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Preparation of potassium acetate intercalated metakaolinite

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ABSTRACT

Potassium acetate was intercalated into metakaolinite by grinding. The effect of grinding time, potassium acetate content and heat was investigated by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermal analysis (TG–DSC) and transmission electron microscopy (TEM). Thermal analysis revealed that the melting temperature of potassium acetate in the intercalated complexes slightly increased, suggesting that the potassium acetate was present in the interlayer space of metakaolinite. TEM showed that metakaolinite was delaminated after intercalation of potassium acetate. The intensity of the characteristic reflections of metakaolinite decreased during heating, increasing grinding times and potassium acetate content.

1. Introduction

Metakaolinite is derived from dehydroxylation of kaolinite at 450–980 °C. The alumina polyhedron sheets of metakaolinite include 4-, 5-, and 6-coordinated aluminum ions (MacKenzie et al., 2008). The properties of the alumina polyhedron sheets of metakaolinite are very similar to γ-Al_2O_3 which has porosity, high specific area and good absorbability and forms rather strong coordinative bonds with many organic compounds (Kwak et al., 2007; Noguchi et al., 2008; Wu et al., 2008).

The ability to modify clay minerals by insertion of organic guest species into the interlamellar region opens up a range of potential uses for these materials. Kaolinite can be intercalated with a small group of organic molecules. The alumina polyhedron sheets in metakaolinite, including quadricdentate and pentacoordinated aluminum ions, have higher activation than kaolinite (Wang et al., 2005). Metakaolinite can form rather strong coordinative bonds with some directly intercalated organic compounds in the interlayer space.

Not many organic compounds have the ability to directly intercalate. Their number may be extended by the so-called “displacement method”. Displacement involves the replacement of a directly intercalated species (e.g., potassium acetate) by a second organic molecule. The resulting clay–organic composites have the potential to be used as electronics products (Wang et al., 1996; Chen et al., 2001), adsorbents (Frost et al., 2002), heat resistant (Vaia et al., 1999) and catalyst supports (Sabu et al., 1999). No studies on intercalation of metakaolinite have been reported. In this paper, metakaolinite directly intercalated with potassium acetate was prepared.

2. Experimental

2.1. Materials

Metakaolinite was obtained from ENGELHARD Corporation in USA, with the chemical composition given in Table 1. Chemically pure potassium acetate and ethanol were obtained from Shanghai Experiment Reagent Co. Ltd, China.

2.2. Preparation of metakaolinite–potassium acetate complexes

The intercalated complexes were prepared by direct mixing of metakaolinite with different contents of potassium acetate (33.3, 50.0 and 60.0% of mass). A small amount of water was added to the reaction mixture during the mixing process. The pasty mixture was ground at room temperature for different times (0.5, 1 and 1.5 h). Then the samples were kept 100 °C for 3 h. Finally, the samples were washed with ethanol several times to remove excess potassium acetate, dried and stored in a desiccator.

2.3. Characterization

X-ray diffraction (XRD) measurements were carried out on a diffractometer (X' pert PRO X, Holland) using Cu Kα radiation at a generator voltage of 40 kV and a current of 40 mA. The diffractogram was scanned in the 2θ range from 3 to 40° at a rate of 2°/min.

Fourier transform infrared spectrometer (FTIR) spectra between 400 and 4000 cm^{-1} were obtained on an Nicolet 170 SX spectrometer (USA). The samples (1 mg) were finely ground for 1 min, combined with

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Table 1
Composition of metakaolinite as determined by XRF analysis (mass %).

Element as oxide	
SiO_2	53.55
$A1_2O_3$	44.02
Fe_2O_3	0.45
CaO	0.17
K_2O	0.17
Na_2O	0.26
SO_3	0.02
P_2O_5	0.46
TiO_2	0.83
SrO	0.006
ZrO_2	0.029
Loss on ignition	0.67

(100 mg) oven dried spectroscopic grade KBr and pressed into a disc for 5 min under vacuum.

Transmission electron microscopy (TEM) was performed on a JEM-2100F STEM/EDS (Japan) transmission electron microscope with an accelerating voltage of 200 kV. The samples were sliced with a microtome and the slices were placed in 200 mesh copper grids for analysis.

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TG) were carried out on a NETZSCH STA 449 C (Germany). All samples (about 10 mg) were heated under N_2 atmosphere from ambient temperature to 800 °C at the heating rate of 20 °C/min.

3. Results and discussion

To clearly illustrate the change of XRD patterns, the Gaussian function has been fitted to the characteristic reflections of the complexes. Fig. 1 shows a broad reflection extending from approximately 15° to 25° which was attributed to the overlapping of the (110), $(1\bar{1}1)$ and $(0\bar{2}1)$ reflections of metakaolinite. Two sharp reflections at 25.3° and 26.7° were assigned to (002) reflection of metakaolinite and to α-SiO_2 (Carmody et al., 2005). After the intercalation of potassium acetate, the broad reflection and the sharp reflection at 25.3° almost disappeared, whereas the quartz reflection at 26.7° still existed. It indicated that the potassium acetate was intercalated and, probably, exfoliated the metakaolinite particles. The XRD patterns also revealed that heating promoted intercalation or exfoliation of metakaolinite.

Fig. 2 shows slight changes in the XRD patterns of the intercalated complexes with different contents of potassium acetate. The intensity of the broad reflection and the sharp reflection at 25.3° slightly decreased with increasing content of potassium acetate. Thus, increasing potassium acetate content was favorable for the exfoliation and intercalation of metakaolinite.

The broad reflection extending from approximately 15° to 25° (2θ) and the sharp reflection at 25.3° rapidly decreased in intensity after 0.5 h of grinding (Fig. 3). After longer grinding time, these two reflections hardly appeared, indicating increased exfoliation.

In the FTIR spectra (Fig. 4), the absorption bands at 3434 and 1641 cm^{-1} were due to the –OH stretching and deformation vibration of the adsorbed water. After intercalation, four new bands appeared at

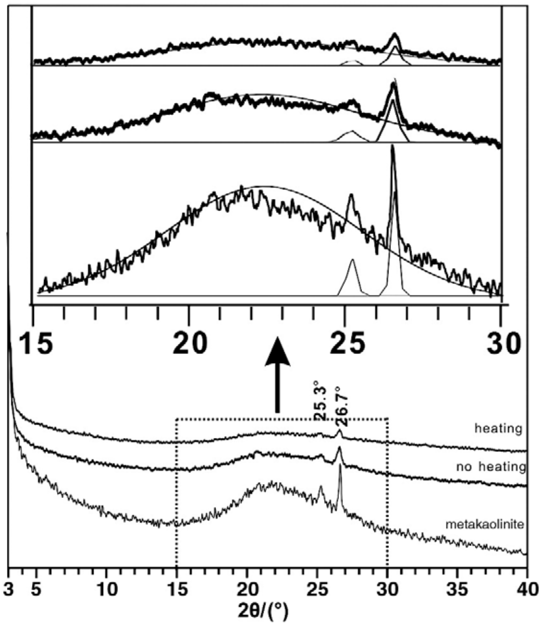

Fig. 1. XRD patterns of metakaolinite and intercalated complexes with and without heating.

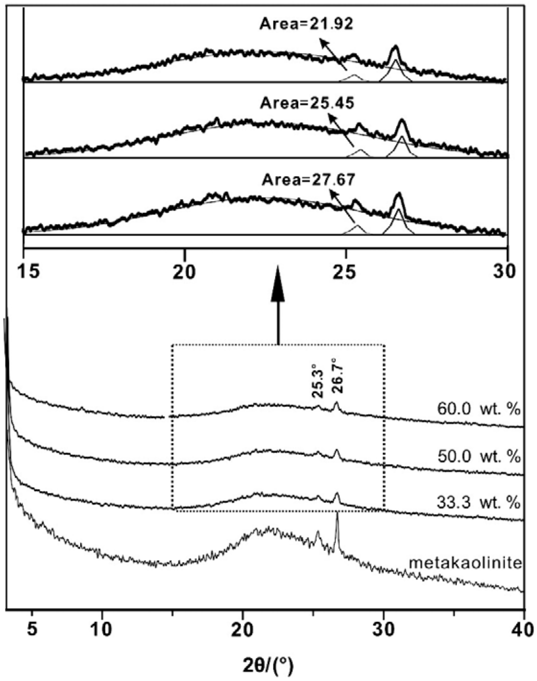

Fig. 2. XRD patterns of intercalated complexes with different content of potassium acetate.

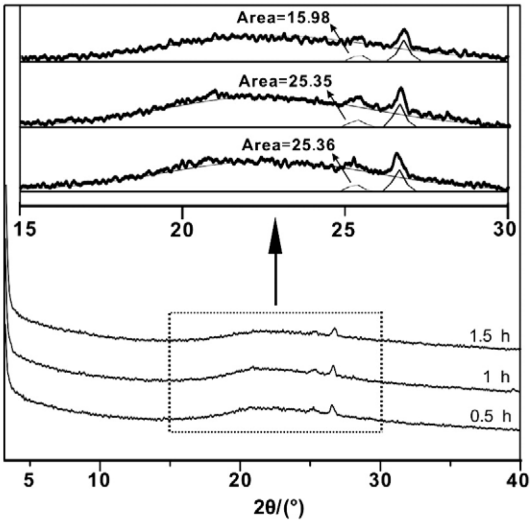

Fig.3. XRD patterns of intercalated complexes ground for different times.