

# Effect of ash existence in the coal particles on the dewatering performance of fine coal

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## Research

**Keywords:** fine coal, hydrophobic property, dewatering performance, ash

**Posted Date:** September 10th, 2020

**DOI:** <https://doi.org/10.21203/rs.3.rs-73624/v1>

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# Abstract

The dewatering experiments of fine coal with different ash contents in the particle size range of 0.125 mm – 0 mm were investigated in this study. Structures of coal samples were characterized by X-ray diffractometer (XRD) and surface functional groups were detected by Fourier transform infrared (FTIR). Wettability and wetting heats of coal samples were determined by contact angle measurements and micro-calorimeter system, respectively. In this study, the dewatering results indicate that the ash content of fine coal had less effect on the coal dewatering than the coalification degree in the dewatering process. However, for the given coal sample the moisture content was significantly affected by the ash content while the coal particle size was less than 0.125 mm. The decrease of moisture content in coal sample after the ash was removed indicating that the hydrophobic property of coal surface was enhanced based on contact angle measurements and wetting heats. In addition, kaolinite played a primary role of minerals in coal for the coal dewatering.

## Introduction

Flotation is the most effective method of separating fine coal from clay, sulfide, carbonate, silica, sulfate and other minerals (Xing et al., 2017; Zhang et al., 2018; Hacifazlioglu, 2018). After flotation the obtained fine coal must be dewatered for practical application and economic cost (Wang et al., 2014; Zhang et al., 2017). The moisture in the coal was reduced as much as possible, which has played an important role of clean coal technology. Meanwhile, negative impacts of high moisture content in coal such as transportation costs, difficult storage and lowered calorific value, were resulted from the additional moisture in the coal (Gong et al., 2010). These methods, including mechanical compression, thermal drying, filtration, are employed in the dewatering process of fine coal (Roux et al., 2005; Asmatulu, et al., 2009; Burat et al., 2015). Among these methods, filtration methods such as pressure filters, belt presses, vibrating basket and vacuum filters, have been widely used in the dewatering of coal product (Alam et al., 2011). Several process parameters (flocculant conditioning time, cake formation and drying time, size distribution etc.) were investigated in the dewatering process of ultrafine coal via vacuum filtration. The results indicate that the coal dewatering performance can be enhanced by optimizing these process parameters (Tao et al., 2000). Effects of filter cakes, equilibrium desaturation and filtration rates were also studied in the dewatering process of fine coal (Gala et al., 1981). The vacuum filtration was developed in the dewatering of fine coal (Roux et al., 2003). Tests on a bench scale vacuum filter demonstrated that the increase of the air flow passing a filter cake was favorable to the dewatering of fine coal.

Further fundamental investigations and auxiliary measures of coal dewatering were reported in the literature. Some surfactants were used in the tests of increasing coal dewatering performance (Hassas et al., 2014). The results indicate that low HLB (hydrophile- lipophilic balance) surfactants were efficient in the dewatering of bituminous and anthracite coals while not efficient in the dewatering of low rank coals such as lignite. The dewatering performance of fine coal from flotation was investigated and the dewatering process was investigated by the oscillatory rheology of froth (Zhang et al., 2018). Their

results indicated that the presence of stabilized froth on top of water was not favorable to the decrease of the moisture of filter cake. Surface hydrophobicity and air bubble entrapment on the filtration were studied in the fine coal dewatering (Asmatulu, 2008). It can be found that after hydrophobic reagents and air bubbles were introduced into the coal slurry the amount of moisture in fine coal was decreased and the efficiency of the filtration was increased.

Besides the above dewatering methods, coal ash from minerals in the coal may play an important role in the dewatering process although a large part of the minerals in the coal were removed by flotation. There are few detailed studies about the effects on filtration behavior and the fine coal dewatering. In this study, the effect of ash content on the dewatering performance of the fine coal was investigated based on coal samples in different coalification degrees, ash contents for a given coal sample and mineral types. The obtained results were hopefully provided for understanding the dewatering process of fine coal in the coal preparation plants.

## Experimental Section

### 2.1 Materials

Coal samples are collected from the coal preparation plant in Shanxi, China. Coal particle size is less than 0.125 mm. Two sub-bituminous coal samples are denoted as C<sub>1</sub> and C<sub>2</sub> according to the increase of coal deterioration. The bituminous coal is denoted as C<sub>3</sub>. The results of the proximate analysis of coal samples are listed in Table 1. Kerosene (Xi'an Chemical Reagent Factory) was used as a collector and 2-octanol was used as a frother (CP. 97%, Xi'an Chemical Reagent Factory).

### 2.2 Methods

#### 2.2.1 Sample characterization

Before the proximate analysis was carried out, the coal samples were placed in a 105 °C vacuum drying oven and dried for 2 h. The ultimate analysis of coal was performed by the elemental analyzer (vario MACRO cube elementar, Germany). The crystalline structures of the prepared samples were detected by a Rigaku X-ray diffractometer (XRD) equipped with Cu-K α radiation at 40 kV and 40 mA. The powder diffraction pattern was studied in scanning range from 5° to 60° at 4°/min and 0.02 (2q) step size. Fourier transform infrared (FTIR) spectra were collected on a Bruker Tensor 27 spectrometer at room temperature and the mass ratio of sample powder to KBr (100 mg) was 1:100. The FTIR spectra were ranged from 4000cm<sup>-1</sup> to 400 cm<sup>-1</sup>. Contact angle measurements (JC 2000C) of coal samples were performed for wettability of coal samples. Coal samples (<74μm) were pressed to form a plate under a 40 MPa pressure, and then a water droplet was contacted with the coal plate. Wetting heats of coal samples (<74μm) were determined by using a micro-calorimeter system (C80, Setaram, France). The thermostatic mode at 303 K and membrane-mixing stainless steel cell (volume: 8.5 mL) were adopted. The aluminum foil film was fixed between deionized water (2 ml) and coal powder sample (100 mg) in

stainless steel cells. When the system of apparatus has no change at room temperature, the wetting heat was obtained after the aluminum foil film was pierced by integration of the heat-flow curve.

## 2.2.2 Flotation, ash removal and dewatering tests

The flotation test was performed in a 140 mL micro-flotation cell with the speed of 1800 r/min (XFG, Wuhan Exploring Machinery Factory), the airflow rate was 0.2 L/min, and the pulp density was 100 g/L. The conditioning before flotation lasted for 3 min. Kerosene as a collector(600g/t, 800g/t, 1500g/t) was added after 2 min of conditioning and 2-octanol as a frother (100g/t)was added after 3 min of conditioning. The mixing time was further for 10 s, and then clean coal product was collected. The ash in coal (20g) was removed by using 80 ml hydrochloric acid (37%, Tianjin Chemical Reagent Factory) and 80 ml hydrofluoric acid (40%, Tianjin Chemical Reagent Factory)at 60 °C for 1 h, respectively. And then the sample was again mixed with 80 ml hydrochloric acid (37%) at 60 °C for 1 h. The obtained sample was rinsed with distilled water three times and dried in a vacuum drying box at 60 °C for 4h.The coal slurry was prepared via pouring the coal particles with a given mass into the distilled water, and then the coal slurry was filtered by a vacuum pump at 50 kPa. The coal sample was dried in an oven at 105 °C for 90 min and the moisture content in coal was obtained.

# Results And Discussion

## 3.1 Proximate and Ultimate analysis

The proximate analysis and ultimate analysis of coal are listed in Table 1. From the results, it is found that the ash content in the C3 coal sample is highest among the three samples reaching up to 37.52%. The moisture content decreases with the increase of coal rank (C1 < C2 < C3), which is related to the abundant oxygen-containing functional groups on the surface of the C1 coal sample as shown in IR spectra (Figure1). The oxygen content of C1 was more than those of C2 and C3 based on the ultimate analysis indicating that more oxygen-containing functional groups on the surface of the C1 coal sample were existed. The volatile matter content increased with the increase of coal rank (C1 < C2 < C3). The fixed carbon content was significantly affected by the above three contents of fine coal, and thus the fixed carbon content of C3 sample was lowest due to the ash content of 37.52%.

## 3.2 XRD and IR characterization

XRD spectra of coal samples are shown in Fig. 1. The XRD characterization result indicates that there are three main kinds of minerals such as kaolinite, quartz and pyrite besides amorphous organics in coal samples. According to the XRD spectrum of C1 coal sample, kaolinite and quartz are main minerals and a small amount of pyrite is presented in the coal. The XRD spectrum of C2 coal sample is similar to that of C1 coal sample. However, three kinds of characteristic peaks in relatively strong intensities could be found in the XRD spectra of C3 coal sample due to the presence of minerals, mainly including kaolinite, quartz and a small amount of pyrite (Lin et al., 2017; Zhou et al., 2019; Zhou et al., 2019), which can be supported by the above proximate analysis. In addition, from XRD spectra pyrite content of C3 coal

sample was more than those of C1 coal sample and C2 coal sample, which was illuminated by the sulfur content in the proximate analysis.

IR spectra of coal sample in different coalification degrees are shown in Fig. 2. The peaks at 3438 cm<sup>-1</sup> and 1097 cm<sup>-1</sup> are ascribed to the presence of hydroxyl groups such as alcohol or phenolic hydroxyl, and this peak intensity decreases with the increasing coal rank (C1 > C2 > C3) (Zhen et al., 2018). Three characteristic peaks at 2920 cm<sup>-1</sup>–1440 cm<sup>-1</sup> and 1375 cm<sup>-1</sup> are due to the stretching vibration of methyl and methylene groups that increase with the increasing coal rank (C1 < C2 < C3) (Chen et al., 2019). The strong absorption peak at 1600 cm<sup>-1</sup> may be attributed to the overlapping of carbonyl group and C=C double bonds in the aromatic ring (Zhao et al., 2018). Five peaks at 3695 cm<sup>-1</sup>, 3617 cm<sup>-1</sup>, 1032 cm<sup>-1</sup>, 1010 cm<sup>-1</sup> and 910 cm<sup>-1</sup> are assigned to the minerals in the coal and the peaks of C3 coal sample is strongest among three samples, which indicates that the highest content of minerals is in C3 coal sample supported by the above proximate analysis (Valeria et al., 2000; Yang et al., 2019; Zhao et al., 2017). In addition, the amount of oxygen-containing functional group decreases with the increase of coalification degree, which may result in the hydrophobicity increase of coal and may be advantageous for the dewatering of fine coal (Xia et al., 2019).

### 3.3 Effect of coal samples with different ash contents

Firstly three coal samples with different ash contents were employed in the dewatering tests. As shown in Fig. 3, the moisture content in the filter cake changes in the time range 2.5 min to 8.5 min. The solid content in the slurry is 200 g/L and the particle size is less than 0.125 mm. For C1 coal sample, the moisture content was up to 30.7% on 2.5 min and decreased with the filtration time increase. The decrease about 3.8% was gained on 8.5 min. For C2 coal sample, the moisture content in the filter cake decreased in the experimental time and the final moisture content was 17.8%. For C3 coal sample, the moisture content decreased from 22.4% to 17.1%. When the filtration time was 8.5 min, the decrease of moisture content was hardly found. According to the above results, it can be found that the moisture content in the filter cake deceases with the increase of coalification degree and filtration time. Although the ash content of C3 was high about 37.52%, the final moisture content in the filter cake was only 17% indicating that the coalification degree had more important role in the moisture content in the filter cake than ash content in the coal.

### 3.4 Effect of ash contents in the coal samples

The presence of clay minerals in coal is not beneficial to the solid–liquid process (Ofori et al., 2011; Rong et al., 1995; Tao et al., 2000). Therefore, the effects of ash contents in coals should be investigated in the dewatering process. The ash content of coal was achieved by the coal flotation using different dosage of collector. The C3 coal sample was used in this model test due to its high ash content. The solid content in the slurry is 200 g/L and the particle size is less than 0.125 mm. The filtration time is 6.5 min. Flotation and dewatering results are shown in Table 2. The ash content in the coal decreases about 10% for collector dosage of 600 g/t, and increases slightly for collector dosage of 800 g/t and 1500 g/t. From

Table 2, it can be seen that the formation time of cake decreases from 46.1 s to 28.8 s and the moisture content of cake decreases from 17.10% to 14.18% when the ash was removed from coal by the flotation. However, for three coal samples by the flotation similar formation times were gained and the moisture content of cake increased about 1.2% and 1.4% for collector dosage of 800 g/t and 1500 g/t, respectively. The obtained results indicate that the presence of ash in coal is harmful to the dewatering of fine coal.

In order to investigate the further effect of ash on the dewatering of fine coal, the ash in coal was mostly removed by using hydrochloric acid and hydrofluoric acid. Residual ash contents are 1.20% for C1, 1.01% for C2 and 3.85% for C3, respectively. The solid content in the slurry is 200 g/L and the particle size is less than 0.125 mm. The filtration time is 6.5 min. Moisture contents in the filter cakes before and after ash removal are shown in Fig. 4. From this figure, the moisture content decreased after the ash was removed from coal. The decrease of moisture content was about 2.3% for C1, 2.6% for C2 and 3.8% for C3 indicating the ash in the coal is responsible for the bad dewatering performance.

Images of water droplet contacting with coal surface before and after ash removal are shown in Fig. 5. With the increasing coalification degree, the contact angle increased from 7.8° to 46.8°(Fig. 5 a, c, e) demonstrating the hydrophobic property of coal surface became enhanced. When the ash in the coal was removed, all hydrophobic properties of three coal samples were enhanced and the C3 coal sample still presented the strongest hydrophobic property (Fig. 5 b, d, f). Moreover, based on the dewatering result in Figure 4, the decrease of moisture content in coal sample after the ash was removed indicating that the hydrophobic property of coal surface was enhanced, which may be responsible for the decrease of moisture content in the filter cake.

The resulting heat flow curves are shown in Fig. 6. With the increase of coalification degree, the wetting heat flow significantly decreased indicating that the hydrophilicity of coal became weak (C1:33.9J/g, C2: 8.9 J/g, C3: 3.6J/g). After the removal of ash, the wetting heat flows were 29.4J/g for C1, 6.4 J/g for C2, 1.6 J/g for C3, respectively. According to the results of XRD, it can be known that kaolinite and quartz are predominant minerals in the coal sample. Therefore, heat flow curves of kaolinite and quartz were given in Fig. 7. Form this figure, the wetting heat value of kaolinite was 2.6 J/g and larger than that of quartz (0.4 J/g). And thus kaolinite played a primary role of minerals in coal for the coal dewatering. Based on the above results, three coal samples presented the wetting heat decrease after ash removal indicating that hydrophobic properties of three coal samples were enhanced. Therefore, coal samples exhibited the decrease of moisture content in the filter cake.

## Conclusion

Three coal samples were used in the dewatering tests and the particle size was less than 0.125 mm. The proximate analysis and ultimate analysis of coal indicate that the ash content in the C3 coal sample was highest among the three samples reaching up to 37.52%, mainly including kaolinite and quartz, a small amount of pyrite supported by XRD characterization. IR result indicates that the amount of oxygen-containing functional group decreases with the increase of coalification degree. According to the

obtained results from three coal samples, it can be found that the moisture content in the filter cake deceases with the increase of coalification degree and filtration time. The ash content had less effect on the moisture content in the filter cake than the coalification degree of the coal sample. After the ash was removed by flotation or by using hydrochloric acid and hydrofluoric acid, the moisture content of filter cake decreased indicating that the presence of ash in coal is not beneficial to the dewatering of fine coal. Contact angle increased and wetting heats decreased after ash removal in coal samples indicate that coal samples presented stronger hydrophobic property. Based on the XRD characterization and wetting heat values of the minerals in the coal sample, it can be found that kaolinite played a primary role of minerals in coal for the coal dewatering.

## Declarations

### Compliance with Ethical Standards

### Conflict of Interest

The authors declare that they have no conflict of interest.

### Acknowledgments

The authors gratefully acknowledge the financial support of the Natural Science Foundation of Shanxi Province (Grant number: 201901D111072), the National Natural Science Foundation of China (Grant number: 51304144), the Study Abroad Program for the University-Sponsored Young Teachers (Grant number: 2017, Taiyuan University of Technology).

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## Tables

**Table 1** Proximate and Ultimate analysis of coal in different coalification degrees <sup>a</sup>

sample	Proximate analysis (wt. %)				Ultimate analysis (wt. %)				
	M <sub>ad</sub>	A <sub>ad</sub>	V <sub>ad</sub>	FC <sub>ad</sub>	C <sub>daf</sub>	H <sub>daf</sub>	O <sub>daf</sub> <sup>b</sup>	N <sub>daf</sub>	S <sub>daf</sub>
C1	2.97	15.10	29.60	52.33	79.58	4.64	14.37	0.93	0.49
C2	1.30	11.73	20.88	66.09	85.31	4.17	9.29	0.79	0.44
C3	1.16	37.52	22.14	39.18	78.28	4.99	13.41	1.42	1.91

a. ad, air-dry basis; daf, dry ash-free basis M, moisture content; A, ash content; V, volatile matter; FC, fixed carbon;

b. by difference

**Table 2** Ash content, the cake formation time and the cake moisture in the condition of different dosage of collectors

Collector dosage kg/t	Ash content <sup>a</sup> wt. %	Formation time of cake (s) <sup>*</sup>	Moisture of cake (wt. %)
0	31.93	46.1	17.10
600	22.72	28.8	14.18
800	23.28	28.7	15.39
1500	23.82	28.5	15.54

<sup>a</sup>Water droplets did not drip in the filtration process.

## Figures

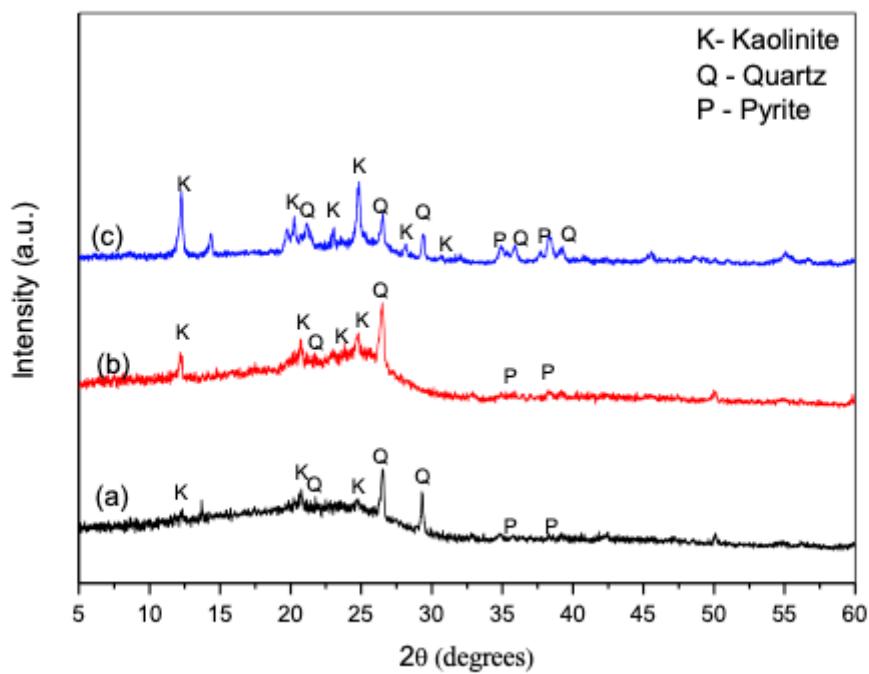
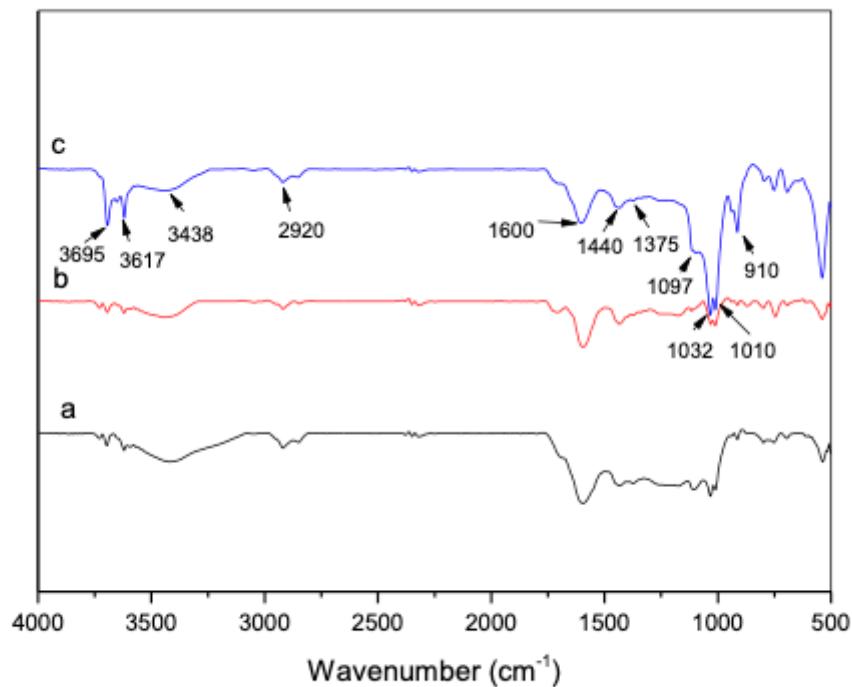


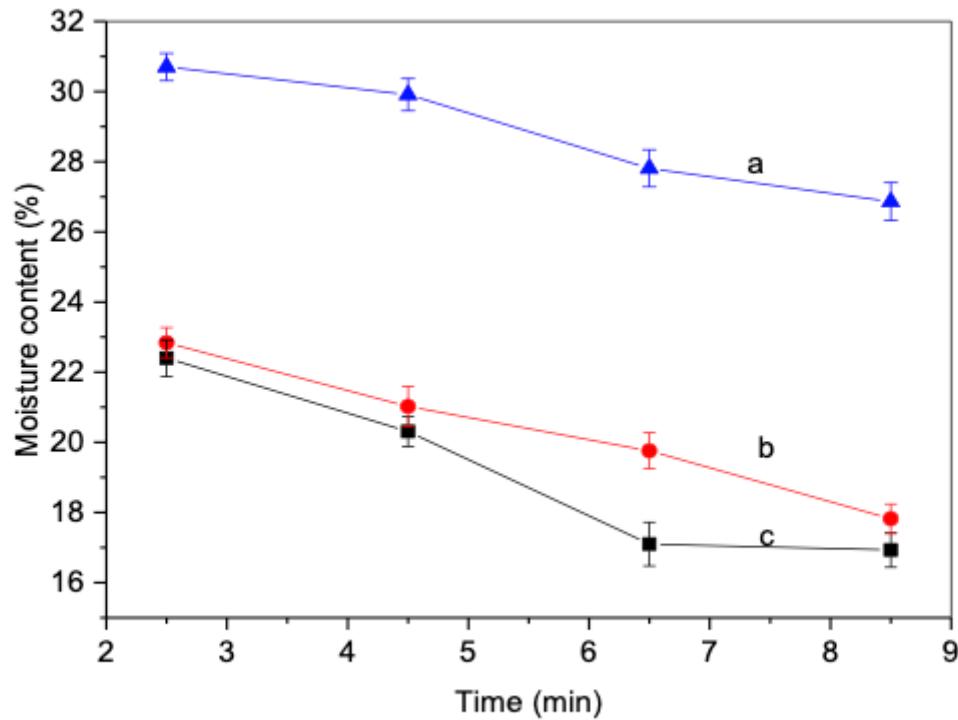
Figure 1

XRD spectra of coal samples in different coalification degrees, C1(b) C2(c) C3



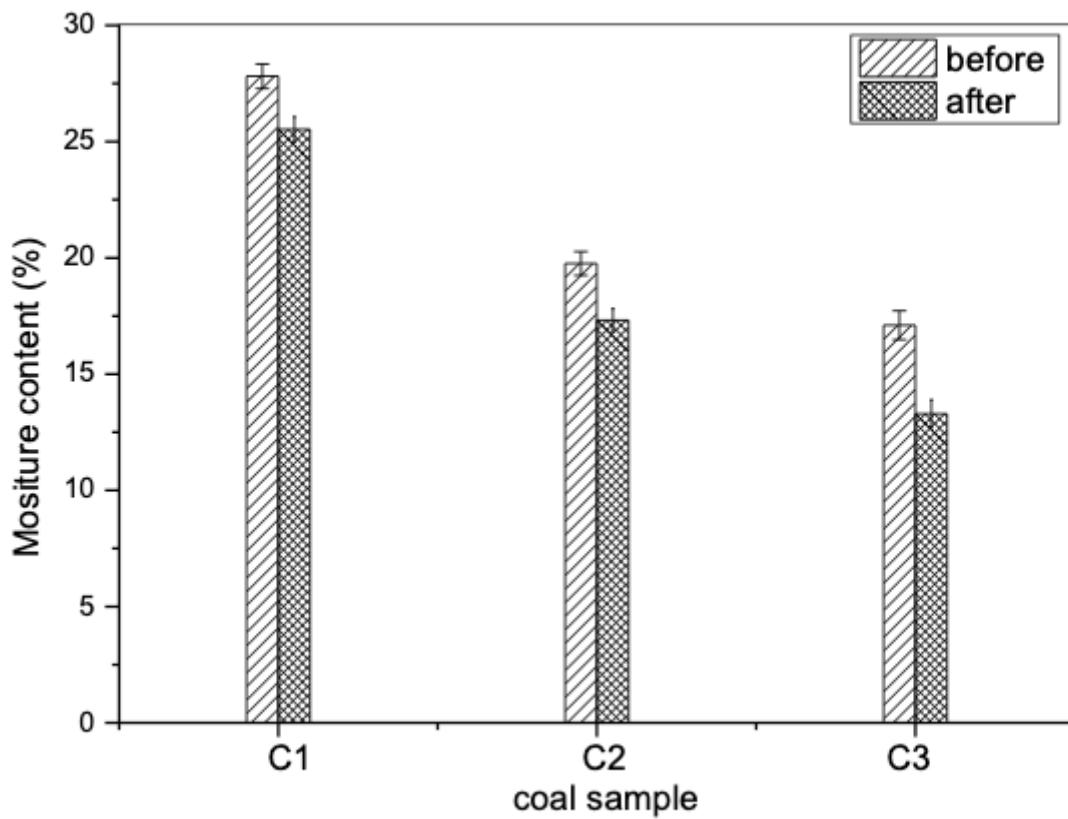
**Figure 2**

IR spectra of coal samples in different coalification degrees, (a) C1 (b) C2 (c) C3



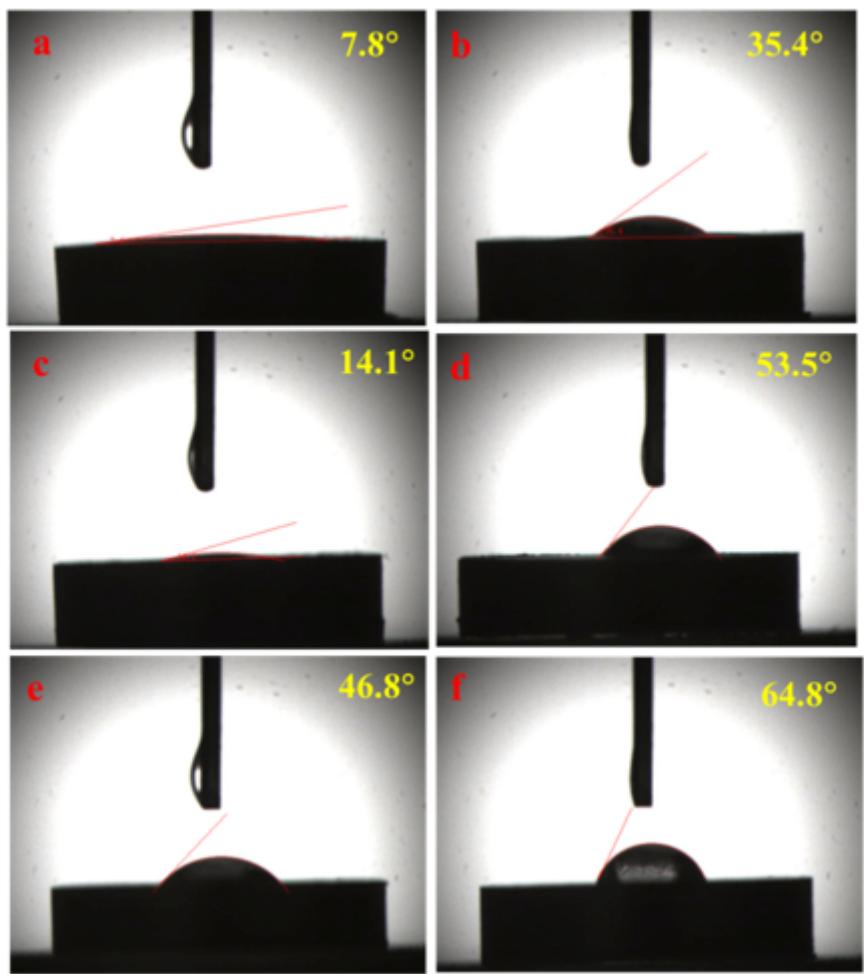
**Figure 3**

Effect of filtration time on cake moisture content, (a) C1 (b) C2 (c) C3; (The error bars represent one standard deviation obtained from three independent runs.)



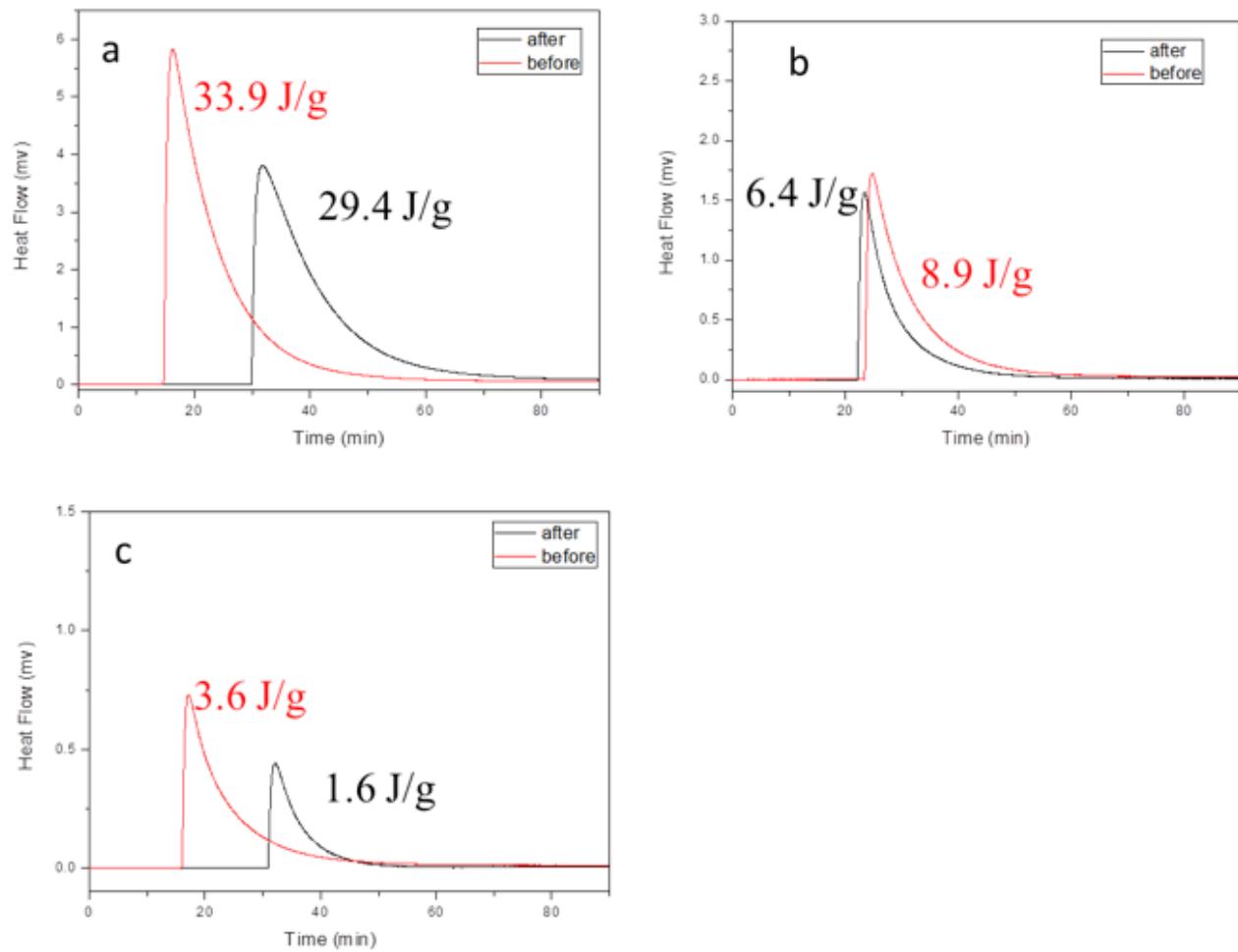
**Figure 4**

Cake moisture contents before and after ash removal; (The error bars represent one standard deviation obtained from three independent runs.)



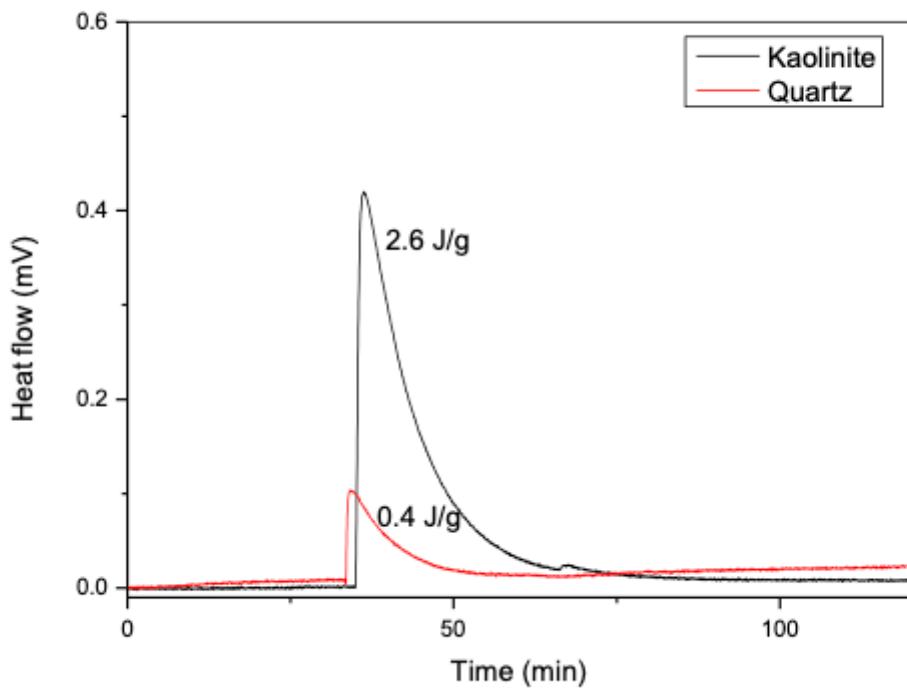
**Figure 5**

Images of contact angle measurements before and after ash removal: (a), (b), C1; (c), (d), C2; (e), (f), C3.



**Figure 6**

Wetting heat flow curves of coal samples before (red line) and after (black line) ash removal: (a) C1 (b) C2 (c) C3



**Figure 7**

Wetting heat flow curves of kaolinite (black line) and quartz (red line).