

PREPARATION OF NANO-KAOLINITE

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Introduction

Since 1970's, people pay close attention to organic molecules intercalated into the interlayer of kaolinite^[1-3]. It has been found that many strong polar or polar organic molecules can enter into kaolinite between crystal layer, which makes so much novel changes for kaolinite in rheology, surface properties and structure. Those new features are very useful in the paper industry, synthetic polymers, as a sustained-release matrix elements, modification of soil and so on. It was also found that many polar molecules such as formamide, acetamide, urea, and diamide hydrated potassium acetate, etc. can damage the bonds between neighboring siloxane [$\text{H}_3\text{Si}(\text{OSiH}_2)_n\text{OSiH}_3$] and aluminum hydroxide [$\text{Al}(\text{OH})_3$] of interlayer, which simultaneously enter layer spacing of kaolinite and connect with the two hydrogen bonds forming a complex^[4-6]. The crystal spacing of kaolinite (0.7nm) may be inflated in this manner to the 1.0-1.2nm or greater. Some polar organic molecules can weak bond force between neighboring crystal layer of kaolinite in varying degrees when the layer spacing expand. In accordance with the cavitation of power ultrasonic^[7], Author and other researchers^[8-10], made a series of experiments in delamination of kaolinite by using the "kaolinite-organic complex" and the power ultrasound^[11]. It has shown that this processing methods do not use grinding medium to reduce product pollution, can greatly reduce the damage of kaolinite crystal structure to weaken the amorphous powder phenomenon. In addition, the power ultrasound equipment is simple, easy to operate, and choosing the appropriate organic molecules (such as hydrated diamide $\text{N}_2\text{H}_4\text{H}_2\text{O}$; potassium acetate

CH_3COOK , etc.) as kaolinite intercalation agent will have no environmental pollution.

Materials and methods

The samples were from interlayer of coal-seam No.4, Shanxi Formation, Lower Permian, Pinglu coalfield, Shanxi province, China. By chemical analyzing, the chemical constitution of kaolinite is SiO_2 46.38%, Al_2O_3 0.2%, K_2O 0.55%, Na_2O 0.1%, and loss on ignition 14.29%. The samples were milled pass through 40 μm mesh, organic carbon was removed by H_2O_2 (concentration: 30%) and then was repeated washed and separated by centrifugation. Finally, the section of less than 2 μm of kaolinite grains was chose for experiment.

The XRD patterns were recorded using a D/max-RA diffractometer (count time=3.5 s, step size=0.04°, CuK α radiation at 40kV and 100mA,). The infrared spectra (IR) were recorded with RFS-113V and a resolution of cm^{-1} , using KBr pressed-disc method. The morphological studies (micrographs) were performed using Philips EM 400T transmission electron microscope operated at 100V.

Results and Discussion

Intercalation of potassium acetate into kaolinite resulted in a considerable degree of expansion, from a basal spacing of 0.712 nm to 1.408nm. Washing the complex with purified water caused contraction of the basal spacing to its original value (0.712nm). The experiments show that an initial rapid expansion after 5 h of treatment and a slower expansion between 12 h and 7 days of treatment according to the formula: Intercalation rate= $[\text{I}_{(001)\text{complex}} / (\text{I}_{(001)\text{complex}} + \text{I}_{(001)\text{kaolinite}})] \times 100\%$.

K ions occupy the ditrigonal holes in the OH surface of the kaolinite layers, whereas water is

probably located between the potassium acetate layer and the OH surface of the kaolinite. This structural arrangement would favour the H-bonding between inner-surface OH groups and water and justifies the presence of new bands at lower frequencies. Electrostatic interactions between the keyed K ions and O of the inner OH groups would justify the modifications of the 3619 cm^{-1} OH-stretching band. The relatively weak bonding between the 1:1 layers of kaolin minerals favours the formation of intercalation complexes by introduction either of organic or inorganic materials with appropriate size and polarity, in the interlayer space.

It is shown that many factors affect power ultrasonic peeling of kaolinite. Firstly, the kaolinite/potassium acetate intercalation complex should be prepared. Secondly, the proper parameters of power ultrasonic should be chosen. Thirdly, the circumstance of medium (i.e. ore-slurry concentration; dispersant, pH and so on) is also an important aspect. As expected, the intercalation / power ultrasonic peeling process separates very thin lamellae from the kaolinite particles. The mean thickness of particles is 30~40 nm, and the mean diameter of particles is ~200 nm. It is actually a two-dimensional nanomaterial with bulk density of $0.06\sim 0.095\text{g/cm}^3$ and specific surface area of $30\sim 35\text{m}^2/\text{g}$.

Conclusions

(1) To make nano-kaolinite by using of power ultrasound, we should purify the raw kaolinite first and then prepare kaolinite organic complexes in advance. The unstable complex structure is in favor of peeling.

(2) Application of power ultrasound is based on the role of acoustic cavitation. The energy of peeling is from the ultrasonic, and the transient cavitation play a key role in peeling of kaolinite by power ultrasonic.

(3) The nano-kaolinite has been prepared by using of kaolinite/ potassium acetate complex under the condition of power ultrasonic. The characterization of nanokaolinite is as following: the thickness of particles is 30~50 nm, and the diameter of particles is 200 ~500 nm, the bulk density of powder is 0.06

$\sim 0.095\text{g/cm}^3$ and its specific surface area is $30\sim 35\text{m}^2/\text{g}$.

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