture content and dielectric constant of fly ash, which could effectively improve the separation efficiency of wet fly ash.

It can be seen that the above research work focused on triboelectrostatic beneficiation equipment, process operating parameters and external environmental interference factors. The feasible technical scheme of high efficiency charge of fly ash was explored from the aspects of triboelectrostatic material, structure and charged dielectric. By means of experimental research, the reasonable operating parameters are determined and the interference factors of the external environment are overcome by using electric heating and microwave heating. All of these have good guiding significance to the engineering practice of triboelectrostatic beneficiation of fly ash.

However, the size of most fly ash particles is less than 74 μm , more abundant information can be obtained by studying from the microscopic perspective. Xing et al. (2019) analyzed the three forms of carbon and ash in fly ash by scanning electron microscopy and pointed out the existence position and shape of carbon. The results showed that triboelectrostatic beneficiation is suitable for the separation of monomer carbon, and the surface attached carbon particles can be flotation. He et al. (2017) compared raw coal before and after crushing by scanning electron microscope and X-ray energy spectrum. According to the polarity of the components and the ratio of charge to mass, the feasibility of separation of raw coal can be judged. He et al. (2020) analyzed the elements of minerals using X-ray diffraction and X-ray fluorescence technology and studied the feasibility of electrical separation of kaolin, montmorillonite and quartz. Li et al. (2006) analyzed the element composition of fly ash by X-ray diffraction and scanning electron microscopy. The composition of fly ash particles was obtained, including unburned carbon, magnetic bead, calcium bead and Si-Al glass microbead. Brown et al. (2011) found that there is a big difference between the particle size and element composition of fly ash by ICP-MS, FE-SEM, XRD and laser diffraction technique. The micro-morphology of fly ash treated with hydrofluoric acid was observed.

There are differences among fly ash particles in appearance morphology and structure composition. Carbon particles and ash particles are not all spherical. The size is also different. At the same time, due to the pure natural structure of raw coal, the carbon particles and ash particles are not independent. There are natural adhesion and connection structures. These factors are closely linked to raw coal production area, combustion conditions and processing process. Therefore, it is meaningful to explore

the influence of micro-morphology and micro-structure on the triboelectrostatic beneficiation of fly ash. First of all, the fly ash composition is complex and changeable, and there are complex relationships between the particles of different elements. It is necessary to reveal the influence of these complex relationships on the triboelectrostatic beneficiation from the microscopic perspective. Secondly, the study on the separation of charge and electric field of fly ash only focuses on the particle group, and it is incapable to study single-particle and consider the differences among particles. The influence of single-particle behavior on triboelectrostatic beneficiation was neglected. According to the difference of micro-morphology and micro-structure of fly ash samples on positive and negative electrode plates, technical guidance can be provided for improving the decarbonization efficiency.

2. Materials and methods

2.1. Material

2.1.1. Size distribution

The particle size of the fly ash was analyzed by using JL-1197 laser particle distribution tester. The results of particle size analysis are shown in Fig. 1. It can be seen from Fig. 1 that the particle size distribution of fly ash is roughly between 3 μm and 350 μm . The median diameter of particles is 48.23 μm , and particles with particle size less than 10 μm account for about 2% of the total. Particles with diameters between 10 μm and 30 μm account for about 18% of the total. In the range of 30-100 μm , particles accounted for 64% of the total particles. Particles larger than 100 μm make up about 16 percent of the total. On the whole, the particle size distribution of the sample particles tends to be between 30 μm and 100 μm .

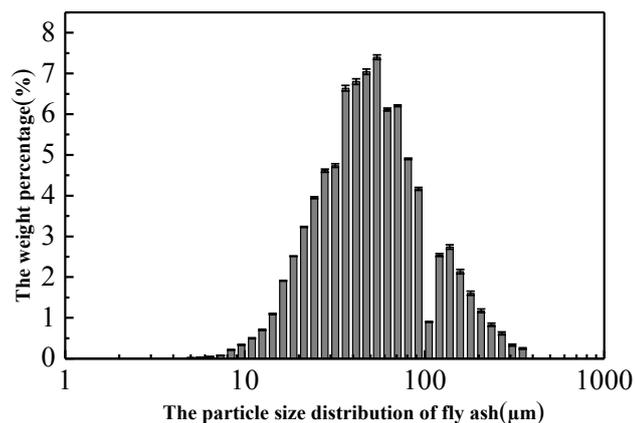


Fig.1. The particle size distribution of fly ash

2.1.2. Fly ash composition

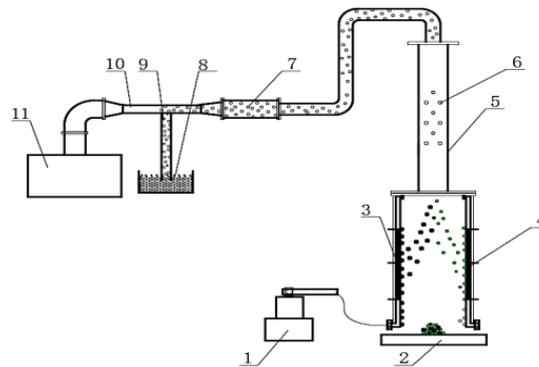
The fly ash samples used in the experiment came from a coal-fired power plant in Jungar Banner, Inner Mongolia, China. The experimental samples were screened to obtain fly ash samples with particle size less than 74 μm . Five samples were selected and mixed, totaling 500 grams. In order to better understand the composition of the fly ash, a sample of 10g fly ash was taken and dried at 105°C for 24 hours. In Table 1., the oxide composition of the fly ash sample is SiO_2 , Al_2O_3 and a small amount of Fe_2O_3 , CaO , MgO , Na_2O , K_2O , TiO_2 , etc. The total results of X-ray fluorescence spectrum analysis of the sample are shown the content of SiO_2 and Al_2O_3 is more than 80%. The content of CaO is less than 10%. Ten groups of samples were measured and the average loss-on-ignition was 5.12%. The loss-on-ignition of fly ash varies between 5.12%±0.06%.

Table 1. The main chemical constituents of fly ash

Chemical constituents	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	SO_3	Other oxides	Loss-on ignition
Content (%)	39.10	46.67	2.42	2.54	0.26	0.07	0.37	1.70	0.19	1.56	5.12

2.2. The experimental system

The triboelectrostatic beneficiation experimental system of fly ash is shown in Fig. 2. Weigh 100g of fly ash and place them in a container. After the centrifugal fan is started, the gas velocity in the pipe increases gradually at the nozzle due to the reduction of the cross-sectional area of the pipe, thus forming the negative pressure adsorption. Therefore, the fly ash sample enters the nozzle through the negative pressure pipeline, is fully mixed in the gas-solid mixing pipeline, and then enters the friction charge apparatus. The collision between the fly ash particles and the friction bar in the triboelectrostatic beneficiation apparatus makes the ash particles positively charged and the carbon particles negatively charged. Under the action of airflow, charged particles enter a 5kV high-voltage electric field. Under the action of electric field force and gravity, ash particles are absorbed by the negative plate, while carbon particles are absorbed by the positive plate. Some neutral particles or particles with small electric charge leave the electric field and become intermediate products. The separation and collection of carbon ash are realized.



1. High voltage power, 2. Intermediate product, 3. Positive plate, 4. Negative plate, 5. Friction charge electrical appliance, 6. Friction rod, 7. Gas-solid mixing pipeline, 8. Container, 9. Negative pressure pipeline, 10. Nozzle, 11. Centrifugal fan

Fig. 2. The experimental system diagram of triboelectrostatic beneficiation

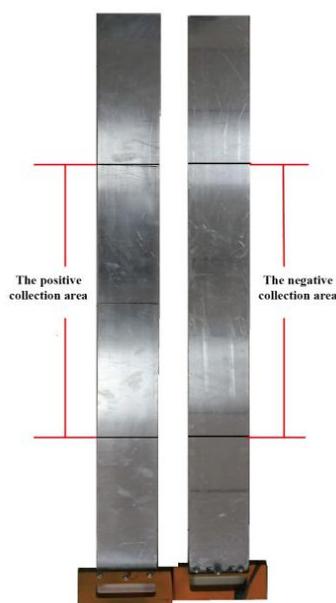
2.3. Experimental methods

The triboelectrostatic beneficiation experiment of fly ash was carried out under the condition of wind speed of 100m³/h and voltage of 5KV. The lengths of the two electrode plates are both 1.4m and are divided into three regions, with the lengths of 0.3m, 0.8m and 0.4m respectively, as shown in Fig. 3. The length of 0.3m region is close to the entrance of the electric field, which is greatly affected by the action of high-speed airflow. The length of the 0.4m region is located at the lowest end of the electric field and is likely to be contaminated by neutral particles. The electrode plates are installed in the groove on the wall of the electric field to ensure that it can be inserted or removed before and after the experiment. At the end of each experiment, the electrode plates were removed to collect samples from an intermediate length of 0.8m. To guarantee the accuracy of the experimental results, five experiments were carried out. The fly ash samples collected from the electrode plate were mixed, and the raw ash samples and positive and negative plate samples were detected by SY/ T 5162-2014 scanning electron microscopy. Adobe Photoshop CS6 and Adobe Illustrator CS6 were used to process the SEM photographs, and the particle size was measured according to the scale on the scanning electron microscope images. The energy spectrum quantitative analysis of fly ash was carried out by SY/T 6189-1996. The composition of the samples is detected by BRUKER S8 TIGER X - ray fluorescence spectrometer.

2.4. Experimental results

The recycling efficiency of unburned carbon (abbreviated as RC) is defined as follows:

$$RC(\%) = P \times \frac{LOI_+}{LOI_S} \quad (1)$$



where LOI_s is defined as the LOI of fly ash sample before experiment, LOI_+ describes the LOI of fly ash obtained from the positive plate, P is the productivity of positive plate.

Table 2. shows the LOI and yield of the positive and negative plate under the condition of $100\text{m}^3/\text{h}$ wind speed and voltage of 5kV. It can be seen from the Table 2. that the LOI and yield of the positive plate are 8.45% and 35.67%. The LOI and yield of the negative plate are 3.08% and 49.67%. The LOI and yield of the intermediate products are 4.19% and 14.66%. The RC of fly ash is 58.97%. The carbon particles are negatively charged and the ash particles are positively charged, the LOI of the positive plate is higher than that of negative plate. The yield of the intermediate products is much smaller than that of the positive and negative plate products, indicating that the separation effect is better.

Table 2. Product analysis

	LOI (%)	Productivity (%)	RC(%)
Positive plate products	8.45	35.67	58.97
Negative plate products	3.08	49.67	/
Intermediate products	4.19	14.66	/

3. Results and discussion

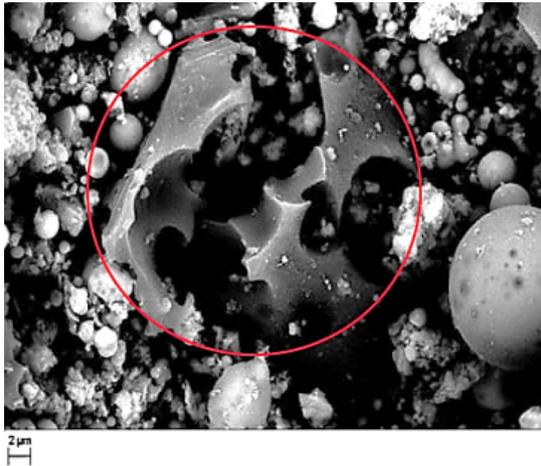
3.1. Effect of micro-morphology on triboelectrostatic of fly ash

3.1.1. Influence of the micro-morphology of carbon particles

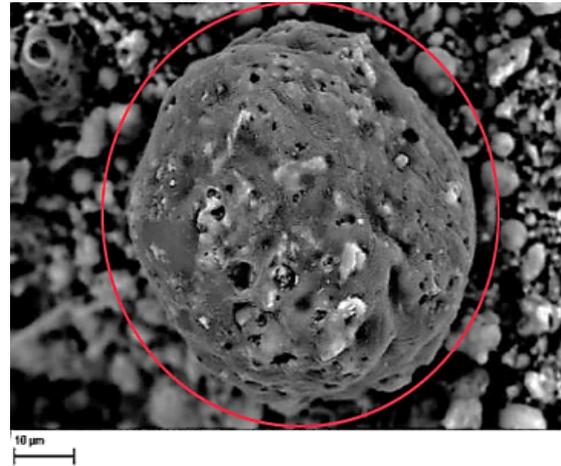
3.1.1.1. Micro-morphology of carbon particles in raw ash

The results of SEM analysis of the raw ash are shown in Fig. 4. As can be seen from the figure. The micro-morphology of carbon particles in the raw ash vary greatly. It can be roughly divided into three main morphological characteristics: flaky, spherical and porous. The size of flaky carbon particles is the smallest and the number is the largest. In Fig. 4 (a), the flaky carbon particles are small in size, irregular in appearance, smooth in surface, and have obvious boundary characteristics with ash particles. Fig. 4 (b) shows spherical carbon particles with larger size, most of which are above $50\ \mu\text{m}$ and have a regular appearance. The surface of the spherical carbon particles is relatively rough. There are micropores under $5\ \mu\text{m}$, and their surface is easy to adhere to ash particles with small particle size, so as to form carbon and ash combinations. As can be seen from Fig. 4 (c), the surface of some carbon particles is loose and

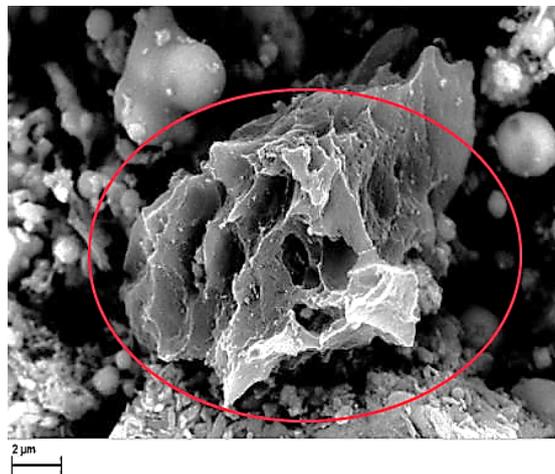
porous, with a large number of irregular pore characteristics, and the particle size is between flaky and spherical carbon particles.



(a) Flaky carbon particles



(b) Spherical carbon particles



(c) Loose porous carbon particles

Fig. 4. SEM analysis results of raw ash

3.1.1.2. Micro-morphology of carbon particles recovered from positive plate

SEM and energy spectrum analysis of positive plate particles are shown in Fig. 5. There is an obvious color difference between carbon particles and ash particles. The color of ash particles is brighter than that of carbon particles. The carbon particles recovered from the positive plate are mostly flaky. The results of energy spectrum analysis showed that the carbon content of flaky carbon particles was more than 80%. This indicates that flaky carbon particles in the raw ash are more conducive to recovery. Compared with other two kinds of carbon particles, the collision probability of flaky carbon particles is higher due to the large number of particles. However, the particle size is small, the specific surface area is large, and the collision contact area is large. All these improve the charge efficiency of particles. Therefore, the flaky carbon particles are more favorable to be removed in the separation process. For spherical and loose porous carbon particles, larger particle size can not only reduce the charge-mass ratio, but also enhance the gravity effect in the separation process. Therefore, the decrease of the charge-mass ratio leads to the decrease of the electric field force, and it is difficult to realize efficient separation in the high-voltage electrostatic field under the action of stronger gravity. Although the specific surface area of the porous structure particles is large, the area that can really participate in the collision electrification is small, and the pore wall cannot play a role in the electrification process.

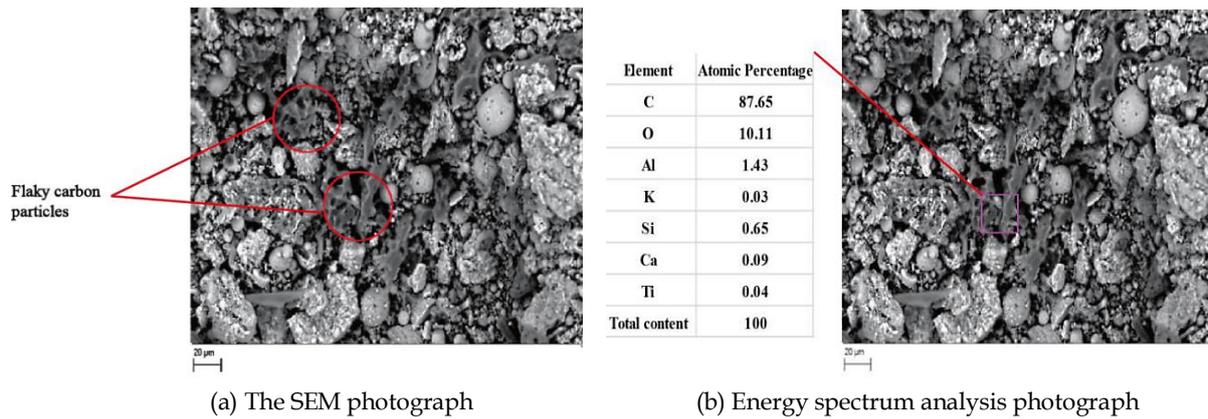


Fig. 5. SEM results and energy spectrum analysis of positive plate particles

3.1.2 The influence of the microscale of ash particles

3.1.2.1. The scale of ash particles in raw ash

Fig. 6 shows the SEM detection and analysis results of raw ash particles. It can be found from the figure that the particle size distribution of the ash particles is not uniform. In the figure, the particle size of most ash particles is less than 4 μm . There are a small number of large particles with particle size between 9 and 15 μm and medium particles with particle size between 4 and 9 μm , which are marked by red circles in the Fig. 6.

3.1.2.2. The scale of ash particles recovered from the plate

Fig. 7 shows the SEM image of two samples of negative plate particles. Particles with particle sizes of 9-15 μm and 4-9 μm have been marked in the figure. In Fig. 7 (a), there are 5 large particles with the particle size of 9-15 μm , and the number of ash particles with the particle size of 4-9 μm is greater than 10. Therefore, it can be seen that the number of particles with a particle size of more than 4 μm in the negative plate increases significantly compared with the raw ash, indicating that ash particles with a larger particle size are easier to be separated.

Fig. 7 (c) shows that ash particles with particle size of 4-15 μm , the surface finish is relatively high, and the collision probability is high in the sorting process, which makes it easier to charge. According to Greason's frictional charge theory:

$$q(t) = q_s(1 - \exp(-at)) \quad (3)$$

In the formula, $q(t)$ is the charge of the particle at time t (C), q_s is the saturated charge (C), t is the time (s) and α is the time constant of charge generation.

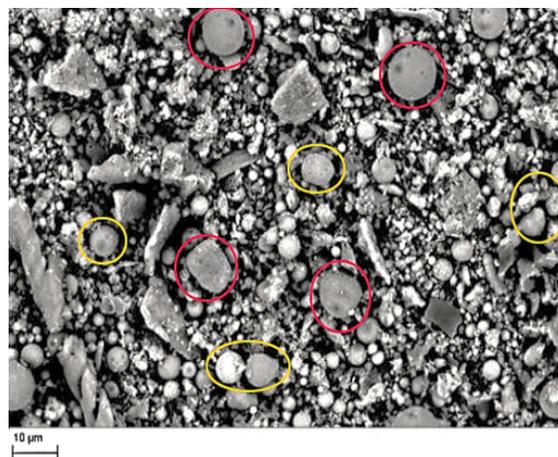


Fig. 6. SEM analysis of raw ash particles

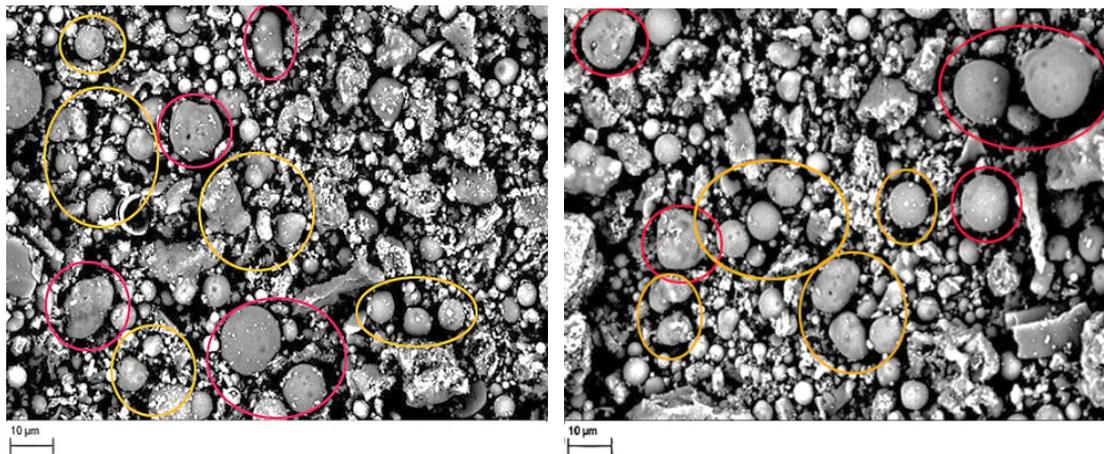
With the increase of ash particle size, both the saturated charge and the positive charge will increase, which is more conducive to separation.

3.2. Influence of micro-structure on decarbonization of fly ash

3.2.1. Influence of porous structure

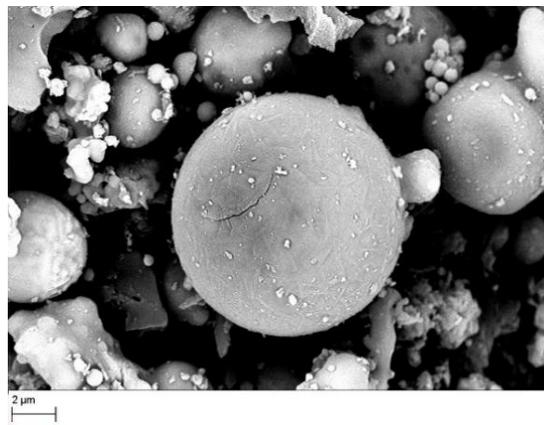
Combined with the analysis in Fig. 4, it can be seen that there are carbon particles with loose porous structure in the raw ash. In order to understand the influence of the porous structure of carbon particles on the separation of fly ash, SEM detection and energy spectrum analysis were conducted on the positive plate products, and the results are shown in Fig. 8(a) and Fig. 8(b). It can be seen from the figure that the carbon content of the ring structure is 81.42%, which proves to be carbon particles. There is a large number of pore structure on the surface of this carbon particle, and the pore size is 4-6 μm .

Energy spectrum analysis showed that the oxygen content of the particles dropped in the pores was 58.27%, the Al content was 8.61%, the Si content was 8.80%, and the oxide content was relatively high, so it could be determined that these particles with small particle size were ash particles. Due to the small particle size of ash particles under 4 μm , porous structure with pore diameter less than 4 μm exists on the surface of carbon particles in the raw ash. Therefore, ash particles under 4 μm are very easy to fall into the pores on the surface of carbon particles and be absorbed by the positive plate. In addition, some ash particles have small charge and strong airflow force, so they fail to be absorbed by the negative plate and become intermediate products. Therefore, the ash particles in the surface pores of the carbon particles will be absorbed by the positive plate together with the carbon particles, resulting in a decrease in the loss-on-ignition of the positive plate and affecting the decarbonization efficiency of fly ash.



(a) The first sample

(b) The second sample



(b) The SEM photograph of ash particles

Fig. 7. SEM analysis results of negative plate ash products

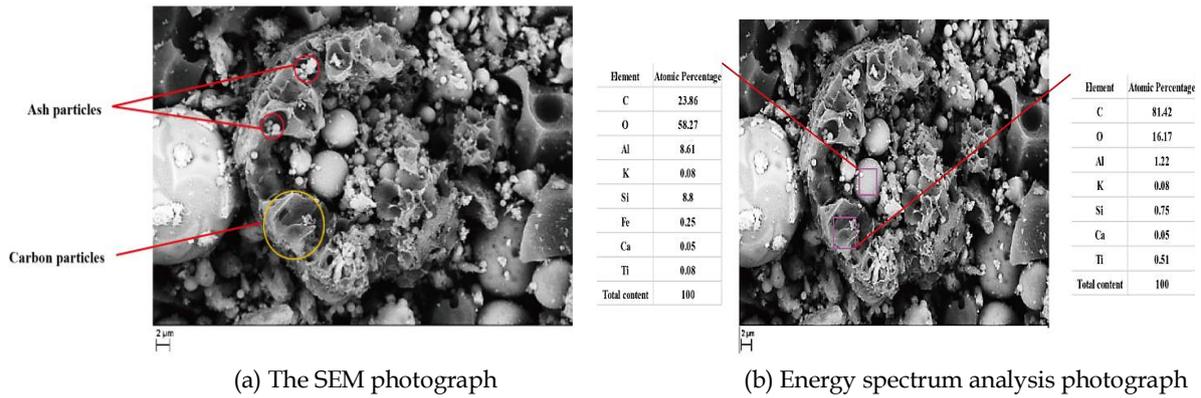


Fig. 8. Porous carbon structure of positive plate

3.2.2. Influence of carbon ash binding structure

3.2.2.1. Carbon ash binding structure of positive plate products

SEM test results of the positive plate products show that there is carbon ash binding structure, as shown in Fig. 9(a), and the energy spectrum analysis of the combination is shown in Fig. 9(b). The gray-black globular particles in Fig. 9(a), whose carbon content is 85.87%, are carbon particles. In Fig. 9(a), the oxygen content of gray particles on the particle surface is 55.72%, the content of Al is 10.23%, and the content of Si is 9.95%. In the process of separation, the carbon particles are negatively charged and the ash particles are positively charged. Due to the large size of the carbon particles, the ash particles stick to a part of the carbon particles. A part of the positive charge of the ash particles is neutralized by the negative charge of the carbon particles, so that the carbon ash combination is negatively charged. During the separation process, it is absorbed by the positive plate, resulting in the reduction of the positive plate decarbonization rate.

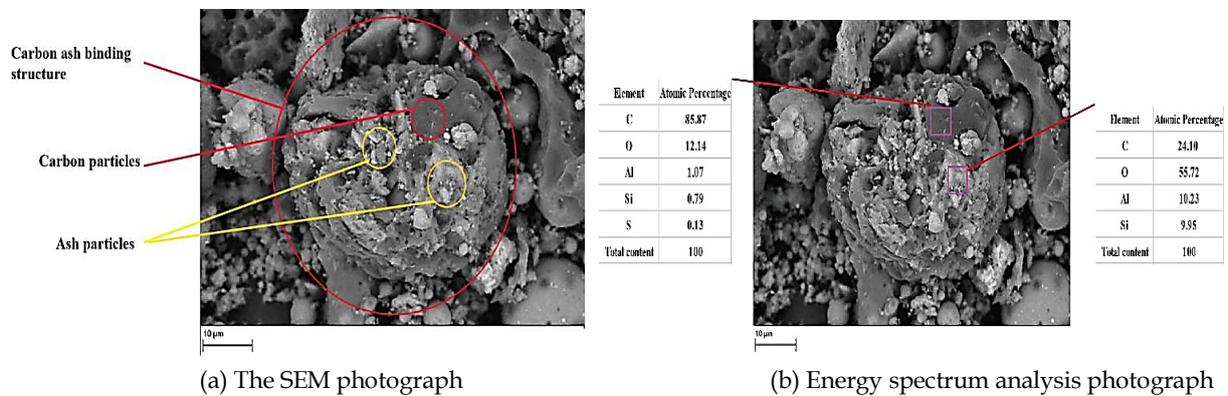


Fig. 9. Carbon ash combination in positive plate products

3.2.2.2. Carbon ash binding structure of negative plate products

The carbon ash binding structure was also found in the negative plate products, as shown in Fig. 10. The carbon particles are coated with ash particles, and only a small part of the carbon is exposed. Fig. 10(a) shows the results of energy spectrum analysis. The C content in this part is 81.42%, which is a carbon particle Fig. 10(b) energy spectrum analysis showed that the O content of the carbon particles was 59.59%, the Al content was 8.35%, and the Si content was 8.03%, which were ash particles. Ash positively charged particles, carbon particles negatively charged, and the surface of the carbon particles with negative charge neutralized by ash particles on the surface of the positive charge. In addition, due to the small surface area and low collision probability of the exposed carbon particles, the charged process of the carbon particles is affected. The carbon ash combination positively charged and adsorbed by the negative plate during the separation.

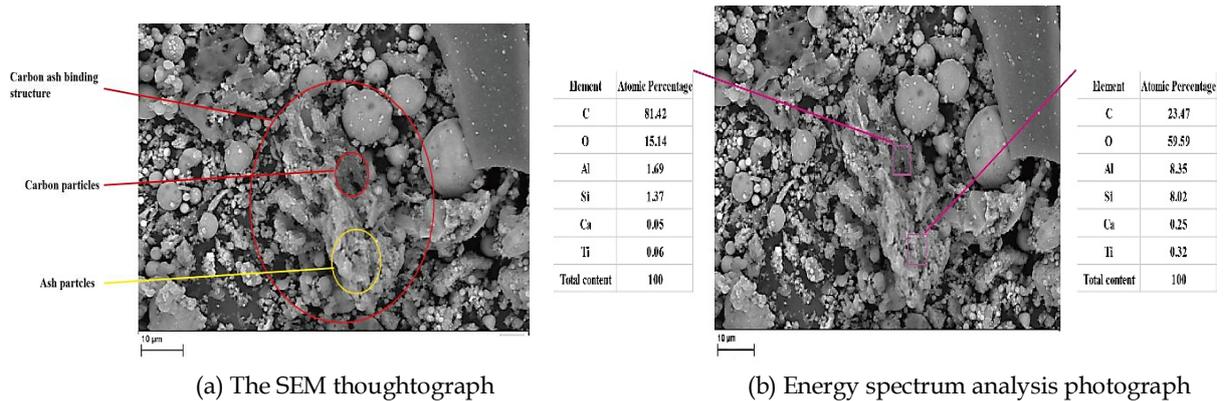


Fig. 10. Carbon ash combination in negative plate products

4. Conclusions

The LOI⁺ and LOI of fly ash is 8.45% and 3.08%, respectively. The RC of fly ash is 58.97%. The micro-structure and micro-morphology of fly ash have an important influence on the fly ash electrical separation and decarbonization efficiency.

Carbon particles in the raw ash mainly exist in three forms: flaky, spherical and porous. By comparing the three particles, the flaky carbon particles with smooth outer surface and small size will be easily removed from fly ash. Ash particles with size bigger than 4 μm will be easily separated because of smooth surface and high charging efficiency. The carbon ash binding structure has an effect on the products recovery of two electrodes plates, which reduces the decarbonization efficiency.

A lot of methods will be taken into account for improving the efficiency of fly ash triboelectrostatic separation, such as increasing the charge-mass ratio of carbon particles and destroying the combination structure between carbon and ash particles.

Acknowledgments

The financial support provided by the Fundamental Research Funds for the Central Universities (grant 2017XKQY035), the China Postdoctoral Science Foundation [grant 2014M560466], the Postdoctoral Science Foundation of Jiangsu Province (grant 1401054B) and the National Natural Science Foundation of China (grant 51674259) for this work is gratefully acknowledged.

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