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## Full Length Article Effect of kaolinite and montmorillonite on fine coal flotation



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#### HIGHLIGHTS

#### G R A P H I C A L A B S T R A C T

- Effect of kaolinite and montmorillonite on fine coal flotation were studied.
- Interaction force between clay and coal was measured by AFM.
- No coating for kaolinite while coating for montmorillonite in deionized water.
- The force between coal and clay became strong attractive with Ca<sup>2+</sup> addition.



#### ARTICLE INFO

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#### ABSTRACT

The effect of kaolinite and montmorillonite on fine coal flotation in deionized water and calcium solution were studied through flotation experiments on a mixed coal-kaolinite/montmorillonite system in the present study, respectively. Fuerstenau upgrading curve was used to evaluate the whole flotation selectivity and the interaction force between kaolinite/montmorillonite and coal was measured using atomic force microscopy (AFM) colloidal probe technique. It was found that kaolinite and montmorillonite caused different issues in coal flotation process. In deionized water, the results show that a lower flotation recovery and worse selectivity were obtained for coal-montmorillonite system compared with that of coal-kaolinite. AFM results show that slightly jump-into-contact phenomenon was observed in coalmontmorillonite interaction while a repulsive force was always detected in coal-kaolinite interaction. The heterocoagulation between coal and montmorillonite was responsible for the low flotation recovery and bad selectivity in deionized water for coal-montmorillonite system. In presence of 0.1 mol/L calcium ion, the flotation recovery increased considerably while the selectivity of both systems was deteriorated. The jump-into-contact and high adhesion force were always found in coal-kaolinite/montmorillonite AFM force curves, indicating attractive force dominated the interaction between kaolinite/montmorillonite and coal. The electrostatic repulsive force between coal and clay particle or air bubble is fully suppressed by  $Ca^{2+}$ , aggravating the kaolinite/montmorillonite coating and also leading to an increase of flotation recovery.

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#### 1. Introduction

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http://dx.doi.org/10.1016/j.fuel.2017.01.058 0016-2361/© 2017 Elsevier Ltd. All rights reserved. The difficulty in treating coal slime in the presence of clay minerals is well known in the coal preparation plant. Clay minerals



Clays belong to the family of anisotropic phyllosilicate minerals and the basic structural elements of the clay minerals are twodimensional arrays of silicon-oxygen tetrahedral and that of aluminum or magnesium oxygen-hydroxyl octahedral [4-11]. A representative of 2:1 type phyllosilicate type montmorillonite and 1:1 type kaolinite are frequently found as gangue minerals treated in coal processing plants. Kaolinite and montmorillonite have puzzled researchers in flotation area for a very long time regarding clay coating. Several studies have been conducted to study the effect of kaolinite and montmorillonite on fine coal flotation over the last 60 years. Xu et al. [3] found that the addition of montmorillonite but not kaolinite clay depressed coal flotation by using zeta potential distribution measurement method. Slime coating by montmorillonite clay particles on coal was observed and this is responsible for the depression of coal flotation by montmorillonite clay addition. The effect of kaolinite clay addition on coal flotation is negligible. However, kaolinite coating on coal surface was observed by using SEM by Zhang et al. [12]. Oats et al. [1] studied the clay coating based on the colloid stability theory which incorporated the van der Waals and double-layer interactions. The clay coating was governed by the van der Waals attraction while the double-layer interaction played a secondary role. Honaker et al. [13] also calculated the interaction energies for the co al-montmorillonite/kaolinite system using the extended Derja guin-Landau-Verwey-Overbeek (DLVO) theory. The result showed that the energy between coal and montmorillonite was negative and attractive, whereas that between coal and kaolinite was positive and repulsive. Recently, coal flotation using saline water has attracted considerable interest because of the scarcity of fresh water and the stringent regulations on the quality of discharged water. Numbers of investigations also have been carried out to study the flotation behavior of clay particles in presence of inorganic electrolyte [14–17]. Xing et al. [18] studied the effect of calcium ion on coal flotation in the presence of kaolinite clay. Kaolinite coating phenomenon was not observed on the coal surface under the neutral condition. However, the interaction force between coal and kaolinite also changed from being weakly repulsive to strongly attractive and increased monotonously indicated by atomic force microscopy (AFM), resulting in the occurrence of the kaolinite-coating phenomenon during flotation [19]. The results among the above literatures are sometimes contradictory and the effect of kaolinite and montmorillonite on fine coal flotation is complex and required to be studied further.

The objective of this study is to explore the effect of kaolinite and montmorillonite on fine coal flotation in deionized water and 0.1 mol/L calcium solution through flotation experiments on a mixed coal-kaolinite/montmorillonite system, respectively. Fuerstenau upgrading curve was used to evaluate the whole flotation selectivity and the interaction force between kaolinite/montmorillonite and coal was measured directly using AFM colloidal probe technique. However, it must be noted that the irregular geometry and surface heterogeneity of clay particles make it difficult to use this technique to obtain reliable and reproducible force measurements for quantitative analysis and so we restrict our discussion to qualitative speculations. This procedure has been also reported by other researchers [18–21]. The results of the present research are expected to provide a more detailed description of the effect of clay particles on fine coal flotation, and also expected to provide new solution to mitigate the negative effect of clay minerals.

#### 2. Experimental

#### 2.1. Materials

Fine ultra-pure coal samples with 2.36% ash content were collected from Taixi coal preparation plant in Ningxia Province, China. Analytical grade kaolinite and montmorillonite powders with 99% purity were collected from Yongcheng City, Henan Province. Pure coal and kaolinite/montmorillonite were mixed at a mass ratio of 3:1 for flotation test. Ultra-pure graphite substrate was used to represent ultra-pure coal in AFM experiment. Deionized water was used in present study and analytical grade CaCl<sub>2</sub> was used as the background electrolyte.

#### 2.2. Methods

#### 2.2.1. Flotation kinetics experiments

Flotation kinetics tests were conducted in an XFDIII 1.0 dm<sup>3</sup> laboratory flotation machine. Kerosene and octanol were used as the collector and frother, respectively. For each test, 80 g of coal-clay samples were added into the flotation cell. The impeller rotation, collector dosage, frother dosage, and air flow rate were kept constant at 1800 rpm, 100 g/t, 100 g/t, and 0.1  $m^3/h$ , respectively. Firstly, the pulp was pre-wetted for 120 s. After the pre-wetting process, collector and frother were added to the pulp step-bystep. The collector and frother conditioning periods were 120 and 30 s, respectively. At the end of conditioning period, bubbles induced by an air pump were passed through the pulp, forming froth on the upper pulp surface. The flotation process was divided into five stages and continued for 300 s. Five flotation concentrate products were consecutively collected after 30, 60, 120, 180 s, and 300 s, respectively, during the flotation process. Flotation concentrates and tailings were filtered, dried at 80 °C and weighed for ash analysis.

#### 2.2.2. Fuerstenau upgrading curves analysis

The Fuerstenau upgrading curves were used for characterization, comparison, and analysis of the whole coal flotation selectivity in presence of clay particles. These curves have proven to be very useful in the analysis of de-ashing and desulfurization of coal flotation results [22]. The kinetic equations relating the recoveries to time of the two components in separation products, when combined together to eliminate the time parameter, provide Fuerstenau upgrading curves, which relate the recoveries of the two components in concentrate [23,24]. Commonly, the flotation kinetics of both the combustible and ash materials can be successfully described by a classical first-order equation, as shown in Eqs. (1) and (2) [25]. Therefore, the combination of the two first-order kinetic Eqs. (1) and (2) yield Eq. (3), which is a mathematical formula for approximation of the Fuerstenau curves. A new parameter,  $k = \frac{k_1}{k_2}$ , was defined as a separation selectivity index, the value of which can be obtained by fitting the flotation kinetics using Eq. (3).

$$\varepsilon = \varepsilon_{\infty} (1 - e^{-k_1 t}) \tag{1}$$

$$\varepsilon_{\mathbf{a}} = \varepsilon_{\mathbf{a}\infty} (1 - \mathbf{e}^{-k_2 t}) \tag{2}$$

$$\varepsilon = \varepsilon_{\infty} \left[ 1 - \left( \frac{\varepsilon_{a\infty} - \varepsilon_a}{\varepsilon_{a\infty}} \right)^{\frac{k_1}{k_2}} \right]$$
(3)

where  $\varepsilon$  and  $\varepsilon_a$  are the combustible matter recovery and ash recovery in the concentrate, respectively;  $\varepsilon_{\infty}$  and  $\varepsilon_{a\infty}$  are the maximum

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