



Research paper

Superior dispersion properties of palygorskite in dimethyl sulfoxide via high-pressure homogenization process



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ABSTRACT

High-pressure homogenization process could well disaggregate the bundles of palygorskite (Pal) in water solution. In this paper, Pal was dispersed in dimethyl sulfoxide (DMSO) solvent via high-pressure homogenization process. The obtained Pal was characterized by infrared spectroscopy (IR), thermogravimetric analysis (TGA), field emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM). The results showed DMSO-Pal interactions involving hydrogen bonding by zeolitic water and coordinated water. The obtained Pal aqueous dispersion displayed good colloidal stability. The reported method can potentially be used in the preparation of nanohybrid material.

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1. Introduction

Palygorskite (Pal) is a natural nanorod silicate mineral with a unique chain structure. Recently, it has attracted widespread attention due to high specific surface area, special porous structure, and pronounced adsorptive, decolorizing, and colloidal properties, which have made it as a widely used adsorbent agent, as carriers for drug and catalyst, as rheological modifier, as well as an inorganic filler (An et al., 2008; Bodzay et al., 2011; Boki et al., 2001; He et al., 2011; Zhang et al., 2010). Experimental research supports that the properties of Pal are mainly affected by its bundles (An et al., 2008; Yin et al., 2010), and nanorod of Pal is usually in a tightly aggregated state. Consequently, the disaggregation of Pal from bundles to individual nanorod by a facile approach is significant to enhance its performance and extend its application.

It was found that the high-pressure homogenization process is an effective technique to disaggregate the bundles of Pal in water solution (Xu et al., 2011). The strong shear forces that can disaggregate the bundles of Pal during homogenization come from the cavitation process (Lander et al., 2000), which is intimately dependent on many factors such as the viscosity, surface tension, and vapor pressure of used dispersed medium. It is confirmed experimentally that while keeping constant of an imposed homogenization pressure with lower vapor

pressure and larger viscosity of solvent, the more energy is released from cavitation effect and the better the dispersion of aggregates (Cheng et al., 2010). Thus, the homogenization of Pal dispersion dispersed by solvents with low vapor pressure and high viscosity is preferred. Most of the available results in the literatures, however, are mainly focused on aqueous dispersions (Azoubel and Magdassi, 2010; Mauricio-Iglesias et al., 2011). Relatively little attention is paid to the disaggregation of Pal aggregates by homogenization of the organic solvent-dispersed dispersion, and there are no reports about the interactions between water molecules contained in Pal and dispersed medium under the action of strong impact forces produced by high-pressure homogenization process. In this report, the dispersion of Pal in DMSO via the high-pressure homogenization process was performed.

2. Experimental

2.1. Materials and method

Pal, with a specific surface area of 175 m²/g and a chemical composition of 64.31% SiO₂, 10.47% Al₂O₃, 0.87% Fe₂O₃, 20.41% MgO, 1.52% Na₂O, 0.13% K₂O, and 1.29% CaO, was obtained from Jiuchuan technology Co. (Jiangsu, China) and extruded for one time before use. Distilled water was used as received. DMSO (analytical grade) was purchased from Aladdin and used as received.

50.0 g of Pal was dispersed in 500 mL of distilled water and DMSO, respectively and stirred at 800 rpm for 120 min at ambient temperature. The obtained dispersion was filtered through the sieve of 74 μm (200 mesh) screen to remove quartz and other impurities. Subsequently, the collected dispersion was homogenized at 30 MPa using a high-pressure

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homogenizer (GYB-3004, Shanghai Donghua High Pressure Homogenizer Factory, Shanghai, China). The homogenized dispersions were centrifuged at 5000 rpm for 20 min, and the solid products were dried at 105 °C for 4 h. Finally, the dry products were grinded and passed through a 74 μm screen.

2.2. Characterization

The infrared (IR) spectra were obtained on a Thermo Nicolet NEXUS TM spectrophotometer. The KBr pressed-disk technique was used. The thermogravimetric analysis (TGA) was performed on a Diamond TG-DTA 6300 thermoanalyzer under a N₂ atmosphere from ambient temperature to 800 °C at a heating rate of 10 °C min⁻¹. Before analysis, all samples were dried at 105 °C for 8 h. The morphology of the samples was observed using field emission scanning electron microscopy (FESEM) (JSM-6701 F, JEOL, Ltd.) and TEM (TECNAI F20, FEI). Before the FESEM observation, all samples were fixed on copper stub and coated with gold. Before the TEM observation, the sample was dispersed in ethanol and a drop of the dispersion was taken and was placed on a copper grid.

The stability of Pal dispersion was investigated by sedimentation experiments. 2.0 g of obtained Pal was dispersed in 120 mL of deionized water with a high-speed mixer (GJB-B12K, Qingdao Haitongda Factory) at 11,000 rpm for 10 min and then transferred to 100 mL graduated cylinder, where it was allowed to stand undisturbed for some time. The sedimentation volume was read directly from the graduated cylinder at fixed time intervals.

3. Results and discussion

The IR spectra of obtained Pal sample dispersed in water and DMSO followed by homogenization at 30 MPa are shown in Fig. 1. As observed in IR spectrum of water-dispersed Pal (Fig. 1a), the characteristic peak at 3613 cm⁻¹ is attributed to the stretching vibration of –OH associated with Al³⁺ cations in dioctahedral coordination (Al₂OH) and to –OH stretching vibration of water coordinated to Mg along the nanorod (Gionis et al., 2006). The adsorption peak located at 3549 cm⁻¹ is related

to stretching vibrations of –OH of (Fe/Mg) –OH and (Al/Mg) –OH. The characteristic peaks at 3422 cm⁻¹ and 1652 cm⁻¹ are attributed to the stretching vibration and the bend vibration of water molecules, respectively, while the characteristic peak at 1452 cm⁻¹ is corresponded to the carbonate phases. Compared with IR spectrum of water-dispersed Pal, a new band at about 1318 cm⁻¹ which is assigned to the C–H bending vibration of S–CH₃ group is found in IR spectrum of DMSO-dispersed Pal (Fig. 1b). In addition, another new band at 1620 cm⁻¹ also appeared, and the characteristic peaks of –OH at 3613 cm⁻¹ and 3549 cm⁻¹ are shifted to 3618 cm⁻¹ and 3557 cm⁻¹ (Fig. 1b). This information provided evidence of the presence of hydrogen bonding interactions between DMSO and zeolitic water or coordinated water in Pal.

Pal is a clay mineral with nano-tunnel structure (0.64 nm × 0.37 nm) filled by zeolitic water and exchangeable ions. Previous works have demonstrated that some small polar molecules or dyes could be encapsulated and bound within the tunnels of Pal (Barrer and Mackenzie, 1954; Kuang and Detellier, 2004; Giustetto and Wahyudi, 2011). So, one can hypothesize that partial DMSO molecules might be incorporated into the tunnels of Pal after homogenization at 30 MPa. To confirm this behavior, the IR characteristic peaks of Pal calcined at 200 °C (Fig. 1c and d) and 380 °C (Fig. 1e and f), as well as the thermal decomposition behavior of obtained samples were discussed.

Compared with the IR spectrum of water-dispersed Pal (Fig. 1c), the adsorption bands located at 1622 and 1318 cm⁻¹ were still observed in the IR spectrum of DMSO-dispersed sample after heating the Pal at 200 °C for 4 h, but the intensities of these two bands decreased sensibly. Whereas, the C–H bending vibration of S–CH₃ group at 1318 cm⁻¹ has disappeared after calcining the Pal at 380 °C for 0.5 h (Fig. 1f), and the shapes of IR peaks were similar to that of water-dispersed sample (Fig. 1e) except for the asymmetric stretching of the Si–O–Si bridges (1196 cm⁻¹) and asymmetric stretching modes of the tetrahedral silicate sheet (984 cm⁻¹), which became broader relative to the characteristic peak of the carbonate (1452 cm⁻¹). These results indicated that the structural degradation that occurred in the tetrahedral sheet became easy at high temperature for DMSO-dispersed Pal. The disappearance of C–H bending vibration of S–CH₃ group in IR spectrum was due to the

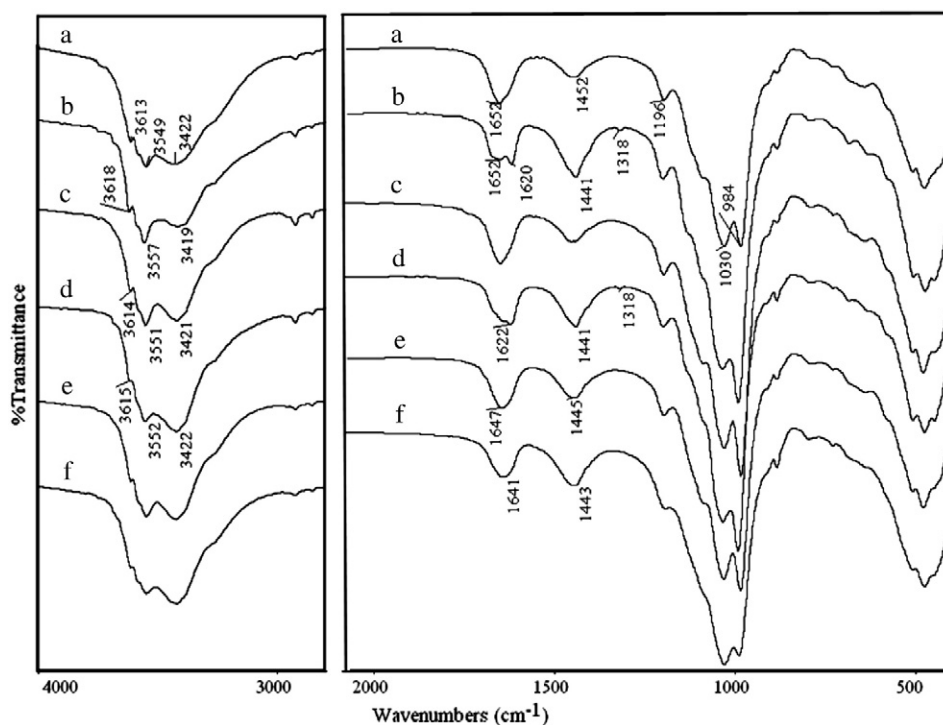


Fig. 1. IR spectra of homogenized Pal dispersed by (a) water and (b) DMSO, compared with the spectra of obtained samples heated at (c, d) 200 °C and (e, f) 380 °C.

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