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Effects of ultrasonic treatment on the particle size, shape and ash content of fine coal

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Abstract: The effects of ultrasonic treatment of different time on the particle size, morphology, ash content, and elementary composition of fine coal particles (74-45 μm) were investigated in this paper. The size reduction of coal particles induced by ultrasonic treatment was analyzed through the Focused Beam Reflectance Measurement (FBRM) and the variation of mechanical pulverization mechanism was discussed. The Scanning Electron Microscope (SEM) measurements showed that the sharp corners on coal particles was ground to round ones after ultrasonic treatment. This may be not conducive to coal flotation. It was also found that the ash content of 74-45 μm coal particles was increased gradually with the increase of ultrasonic time, while the ash content of -45 μm fraction was decreased. This was supported by the EDS measurements, indicating that the carbon content on the surfaces of 74-45 μm coal was reduced while the aluminium and silicon contents were increased after ultrasonic treatment.

Keywords: ultrasonic time, fine coal, particle size, particle shape, ash content

1. Introduction

Up to now, there are diverse treatment methods, e. g., ultrasonic treatment (Barma, 2018; Peng et al., 2018), pyrolysis (Niu et al., 2017; Xia et al., 2017), and grinding (Xia et al., 2017), that can change the size, shape, and surface properties of coal particles. Ultrasound is generally defined as a short wave with a frequency higher than 20 kHz (Bergmann, 1950). With the development of high-power ultrasonic processor, the effect of ultrasonic treatment on the mechanical pulverization of solid particles has attracted the attention of mineral processing researchers and practitioners. Toraman (2017) found that the ultrasonic pretreatment could greatly reduce the particle size of calcite. The calcite powders ($d_{50}=25.23 \mu\text{m}$) could be comminuted to finer ones ($d_{50}=12.89 \mu\text{m}$) under the optimal ultrasonic condition. Lapidés and Yariv (2004) revealed that the particle size of bentonite was reduced by sonication in the dilute suspensions, but it was increased in the concentrated suspensions. Ambedkar et al. (2011) found that the dominance of size reduction and particle breakage effects on coal was more intensive when operated at low-frequency ultrasound. They also reported that the initial sonication stage led to pitting of the coal surface followed by crack formation, then widening of cracks and finally particle breakage. Similarly, Sahinoglu and Uslu (2015) also reported that exposing the coal slurry to longer treatment time and higher power of ultrasound promoted the particle-cavitation interaction and intensified the particle-particle interaction, leading to finer particle sizes.

Besides particle size, the shape and surface properties of solid particles would also be changed by ultrasonic treatment. Franco et al. (2007) studied the effects of ultrasonic treatment on the physical property of dickite. It was found that ultrasonic treatment modified both particle size and morphology of dickite, in addition, the decrease in particle size resulted in significant increase in specific surface area. Kang and Lv (2006) found that ultrasonic treatment changed the size, shape, and surface cleanliness of slime particles as well as impel pyrite and gangue separated from coal. Cao et al. (2017) found that ultrasonication ($\geq 0.3 \text{W}/\text{cm}^2$) treatment within 40 s could result in surface cleaning and

further oxidation of pyrite of both slight and heavy oxidization degrees. However, the particle size and particle shape factors of quartz could hardly be changed by ultrasound of different powers, owing to the high hardness of quartz (Gungoren et al., 2017).

In the previous works, the effect of ultrasonic treatment on mineral particles was usually analyzed to interpret the superiority of flotation performance with ultrasonic pretreatment over conventional flotation (Haghi et al., 2016; Ozkan, 2002; Ozkan and Gungoren, 2012). Ozkan (2017) reported that a certain degree of agglomeration of the froth products and the removal of a certain amount of finely size ash-forming clays were observed during ultrasonic treatment. Cliek and Ozgen (2009) found that there was a much better cleaning action on the coarse copper-iron sulphide particles in the froth than the fine ones when ultrasonic treatment was applied. The efficient cleaning effect of ultrasound on coal particles was also found by Ozkan et al (2006). Mao et al. (2018) found that +74 μm lignite coal particles could be comminuted to be finer than 74 μm by ultrasonic treatment, and the coverage of high-ash coal fines on the +74 μm coal particle surfaces could be removed. Kang et al. (2008) found that the sulfur and ash contents of coal particles of 125–75 μm and 75–45 μm were reduced to different extents after ultrasonic conditioning, which indirectly reflected that ultrasound has comminuting and separating effects on the coal particles. In addition, coal flotation rate could be increased by using ultrasonic conditioning in comparison with the conventional conditioning (Ghadyani et al., 2017; Kopparthi et al., 2017).

The ultrasonic treatment during coal flotation positively affects the properties of floated coals (Ozkan and Kuyumcu, 2007). This is mainly because the size, shape and surface physico-chemical properties of the coal particles are changed by ultrasound (Ozkan, 2012). However, comprehensive research in the effects of ultrasonic treatment on coal properties was still scarce. Therefore, the aim of this paper was to directly investigate the effect of ultrasonic treatment of different time on the size distribution of fine coal particles (74–45 μm). More importantly, the effects of ultrasonic treatment on the shape, ash content, and elementary composition of fine coal particles were also studied which has been scarcely explored previously. This research is expected to provide guidance for ultrasonic-assisted coal flotation.

2. Materials and methods

2.1. Coal samples

The lignite sample (-0.5 mm) was obtained from a coal preparation plant located in Jungar Banner, China. The proximate analysis is listed in Table 1, where M_{ad} is the moisture content, A_{ad} the ash content, V_{ad} the volatile content, FC_{ad} the fixed carbon content on air dried basis, $G_{\text{R.I}}$ the caking index, and P_{M} is the transmittance. The macerals of lignite sample is shown in Table 2. The vitrinite content of lignite sample is 80.28%. The sample preparation flowsheet is shown in Fig. 1. The 74–45 μm coal sample was used in the ultrasonic treatment experiments and its ash content was 25.40%. Tap water was used in the ultrasonic treatment experiments. The properties of tap water are shown in Table 3.

Table 1. Proximate analysis of lignite sample (air dried)

M_{ad} (%)	A_{ad} (%)	V_{ad} (%)	FC_{ad} (%)	$G_{\text{R.I}}$	P_{M} (%)
6.32	7.70	35.36	50.62	0	91.00

Table 2. The macerals of lignite sample (%)

Vitrinite	Inertinite	Exinite	Clay class	Carbonates
80.28	18.18	0.52	0.51	0.51

Table 3. The property analysis of tap water

Conductivity ($\mu\text{s}/\text{cm}$)	PH	Ion content (mg/L)					
		Ca^{2+}	Mg^{2+}	Na^{+}	K^{+}	Cl^{-}	SO_4^{2-}
158.90	7.42	70.24	25.57	45.66	9.15	76.12	78.20

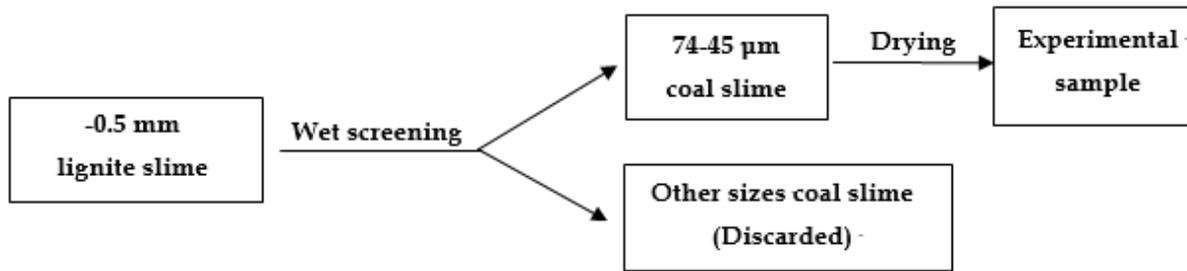


Fig. 1. The sample preparation flowsheet

2.2. Ultrasonic treatment experiment

In the ultrasonic treatment experiments, the VCX 800 ultrasonic processor (SONICS, USA) was used as shown in Fig. 2. The ultrasonic power and ultrasonic frequency were respectively kept at 800 W and 20 kHz and there was no ultrasonic pulse time. The ultrasonic amplitude was 100%, which directly represented the magnitude of ultrasonic output energy. The coal particles of 4.5 g were dissolved in a plastic beaker with 700 cm³ of water. Then, the ultrasonic treatment was implemented on the coal suspension for different time ranged from 0 to 140 min. The slurries of ultrasonic treatment tests of different time were kept for cooling down to the room temperature (28 °C) before the particle size distribution measurement and SEM/EDS measurements.



Fig. 2. Schematic of ultrasonic treatment experimental setup

2.3. In-situ particle size distribution measurement

Focused Beam Reflectance Measurement (FBRM) provides the possibility of in-situ size characterization of particles and aggregates over a wide range of solids concentrations (Peng and Williams, 1994). After ultrasonic treatment experiments, the FBRM (Particle Track G400, Mettler Toledo, Switzerland) was used to obtain the in-situ particle size distribution of coal particles after ultrasonic treatment of different time. Before measurement, the coal suspension was agitated using an impeller agitator at 500 rpm for 5 min to fully disperse the sample. Then the agitation speed was decreased to 400 rpm and the FBRM measurement commenced. The particle size distribution of the coal suspension was obtained every 2 s and FBRM measurement lasted for 30 min for each suspension. A volume weighted distribution was achieved by applying a square-weighting to the original chord length distribution.

Eqs. (1) and (2) were used to calculate the square-weighted counts in a chord channel and mean square-weighted chord length.

$$\text{The square-weighted counts} = n_i M_i^2 \quad (1)$$

$$\text{Mean square-weighted chord length } (\mu\text{m}) = \frac{\sum_{i=1}^k n_i M_i^3}{\sum_{i=1}^k n_i M_i^2} \quad (2)$$

where n_i is the count in chord channel i and M_i is the chord length at the midpoint of chord channel i .

After the FBRM measurement, the representative samples taken from coal suspensions after ultrasonic treatment of different time were classified into $-45\ \mu\text{m}$ and $74-45\ \mu\text{m}$ size fractions through wet screening. Then they were filtered, dried, weighed, and submitted to ash content determination. After the repeated measurements, the average ash content of the $-45\ \mu\text{m}$ and $74-45\ \mu\text{m}$ coal particles were calculated on dried basis, respectively. The ash content on dried basis can be calculated using Eq. (3).

$$\text{Ash content} = \frac{m_1}{m} \times 100 \quad (3)$$

where m_1 is the weight of residue after burning (g), and m is the weight of the coal sample before burning (g).

2.4. SEM and EDS measurements

The particle shape and surface elementary composition of the coal particles of $74-45\ \mu\text{m}$ after ultrasonic treatment of different time were analyzed by Quanta 250 SEM (FEI, Hillsboro, Oregon, USA) and Bruker Quantax 400-10 EDS. The coal samples were prepared by surface cleaning using ethylalcohol, followed by air drying. Before SEM tests, the coal samples were sputter-coated with a layer of gold to increase the conductivity. About 60 particles were selected for the measurement of particle shape. The particles for EDS tests were selected randomly to obtain representative results of the actual experimental data.

3. Results and discussion

3.1. Results of particle size distribution

Chord length distribution of particles can be considered to be the size distribution in FBRM measurement. Figs. 3 and 4 show that the particle size of coal was reduced gradually with the increase of ultrasonic time as well as the volume percentage of $-45\ \mu\text{m}$ particles was gradually increased. Fig. 4 illustrates that the mean particle size for $74-45\ \mu\text{m}$ coal sample was decreased from $100.10\ \mu\text{m}$ to $22.80\ \mu\text{m}$ with the increase of ultrasonic time from 0 to 120 min, while the volume percentage of $-45\ \mu\text{m}$ coal particles was increased from 10.50% to 92.89%. The greatest pulverization effect was obtained when ultrasonic time was 120 min, and the particle size distribution was hardly changed with further increase of ultrasonic time.

Fig. 3 shows that the coal particle size distribution of the coarse fraction ($>74\ \mu\text{m}$) was changed insignificantly whereas the distribution of fine fraction was changed observably when the ultrasonic time was within 10 min. Fig. 4 illustrates that the volume percentage of the $-45\ \mu\text{m}$ coal particles was increased from 10.50% to 45.74%. The mean particle size of coal was decreased from $100.10\ \mu\text{m}$ to $55.70\ \mu\text{m}$ because a large number of $-45\ \mu\text{m}$ particles were produced. The energy input by ultrasound at this stage was not profound enough, so the surface pulverization caused by cavitation shock wave was the main reason for the reduction of particle size (Popplewell and Peleg, 1989). Plenty of $-45\ \mu\text{m}$ coal particles were mainly produced by friction and attrition of the surfaces of the $74-45\ \mu\text{m}$ particles and the size of the $74-45\ \mu\text{m}$ coal particles was left unchanged.

The particle size distribution was significantly changed when the ultrasonic treatment time was between 10 and 50 min. Fig. 4 shows that the volume percentage of $-45\ \mu\text{m}$ coal particles was increased by 69.54% compared with untreated sample as well as the mean particle size was decreased by $70.04\ \mu\text{m}$. As the ultrasonic time increases, the volume pulverization effect produced by ultrasonic micro jet can enable the $74-45\ \mu\text{m}$ particles to become smaller particles and the mean particle size of coal sample decreases rapidly (Popplewell and Peleg, 1989). It should be noted that surface pulverization also existed at this stage.

The particle size distribution was completely different from that of the original coal sample when ultrasonic treatment time was longer than 50 min. After 50 min ultrasonic treatment, the particle size distribution was hardly changed whereas the volume percentage of $-45\ \mu\text{m}$ particles was still increasing. With the reduction of coal particle size, it is difficult for the limited ultrasonic energy to change particle size distribution any further. Therefore, the surface pulverization became the main mechanical pulverization mechanism again and the volume pulverization might also exist in the process.

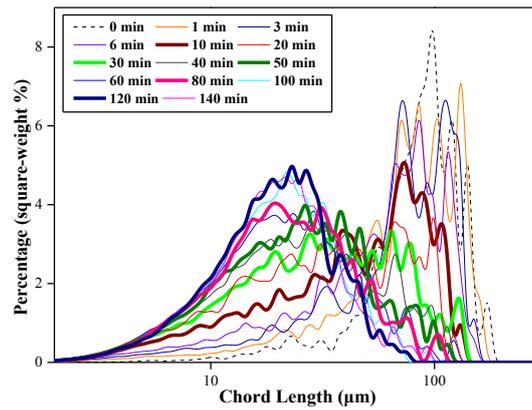


Fig. 3. Square-weighted FBRM chord length distribution of coal particles after ultrasonic treatment of different time

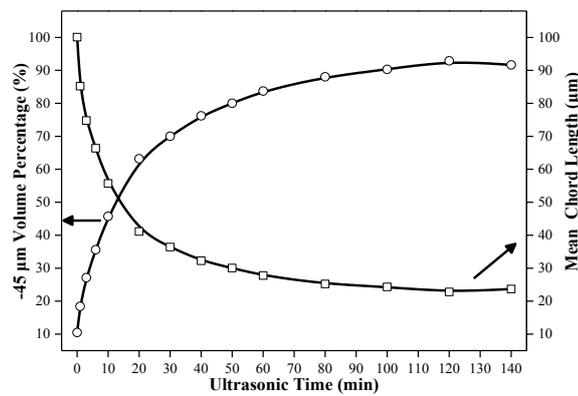


Fig. 4. Volume percentage of $-45 \mu\text{m}$ coal particles and mean chord length of coal particles after ultrasonic treatment of different time (square-weighted)

3.2. Results of particle shape measurement

Figs. 5 and 6 are the SEM pictures of $74\text{--}45 \mu\text{m}$ coal particles after 0, 10, 50, and 120 min ultrasonic treatment before and after image processing. It is obviously that the coal particles without ultrasonic treatment possessed many sharp corners whereas the coal particles after 10, 50, and 120 min ultrasonic treatment possessed many round corners. Based on Figs. 5 and 6, the roundness of each particle in the green rectangular frame was obtained using the image processing technology with ImageJ software.

Fig. 7 shows that the average roundness of $74\text{--}45 \mu\text{m}$ coal particles after 10 min, 50 min, and 120 min ultrasonic treatment were around 0.690, 0.689, and 0.741, respectively, while that of the coal particles without ultrasonic treatment was around 0.642, indicating that the roundness of coal particles was increased when ultrasonic treatment was applied. Generally, a higher roundness is not conducive to coal flotation (Dehghani et al., 2012; Ma et al., 2018; Rahimi et al., 2012; Rezai et al., 2010; Xia et al., 2017). Therefore, we can conclude that ultrasonic treatment of very long time is not conducive to coal flotation.

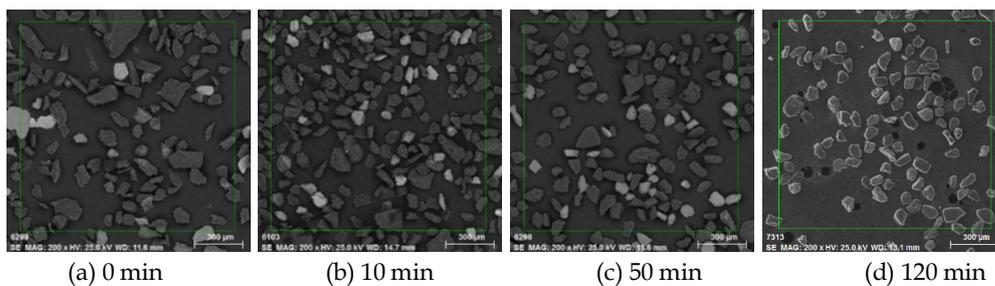


Fig. 5. SEM pictures of $74\text{--}45 \mu\text{m}$ coal particles after 0, 10, 50, and 120 min ultrasonic treatment before image processing

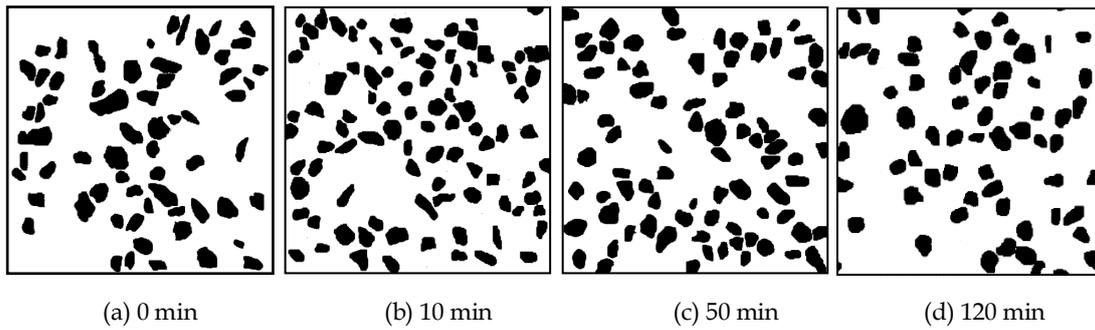


Fig. 6. SEM pictures of 74-45 μm coal particles after 0, 10, 50, and 120 min ultrasonic treatment after the image processing

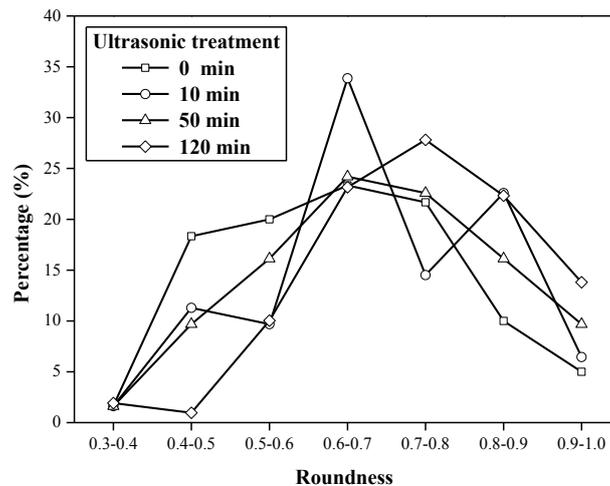


Fig. 7. Roundness distribution of 74-45 μm coal particles after 0, 10, 50, and 120 min ultrasonic treatment

The coal particles are mainly shattered by the friction and attrition of particle surfaces when ultrasonic treatment time was within 10 min, so the sharp corners on coal particles was changed to be round ones after 10 min ultrasonic treatment. However, the volume pulverization effects produced by ultrasonic micro jet played a dominant role in the reduction of particle size when ultrasonic treatment time ranged from 10 to 50 min. For the volume pulverization, individual particles were directly broken into several smaller ones without significant surface friction and attrition (Popplewell and Peleg, 1989). Therefore, the average roundness of coal particles after 50 min ultrasonic treatment was similarly to that after 10 min ultrasonic treatment. As the ultrasonic time continued to increase, limited ultrasonic energy could no longer cause volume pulverization of fine coal particles. But the friction and attrition of particle surfaces were more easily to change the sharp corners on the coal particles to round ones. As a result, the average roundness of coal particles after 120 min ultrasonic treatment was increased significantly compared with that after 0, 10, and 50 min ultrasonic treatment.

3.3. Results of particle ash content and EDS measurement

The 74-45 μm coal particles after 10, 30, 50, 80, and 120 min ultrasonic treatment were classified into 74-45 μm and <45 μm size fractions to investigate the effect of ultrasound on the ash content of coal. Fig. 8 indicates that the ash content of 74-45 μm coal particles was increased gradually with the increase of ultrasonic time, while the ash content of <45 μm fraction was decreased. The ash content of <45 μm size fraction was approximately the same to that of 74-45 μm coal particles and the difference was only 0.86% after 50 min ultrasonic treatment. This is because the surface cleaning and crushing caused by ultrasound changed the mineral composition of 74-45 μm coal surface. The reason for the effect of ultrasound on coal ash content will be further interpreted based on the EDS results.

Fig. 9 shows the representative EDS results for 74-45 μm coal particle surfaces after 0, 50, and 120 min ultrasonic treatment, respectively. The contents of oxygen, aluminium and silicon elements on the 74-45 μm coal surfaces in Fig. 9(b) and (c) were higher than those in Fig. 9(a). The surface elementary composition of each tested coal particle was obtained using the EDS picture. Based on the mathematical averaging method, the average surface elementary compositions of 74-45 μm and -45 μm coal particles after 0, 10, 30, 50, 80, and 120 min ultrasonic treatment were obtained respectively, as shown in Fig. 10 and Fig. 11.

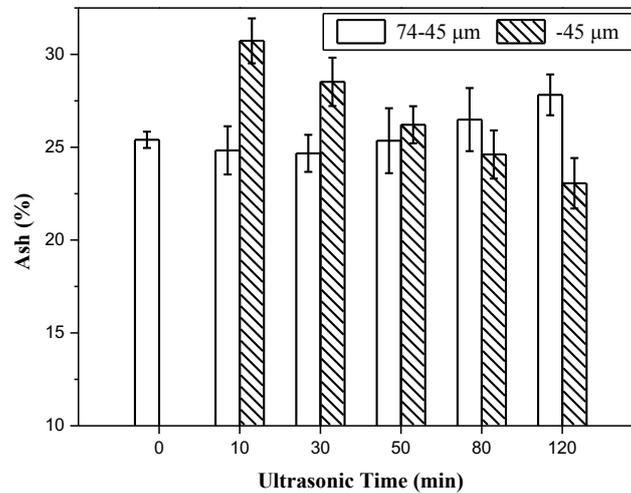


Fig. 8. Ash content of 74-45 μm and -45 μm size fractions after 10, 30, 50, 80 and 120 min ultrasonic treatment

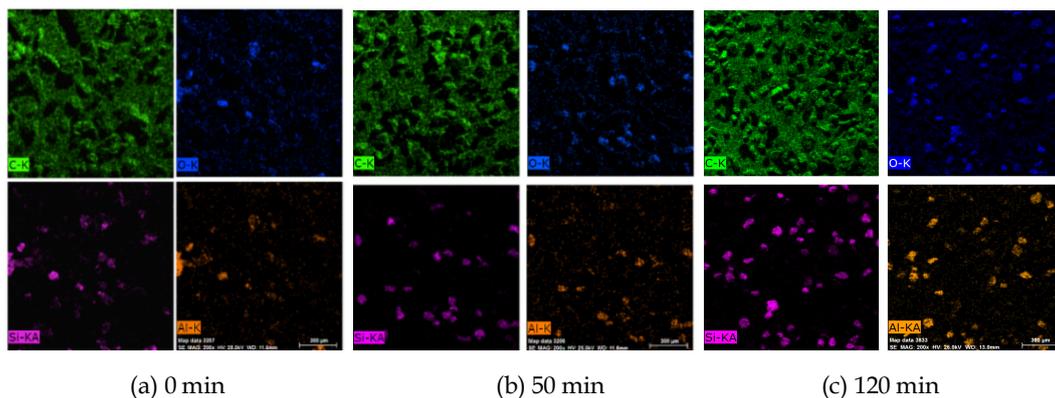


Fig. 9. EDS picture of representative 74-45 μm coal particles after 0, 50, and 120 min ultrasonic treatment

Fig. 10 indicates that the content of carbon element on 74-45 μm coal surfaces without ultrasonic treatment was higher than that with ultrasonic treatment longer than 80 min. The contents of aluminium and silicon elements on coal surfaces became high obviously when ultrasonic time was longer than 80 min. The contents of carbon, aluminium, and silicon did not change much when ultrasonic time was lower than 80 min. If we look back to Fig. 8, it is found that the ash content of 74-45 μm coal particles also changed small when ultrasonic time was lower than 80 min. It indicated that change of elements on coal surface can further affect the ash content. Fig. 11 shows that the content of carbon element on -45 μm coal surfaces was increased with the increase of ultrasonic time whereas the contents of aluminium and silicon elements gradually reduced. It indicates that the carbon or organic components were ground off from 74-45 μm coal surfaces and then comminuted during a long period ultrasonic treatment (Xia et al., 2017; Xia et al., 2014). This is due to the cleaning and crushing effect of ultrasonic cavitation on the surface of coal particles. When ultrasound spreads in the liquid, the core of the bubble generated by the ultrasonic cavitation has very high temperature and pressure (Chen et al., 2008). The rupture of cavitation bubbles would cause shock waves and micro jets. The surface of 74-45 μm coal particles is continually cleaned and attrited with the help of strong shock waves and micro-jets (Buttermore and Slomka, 1991; Suslick and Matula, 1999). Therefore, a small part of organic matters

could release from 74-45 μm coal surface during the ultrasonic treatment and more silicon and aluminium elements would appear on the fresh coal surface. For coal flotation, the carbon element plays a positive effect on coal natural hydrophobicity whereas the aluminium and silicon elements play negative effects on coal natural floatability (Hornsby and Leja, 1984; Zhang, 2016). After a long period of ultrasonic treatment, the natural floatability of 74-45 μm coal particle would be reduced and hence the flotation performance of long-ultrasound coal particles would be worse than that of short-ultrasound coal particles.

In addition, it is well known that the main components of coal ash are minerals such as kaolin, calcite, etc. The change of elements on the coal surface would further affect the ash content of 74-45 μm and -45 μm after ultrasonic treatment. As the ultrasonic time increases, the carbon or organic components would be gradually released from 74-45 μm coal surfaces while more silicon and aluminium elements would appear on the fresh coal surfaces. Therefore, the ash content of 74-45 μm coal particles increased and that of -45 μm coal particles reduced accordingly, as shown in Fig. 8.

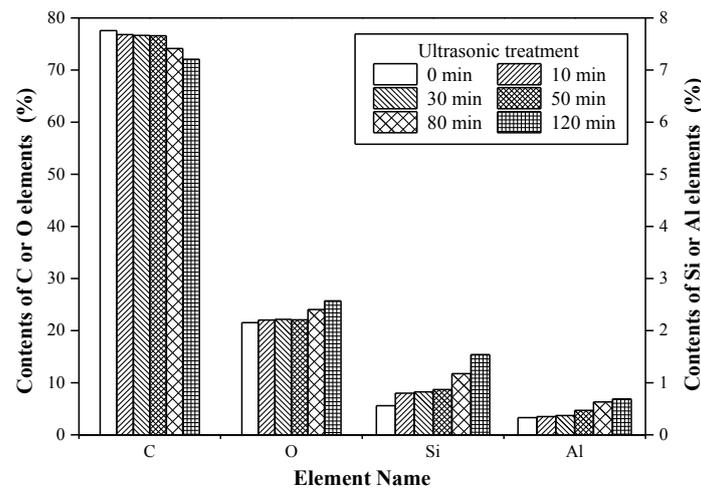


Fig. 10. Elements contents on 74-45 μm coal particle surfaces after 0, 10, 30, 50, 80 and 120 min ultrasonic treatment

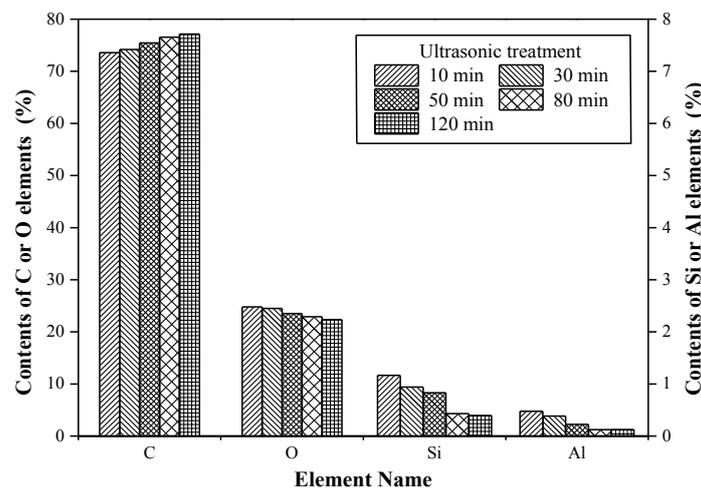


Fig. 11. Elements contents on -45 μm coal particle surfaces after 10, 30, 50, 80 and 120 min ultrasonic treatment

4. Conclusions

The surface pulverization caused by cavitation shock wave played a dominant role in the reduction of coal particle size when ultrasonic time was within 10 min. With longer ultrasonic time, the surface pulverization was gradually replaced by volume pulverization which was produced by ultrasonic micro jet. For ultrasonic treatment longer than 50 min, the surface pulverization became the main

mechanical pulverization mechanism again, although volume pulverization might also exist. In addition, the sharp corners on 74-45 μm coal particles were polished to round ones after ultrasonic treatment. A long period of ultrasonic treatment would be not conducive to coal flotation.

The surface of 74-45 μm coal particles was continually cleaned and attrited during a long period of ultrasonic treatment. The content of carbon element on 74-45 μm coal particle surfaces without ultrasonic treatment was higher than that with ultrasonic treatment of long time, whereas the contents of aluminium and silicon elements were lower than those after ultrasonic treatment. The content of carbon element on -45 μm coal surfaces was improved with the increase of ultrasonic time whereas the contents of aluminium and silicon elements gradually reduced. As a result, the ash content of 74-45 μm coal particles gradually increased with the increase of ultrasonic time, while that of -45 μm gradually decreased. Meanwhile, as the changes in the surface elementary composition on 74-45 μm coal particle, the floatability and hydrophobicity of coal particles would be reduced after a long period of ultrasound process.

Acknowledgements

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