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## ABSTRACT

In this work, the interaction behavior and underlying mechanism of coal and kaolinite particles in the presence of agitation was investigated by flotation tests, homo and hetero settling tests, turbidity meter, zeta potential distribution, focused beam reflectance measurement (FBRM) and scanning electron microscope (SEM). The results show that, in the presence of kaolinite, the flotation combustible matter recovery of coal decreases from 0 rpm to 1200 rpm and then increases from 1200 rpm to 2000 rpm, indicating that the mild agitation enhances kaolinite-coating and the high intensity agitation mitigates the kaolinite-coating, which is demonstrated by the zeta potential distribution and turbidity measurements. However, the degree of the heterocoagulation between coal and kaolinite is not serious. It is found that the kaolinite particles partially cover on the coal surface by two morphologies includes individuals and aggregates through the size-based scanning electron microscope analysis. In addition, the affinity between fine kaolinite and fine coal particles is more pronounced. The kaolinite-coating enhancement caused by the mild agitation is explained by the Derjaguin–Landau–Verwey–Overbeek (DLVO) theory.

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## 1. Introduction

Flotation is widely used in coal industry for treating fine coal. This process greatly relies on the distinct interfacial properties between coal and gangue minerals. Good hydrophobicity of the coal surface is the key to ensure the success of the flotation. Unfortunately, nowadays China coal industry is facing the challenge of processing clayey coal as a result of the depletion of high quality coal deposits.

According to the literature, clay particles, usually  $<2 \mu m$ , have a significant effect on coal flotation [1–3]. For example, fine hydrophilic clay particles are easily dragged by the interstitial liquid film between air-bubbles and enter the froth layer with liquids in flotation, resulting in mechanical entrainment and low concentrate quality [1,2]. Clay minerals can also increase reagent consumption and pulp viscosity, which lowers the flotation efficiency [4,5]. One of the most important effects caused by clay minerals is slime coating, defined as a layer of fine or ultrafine colloidal particles coated on the larger value mineral surface, which has been recognized to have a deleterious effect on flotation. It is speculated that these slimes on mineral surface form a hydrophilic "armor" preventing mineral particles from contacting with collectors and/or air bubbles, consequently resulting in a lower flotation recovery [6–8]. Numerous researchers have demonstrated that hydrophilic clay

\* Corresponding authors. E-mail addresses: mlqiang@sina.com (L. Ma), xuhongxiang001@163.com (H. Xu). the adsorption of collectors and therefore depress coal flotation [4,6,9–12]. Originally, Derjaguin–Landau–Verwey–Overbeek (DLVO) theory

particles may coat the coal surface, making it hydrophilic and preventing

was developed to interpret the aggregate stability of colloidal particles, and recently researchers used this theory to explain the interaction behaviors of mineral particles in suspension. The DLVO interaction energy and force between coal and clay particles was calculated by some researchers [3,13]. The results indicate that clay coating is governed by the van der Waals attraction and that the double-layer interaction played a secondary role [3]. It is interesting to find that there is an energy barrier in the DLVO interaction energy curve as shown in Fig. 8, which means the energy barrier should be overcome when clay coating occurs. Therefore, the kinetic energy input (e.g. agitation) may enhance slime coating by overcoming the energy barrier, aggravating the flotation performance. In addition, agitation will improve the particleparticle collision, providing a higher attachment probability of coal and clay particles. However, so far, many works were conducted to investigate the effect of slime coating on coal flotation, which mainly focused on clay types, slurry pH and water hardness [4,11,12,14,15].

The aim of this work is to investigate the effect of agitation on the interaction of coal and kaolinite in flotation. The interaction behavior and underlying mechanism of agitation on slime coating was investigated by flotation tests, homo and hetero settling tests, turbidity meter, zeta potential distribution, focused beam reflectance measurement (FBRM)







and scanning electron microscope (SEM). The results of this research are expected to provide a more detailed description of the flotation behavior of coal-kaolinite system in the presence of kinetic energy input and give a reference for the coal flotation.

## 2. Experimental

# 2.1. Materials

The pure coal used in this study was obtained by hand-picking the coarse gravity separation clean coking-coal supplied from Huainan coal preparation plant, China. In order to obtain a purer coal, the original coal samples were separated by the heavy liquid of 1.30 g/cm<sup>3</sup> and a light fraction of the coal with the ash content of around 5% was produced. Then obtained coal were crushed, ground and used as the pure coal for the subsequent tests. Some impurities might be re-liberated during crushing and grinding, but their amount should be very low since the ash content of the light fraction is guite low. In addition, most of impurities in coal belong to clay minerals which having a similar property with the kaolinite, therefore, the re-liberated impurities would cause little effect on the tests. Kaolinite, chemical grade (purity >99%), purchased from Sinopharm Chemical Reagent Co., Ltd. was tested as the pure clay mineral in this study. The size distributions of the kaolinite and the pure coal were measured by a laser diffraction particle size analyzer (Mastersizer 2000, UK). As shown in Fig. 1 that D<sub>90</sub> of the kaolinite is 10.832 µm, which approximately matches the magnitude of clay minerals in the coal flotation [1].  $D_{90}$  of the coal is 258.862  $\mu$ m, which also matches the size of flotation feed in coal industry.

#### 2.2. Methods

#### 2.2.1. Batch flotation

Batch flotation tests were conducted using a 1.5 L Denver flotation cell at neutral pulp pH. For the scenario of extra agitation flotation, 40 g of pure coal was first added into a baffle built-in tank and mixed with 1 L tap water by a glass bar until the coal was wetted fully. Then the desired amount of kaolinite was added into the tank and stirred at five different agitation speeds of 400, 800, 1200, 1600, 2000 rpm for 10 min. After that, the suspension was immediately transferred into the flotation cell and additional 200 ml water was added at the same time. 4 µl MIBC was added for each test. No collectors were used during the whole course of flotation since the collector may improve the surface hydrophobicity of coal particles even when kaolinite-coating occurs and cover the changes caused by kaolinite-coating. The impeller speed for flotation was kept constant at 900 rpm and air flow was



Fig. 1. The cumulative size distributions of the kaolinite and coal sample.

kept at 5.23 L/min by an outside independent gas supply system. Froth products were consecutively collected at intervals of 15, 15, 30, 60, 60 s. During the collection, a metronome was placed to obtain a constant scraping speed of 30 times per min. Flotation concentrates and tailings were filtered, dried at 105 °C and weighed for ash analysis and combustible matter recovery calculation. The flotation test was evaluated by the combustible matter recovery, which was calculated by the following formula.

$$\varepsilon = \frac{\gamma_c \times (100 - A_c)}{100 \times (100 - A_f)} \times 100\% \tag{1}$$

where  $\varepsilon$  represents the combustible matter recovery, %;  $\gamma_c$  represents the yield of the concentrate, %;  $A_c$  represents the ash content of the concentrate;  $A_f$  represents the ash content of the feed. All the flotation tests were repeated for three times and the test results were averaged.

#### 2.2.2. Settling test

Settling tests were conducted in 100 ml graduated glass cylinders. Given amounts of solids (5 g for coal and 1.25 g for kaolinite) were accurately weighed and placed in the cylinders separately or mixed together. The cylinders were then filled with 100 ml distilled water and shaken briefly to mix up the slurry. The cylinders were repeatedly inverted for 20 times and left still for 14 h, and then the test photos were taken. This technique was proposed by Xu et al. [11].

#### 2.2.3. SEM analysis

The SEM analysis was conducted by ZEISS MERLIN VP Compact field emission scanning electron microscope. Firstly, the pure coal was wet sieved strictly and a size fraction between 74  $\mu$ m and 125  $\mu$ m was obtained. Then 2 g of the screened coal and 0.2 g of kaolinite were mixed in distilled water for 10 min by a magnetic stirrer. After that the suspension was passed through a sifter with pore size of 74  $\mu$ m and rinsed mildly with distilled water so that the remaining kaolinite particles in the suspension could be separated from the coal-kaolinite aggregation since D<sub>90</sub> of the kaolinite was 10.832  $\mu$ m and the maximum size was 30.200  $\mu$ m known from the laser size analysis in Section 2.1. The oversized coal-kaolinite aggregates were naturally dried in the air in order to avoid any possible change to the surface structure and then detected by SEM.

#### 2.2.4. Turbidity measurement

Mixed solids (5 g for coal and 1.25 g for kaolinite) were accurately weighed and placed into a beaker and agitated by a magnetic stirrer to make sure the solids suspend thoroughly in the water. Ten 10 ml of the stock mineral suspensions was taken and diluted to 200 ml and agitated for 10 min at five different agitation speeds 400, 800, 1200, 1600, 2000 rpm and followed by settling for 1 h, then the turbidity values of supernatants were determined immediately by the turbidity meter, VWR Model 66120-200.

#### 2.2.5. Zeta potential measurement

Zeta potential distribution was measured by CAD Zetaphoremetre IV<sup>TM</sup> (CAD, France). The Smoluchowski model was used to calculate zeta potentials from the measured electrophoretic mobility. Suspensions containing 0.5 g of coal or kaolinite were prepared separately in two jars with  $10^{-3}$  M KCl solution and conditioned by magnetic stirrer for 20 min and then left to stand for 24 h. For the zeta potential distribution measurement of pure mineral, the suspension was stirred for another 5 min and settled for 5 min, and then the upper portion of dilute fine particle suspension was taken for the measurement. In the case of mixed coal-kaolinite, the two suspensions in the jars were totally transferred into the agitation tank which was used in the flotation stage and diluted into 1 L with  $10^{-3}$  M KCl solution and then agitated at each value of desired agitation speeds for 10 min. After settling for 5 min, the upper portion of the suspension was taken for zeta potential

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