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Effect of Mechanochemical Reaction on Palygorskite in Adsorption Properties†

DIFANG ZHAO*, MINGHUA LI and JINSONG XIE

Department of Chemistry & Material Engineering, Hefei University, Hefei 230022, P.R. China

*Corresponding author: E-mail: zdf@hfuu.edu.cn

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Using high-energy facility, the purified palygorskite was activated by mechanochemical treatment and preparing the urtral activated absorbent particle by the method of ball milling. With the aids of themogravimetric analysis and transmission electron microscope, the changes of thermal stability, particle size and microstructure were analyzed. The results show that the adsorption properties of mechanochemical reaction palygorskite were increased greatly, the particles were flat and minimized and the adsorbent particles has the good dispersbility.

Keywords: Mechanochemical, Palygorskite, Thermal stability, Microstructure.

INTRODUCTION

In recent years, clay minerals are widely used for there removal heavy metal ions ,dyes and other organics[1-3] from aqueous solutions because of their higher surface areas. Palygorskite has received much attention in recent years due to its versatile applications in industrial field. It usually forms compact irregular fine-fibre aggregates with high porosity, usually white with yellowish-greyish shade. For a comprehensive description of physical and chemical properties of the materials. The easiness of dispersion in water and the high adsorption capacity suggest using the material as adsorbent. However, it is difficult to significantly remove the heavy ions from water as it has low adsorption activity. A novel method of employing a mechanochemical process was used to produce ultra activated palygorskite from natural clay. Mechanochemistry uses mechanical energy to physically and chemically change the properties of matter by means of compression, shear, friction, impact, bending and extending[4]. Different from traditional methods, it is a technique of preparing nanometer particle products by taking advantage of the rotation and vibration of ball milling in the mechanochemical process to crush, grind and mix the materials.

Based on this principle, the activation process was dramatically improved by the mechanochemical effect, which led to increased adsorption performance of the products[5].

A series of experiments on milling time for developing adsorption properties and pore structures of activated palygorskite was conducted to further explore the effect of mechanochemical processing on palygorskite by hydrochloride acid activated in this work.

EXPERIMENTAL

Chemical composition of the studied palygorskite was: SiO_2 58.58, Al_2O_3 10.19, Fe_2O_3 2.79, FeO 0.15 , CaO 8.43, MgO 2.7, Na_2O 0.50, K_2O 2.35, TiO_2 0.44, MnO 0.04 and 13.26 % ash (weight %). All chemicals used in this work were of analytical grade.

Preparation of activated palygorskite samples: Palygorskite was crushed and sieved to 60-100 mesh. Then, it was air-dried and mixed with a hydrochloric acid (0.1 M) solution for 2 h. It was washed several times with deionized water and dried at 105 ºC. Then the hydrochloric acid palygorskite (HAP) was milled continuously for 5 h by a high-energy ball mill (BXQM 0.4 L, Instrument Co., Ltd. Nanjing) at room temperature with a ball:powder mass ratio of 5:1 and a rotational speed of 400 rpm (MTP).

Characterization: The microstructure of the materials was observed at room temperature on a Japan JM26700F field emissim SEM with power of 10.0 KV. After absorbed the water from the air for 5d, the purified palygorskite (PP), hydrochloride acid activated palygorskite (HAP) and mechano-

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chemical force treatment palygorskite (MTP) were followed by a thermogravimetry in a TGA Q5000 V3.5 Build 252 apparatus. The adsorption performance of the samples were determined by absorbing methylene blue from the solutions.

The heavy metal ions uptake capacities of the adsorbents were determined preliminary as follows: 1.0 g adsorbent (PP, HAP and MTP) was added into 100 mL Cu(II) ions solution with the concentration of 0.3 mg/mL in pH (9.0) and then irradiated ultrasonically for 1 h at room temperature. Then the concentration of the supernatant solutions was tested.

RESULTS AND DISCUSSION

Morphology: Fig. 1 shows (a) the TEM images of purified palygorskite, (b) the sample with 5 h grinding processes. Fig. 1(a) shows that the palygorskite ore existed as a massive solid with some fibers reaching out. Fig. 1(b) shows that the samples after 5 h grinding treatments presented free and easy and most of the palygorskite bundles are not more than 40 nm in diameter, which should be formed by fewer parallel nanorods of palygorskite. It presents that there were uniform fibers 20-40 nm in width and 1-5 µm in length for the samples after hydrochloride acid and mechanochemical treatments[6], which was in good agreement with the single crystals of palygorskite. Comparing Fig. 1(b) with 1(a), it can be seen that the bundles of the treated sample were easy to be dispersed into water by mild agitation. Thus, experiments proved that this method was efficient to separate palygorskite bundles into basic units.

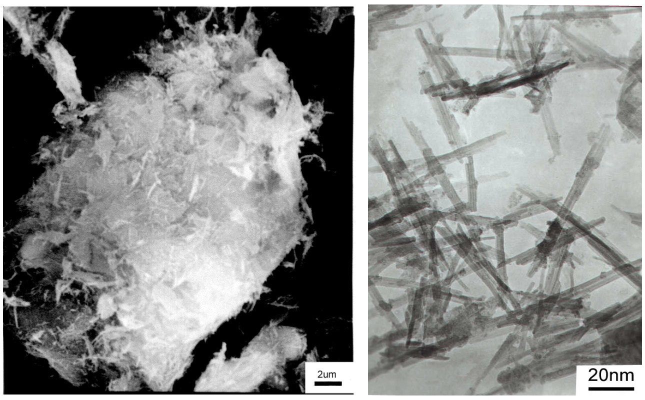

Fig. 1. TEM images of the samples of purified (a) and activated (b) palygorskite

Thermal stability: The TG and DTA curves of the purified palygorskite and 5 h mechanochemical treatment palygorskite are given in Fig. 2 for the temperature range of 25-800 ºC. Three endothermic changes are seen in the DTA curve. The temperature ranges and maximum rate temperature of these changes are shown on the TG and DTA curves in Fig. 2. The first endothermic mass loss between 25-150 ºC with a maximum rate at 120 ºC is due to the dehydration of interparticle water or adsorbed water known also as moisture. The second endothermic mass loss between 150-350 ºC with a maximum rate at 300 ºC is due to the dehydration of the zeolitic water. The third endothermic mass loss between 300-500 ºC with a maximum rate at 400 ºC is due to the dehydration of the bound water[7]. The mass losses are taken place continuously all the temperature from 25 to 800 ºC is ascribed to the high energy

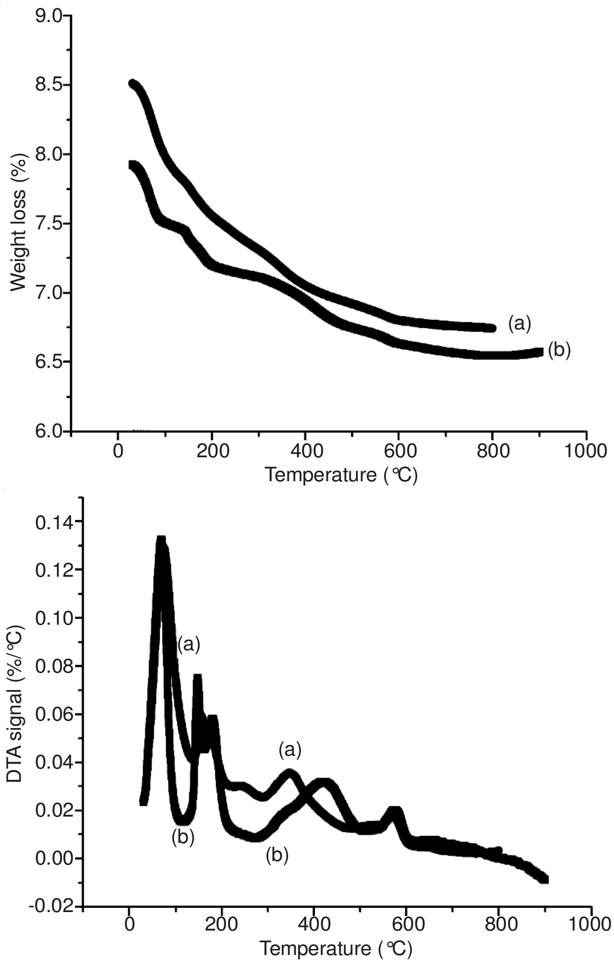

Fig. 2. TG and DTA curves of (a) purified palygorskite (PP) (b) 5 h mechanochemical treated palygorskite (MTP)

storing in the surface of palygorskite in the process of the grinding treatment.

Absorption properties: The adsorption activities of the samples are shown in Fig. 3. Compared to the mechanochemical treatment activated palygorskite (MTP), hydrochloric acid activated palygorskite (HAP) showed a slight adsorption activity to the 0.3 mg/mL, (MTAP) showed more active adsorption. More than 92 % removal rate after 1 h reaction.

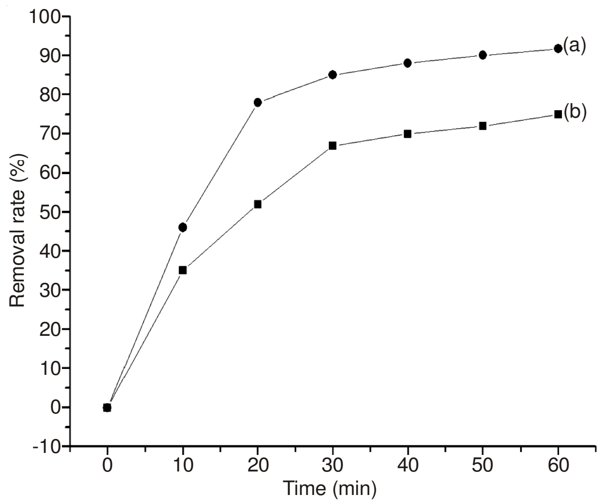

Fig. 3. Curves of removal rate on copper ion in solution with the concentration of 0.3 mg/mL. (a) MTAP (b) HAP

Conclusion

Palygorskite was studied in terms of the possibilities of its application in the process of heavy ions absorption. Palygorskite treatment with mechanochemical force revealed insignificant absorption activity. After purification it with hydrochloric acid, the adsorption activity was considerably increased and the removal rate for copper ion in solution from 75 to 92 %. It seems that mechanochemical force treatment palygorskite can be efficient absorbent for removal of the heavy metal ions in solution.

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