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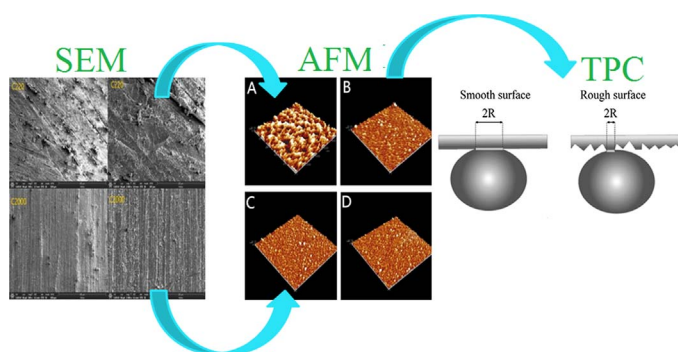
# Effect of surface roughness of Chinese sub-bituminous coal on the kinetics of three-phase contact formation



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## GRAPHICAL ABSTRACT



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## ABSTRACT

In this paper, terminal diameter and expansion velocity of the three-phase contact (TPC) line on the low-rank coal surfaces with different surface roughness were investigated. To eliminate the heterogeneity effect on the hydrophilic surfaces of low-rank coal particles, experimental samples with low ash content were adopted. Moreover, the analysis result of XPS indicated that there were few hydrophilic mineral particles on the low-rank coal surface. Furthermore, from the analyses of SEM and AFM measurements there are many crevices (pillars, protrusions) of different sizes (height and width) in different local areas of low-rank coal surfaces. Therefore, the three-phase contact lines on the low-rank coal surfaces presented different wetting behaviors. It demonstrated that the wetting film diameter became smaller and the expansion velocity of the three-phase contact line was faster while the surface roughness of low-rank coal samples increased.

## 1. Introduction

The expansion process of the three-phase contact line between an air bubble and a solid surface is vitally significant for froth flotation applied in the recovery of coal and minerals [1–4]. In the flotation process, three elementary consecutive steps should successfully take place while an air bubble attaches to a particle surface within a very

short time: (i) the thinning process of the wetting film between an air bubble surface and a particle surface to a critical thickness, (ii) the rupture of the wetting film at the critical thickness and formation of an initial three phase contact nucleus and (iii) the expansion of initial three phase contact to a stable wetting perimeter [5–9]. Meanwhile, it has been verified that a so-called long range hydrophobic force exists between the hydrophobic surface of a solid immersed into aqueous

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solutions and an air bubble, which may be up to hundreds of nanometres [10–13]. Recently, many researchers illustrated that the hydrophobic force played a significant role in the rupture of the wetting film and the formation of the three-phase contact (TPC) under dynamic conditions [1,14–16]. It was also confirmed that the more hydrophobic the surface, the less stable is the wetting film [10,17,18]. Moreover, another important feature of the solid surface, roughness, obviously affects the stability of the wetting film separating the colliding bubble from the solid surface in aqueous solutions. It was reported that the solid surfaces with the same chemical and physical properties but with different roughness presented different wetting behaviors [7,16,19–21]. Furthermore, the stability of the wetting film can be modified by the adsorption of non-ionic, anionic and cationic surface-active substances on the surface of a solid and the concentration of surfactant solutions [15,20,22–25].

In order to investigate the kinetics and mechanism of the three-phase contact (TPC) formation by the colliding bubble to model hydrophobic and hydrophilic solid surfaces, experimental materials such as polytetrafluoroethylene-Teflon and muscovite mica are commonly adopted, respectively. However, the surfaces of coal particles with different surface roughness are rarely used because it is difficult to obtain a homogeneous surface, especially on low-rank coal surface. It is well known that low-rank coals such as lignite, brown coal and sub-bituminous coal are very abundant in China [26]. Especially in the northwest of China, there are many sub-bituminous coal beds in the Shendong Coalfield. Oxidized surfaces of low-rank coal with abundant oxygenated functional groups such as hydroxyl, carbonyl and carboxyl, generally reduce the hydrophobicity and easily form hydrogen bonds with water molecules. Therefore, the surfaces of low-rank coal particles have strong hydrophilicity. To obtain a low-rank coal lump with homogeneous and hydrophilic surfaces, low-rank coal samples were separated firstly by density. Then the  $< 1.3 \text{ g/cm}^3$  density fraction of coal lumps were used as experimental samples, which were polished using sand papers with different meshes by hand. Moreover, in order to investigate the heterogeneity effect on the low-rank coal surfaces, the analysis of X-ray Photoelectron Spectrometer (XPS) was adopted. In this paper, depending on high speed camera technology, the effect of hydrophilic surfaces of low-rank coal with different surface roughness on three-phase solid–gas–liquid contact formation was investigated.

## 2. Experimental method and materials

### 2.1. Materials

Experimental samples of low-rank coal lump were collected from a coal preparation plant in the Shanxi province of China, which is a sub-bituminous coal. In order to eliminate the heterogeneity effect on the coal surface, low-rank coal bulk was separated firstly by density. The  $< 1.3 \text{ g/cm}^3$  density fraction was used as experimental samples, which were polished using sand papers with different meshes by hand. The sand papers were made by the Matador in Germany, on which surfaces the particle mesh is 220, 2000, 3000, and 5000. Polished by these sand papers, surfaces of the low-rank coal lumps with different surface roughness were obtained. For convenience, these four coal samples with polished surfaces will be further referenced in the text as C220, C2000, C3000 and C5000, respectively.

Some part of the  $< 1.3 \text{ g/cm}^3$  low-rank coal lump was used for X-ray Photoelectron Spectrometer measurements. Before the XPS tests, the coal samples were crushed and then screened. The  $-0.500 \text{ mm}$  particle size fraction was followed by grinding process. Grinding process was conducted in a laboratory dry rod mill. Grinding times were set at 5 min. After grinding step, the particle materials were wet screened through  $0.250 \text{ mm}$  sieve to obtain coal samples with the  $0.500\text{--}0.250 \text{ mm}$  size fraction for XPS studies. It should be noted that the mass of the  $0.500\text{--}0.250 \text{ mm}$  size fraction accounts for more than 90% of the total  $< 1.3 \text{ g/cm}^3$  density fraction of low-rank coal bulk

**Table 1**  
Proximate and ultimate analyses of low-rank coal sample.

Proximate analysis, %				Ultimate analysis, %				
$M_{\text{ad}}$	$V_{\text{ad}}$	$FC_{\text{ad}}$	$A_{\text{ad}}$	$C_{\text{daf}}$	$H_{\text{daf}}$	$O_{\text{daf}}$	$N_{\text{daf}}$	$S_{\text{daf}}$
6.07	38.79	53.23	1.91	70.60	3.98	22.09	0.97	2.36

after the crushing process because the grinding times were controlled within 5 min.

Proximate and ultimate analysis results of low-rank coal samples with the  $0.500\text{--}0.250 \text{ mm}$  size fraction are shown in Table 1, where  $M_{\text{ad}}$  is the moisture content,  $V_{\text{ad}}$  the volatile matter content,  $FC_{\text{ad}}$  the fixed carbon content,  $A_{\text{ad}}$  the ash content, and  $C_{\text{daf}}$ ,  $H_{\text{daf}}$ ,  $O_{\text{daf}}$ ,  $N_{\text{daf}}$  and  $S_{\text{daf}}$  are the contents of carbon, hydrogen, oxygen, nitrogen and sulfur, respectively. As shown in Table 1, the content of  $O_{\text{daf}}$  is 22.09%, which is only lower than that of  $C_{\text{daf}}$ , 70.60%. Therefore, it demonstrated that the surface of the low-rank coal sample was heavily oxidized. This will be discussed in detail in the XPS analysis section.

### 2.2. XPS measurement

To investigate the heterogeneity effect on the low-rank coal surface, experimental samples with  $0.500\text{--}0.250 \text{ mm}$  size fraction for X-ray Photoelectron Spectrometer (XPS) analyses were conducted at  $25^\circ\text{C}$  in extreme vacuum environment with a solid surface analysis set (ESCALAB 250Xi, America). The peak fitting of data processing was analysed by the XPS peak fit software [27–29]. The surface binding energies of solid particles were corrected by the C1s hydrocarbon ( $-\text{CH}_2-\text{CH}_2-$  bond) binding energy at  $284.8 \text{ eV}$ .

### 2.3. SEM measurement

In order to analyze the surface morphology of low-rank coal samples with different surface roughness, Quanta 250 SEM (FEI Quanta 250, USA) was used. Before the measurements, low-rank coal samples with flat surfaces were prepared by a surface cleaning process using pure ethyl alcohol. After the surface cleaning process, the low-rank coal samples were dried in air. Magnification times of the SEM measurements were fixed at 500 and 2000.

### 2.4. Surface roughness measurements

The atomic force microscope (AFM) is a powerful tool for surface roughness measurements. In AFM measurements, the tip contacts with the testing sample surface while scanning the sample in a raster way by the driver of a piezo-scanner [30]. Surface heights of low-rank coal samples were defined by the location of the tip apex. Furthermore, the three-dimensional topography data acquired from AFM (Bruker Dimension Icon, USA) tests can be easily used for further evaluation of surface roughness.

### 2.5. Three-phase contact formation measurements

The device photograph for three-phase contact formation measurement is shown in Fig. 1. Inside the column cell, a capillary connected with the microinjector through a soft tube at the bottom was designed and the low-rank coal sample with a flat surface was fixed at the top of the column. The process of three-phase contact formation was recorded by a high speed camera. The frame number of the high-speed camera was set to 750 per second. So, interval time of consecutive photos is  $1.33 \text{ ms}$ . The terminal velocity of the bubble in the three-phase contact formation measurements was determined to be about  $32.75 \text{ cm/s}$ . The bubble size was calculated by Eq. (1):

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