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Effect of sepiolite fibers addition on sintering behavior of sanitary bodies

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ABSTRACT

The work attempts to investigate the effect of sepiolite fibers addition on sintering behavior of sanitary bodies. Raw sepiolite was found to be associated with some impurities such as calcite, amphiboles and dolomite. Sedimentation method was used, and then washed with dilute HCl solution to prepare acid-treated sepiolite fibers. Green bodies with sepiolite fibers were shaped by slip casting process, then dried and sintered in an electric furnace. After cooling to room temperature, sintered samples were characterized by bulk density, bending strength and X-ray diffraction. The results showed that 2% sepiolite fiber addition could increase the bending strength of green and sintered bodies. Sintering activation energies were determined according to the bulk density results. It was found that the activation energy rose with the increase of sepiolite addition.

1. Introduction

Sanitary wares are ceramic materials made from two components: first-supporter, which usually is stoneware, and a second layer of glaze which covers the raw body and provides the whole material protection (Partyka and Lis, 2011; Reinosa et al., 2010). The increasing of densification and bending strength of raw body is good for improving the base for glaze, because, unlike tableware, sanitary ware with thick body and large size can cause cracks when forming, drying and setting. Therefore, to improve the strength of green body not only can reduce the scrap rate, but also can reduce the damage rate of sanitary wares in the transport, sale and installation process. Finally, to increase the bending strength of ceramic body can improve the tolerance and the service life of sanitary wares.

Traditionally, several methods have been used to strengthen ceramic bodies. The most commonly used method is to add nano-fibers to reinforce ceramics (Ge et al., 2012). However, high cost and health hazard of nano-fibers preparation could hinder its application. Another method to improve strength is to add aluminum or magnesium in the ingredients, so as to form mullite or other crystal phase in sintered samples at high temperature (Romero et al., 2005).

Sepiolite is a natural hydrated magnesium silicate clay mineral with a microfibrous morphology and good sorptive property, and thus finds applications in a variety of industries including cosmetics, ceramics, detergents, paper and paint (Gunay, 2011; Zhou et al., 2011; Carretero and Pozo, 2010; Eren et al., 2010; Özdemir et al., 2006; Lemić et al., 2005; Sabah et al., 2002). The abundance and availability of sepiolite reserves together with its relatively low cost guarantee continued its utilization. Some information concerning sepiolite addition in porcelain manufacturing is available (Zhou et al., 2011; Acimovic et al., 2003). Sepiolite not only has fibrous morphology, but also contains magnesium in compositions. So it was chosen to improve the strength of ceramic bodies.

The present study is devoted to determine the correlation of sepiolite addition and bending strength of green and sanitary body. The densification behavior of samples as a function of time was measured. Finally, on the basis of the data of bulk density, the sintering activation energies for different sepiolite additions are calculated.

2. Experimental procedures

2.1. Raw materials

Raw sepiolite (RS) used in this study was obtained from Jingxing (Hebei province, PR China). It was ground by a miller and sieved with a 38 μm sieve. In order to remove the impurities in raw sepiolite, purification was carried out by the sedimentation method firstly and then washed by dilute hydrochloric acid solution. The product obtained was washed with distilled water repeatedly until Cl^- ion free, and then the solid was dried at 110 °C for 2 h and ground before use. The solid obtained was called acid-treated sepiolite (AS). The chemical composition of AS was determined by Agilent ICP-MS 7000e and found to be as 59.58% SiO_2, 4.4% Al_2O_3, 1.6% Fe_2O_3, 17.0% MgO, 0.6% CaO, 1.97% K_2O, 0.3% Na_2O. The ignition loss of the AS was 14.5%.

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2.2. Preparation of slurry, slip casting and sintering

The composition of sanitary body was prepared by using industrial raw materials: 56% kaolinitic clay, 32% feldspar and 12% quartz sands. All the raw materials with mass 3.0 kg were mixed with sodium silicate (0.5%), soda (0.25%) and water (30%). The batch was put in jars and ground for 360 min, then sieved using a 100 μm sieve. The particle size of prepared slurry was measured having the residue of 0.7–0.8% on the 350 mesh (42.5 μm) sieve. Finally the slurry was aged and decayed for further use.

According to solid content in slurry, sepiolite was added in it by masses of 1%, 2%, 3% and 4%, then green bodies with a diameter of 16 mm and with a length of 300 mm were prepared by slip casting process. The body with 1% sepiolite was denoted by S1, and others with 2%, 3% and 4% were denoted by S2, S3 and S4, separately. Reference sample with no sepiolite fibers was denoted by S0. After shaping, samples were dried at 110 °C for 24 h in an oven. Dried samples were fired in an electric furnace with a heating rate of 10 °C/min at 1150, 1200 and 1250 °C for a period of 5, 10, 20, 40 and 60 min. Then, the fired samples were cooled down to room temperature in the furnace.

2.3. Characterization techniques

The fluidity and density of final slurries were measured by Ford cup (Φ4mm, 100 mL) and pycnometer (200 mL capacity). FTIR spectra were recorded on a KBr disc, using a NICOLET-380 model Fourier transform infrared spectrometer in the wave number range from 4000 to 400 cm^{-1} to study acid treatment. The disc was prepared at a ratio of sepiolite: KBr = 1:100. The crystalline phase was performed on an automated powder diffractometer (Rigaku RINT2000). Powder sample was scanned in the range 2θ = 5–80° at a scanning speed of 0.5°/min, using Ni filtered Cu Kα radiation at 40 kV and 150 mA. Finally, bending strength was measured by an electronic universal tester (CMT-6104) on 10 test pieces by a three-point loading test with a span of 50 mm.

2.4. Sintering kinetics

Sintering kinetics study to evaluate the effect of sepiolite addition on sintering activation energy of sintered samples was based on bulk density measurements (Demirkiran et al., 2008). The bulk densities of sintered samples were measured using Archimedes method.

Dried the test sample to constant mass (M_1), then boiled in distilled water for 2 h and kept for an additional 24 h at ambient temperature. After impregnation, the mass (M_2) of each sample which was suspended in water was measured. Then, taken out and wiped water droplet off the surface quickly, and measured the saturated mass (M_3) of each sample in air. The test was carried out on five duplicate samples, and the results were averaged. The bulk density, D (g/cm^3), is calculated as follows (Martín-Márquez et al., 2008):

$$D = (M_1 - M_2)/(M_3 - M_2). \quad (1)$$

The empirical equation for sintering kinetics was given as follows:

$$D = K \log t + C \quad (2)$$

where D is density, C is a constant indicating characteristic of the powders, K is the reaction rate constant and t is the sintering time. In order to calculate sintering activation energy Arrhenius equation was used.

$$K = A \exp(-Q/RT) \quad (3)$$

where Q is activation energy, R is the gas constant and T is the operated absolute temperature and A is the constant.

3. Results and discussion

3.1. Sepiolite characterization

XRD patterns and FTIR spectra of raw and acid-treated sepiolite are given in Fig. 1. XRD patterns (Fig. 1A) show that sepiolite and calcite are the main components in the RS, but with other impurities such as amphiboles and dolomite. After treatment, the characteristic peak of sepiolite at 2θ = 7.4° become strong in intensity, but the peaks distinctive for calcite and dolomite are not obvious. These observations clearly indicate that the raw sepiolite was purified by acid treatment to some extent.

FTIR spectra of sepiolite are shown in Fig. 1B. Bands in the 1200–400cm^{-1} range correspond to the silicate. The wide band centered at 1016 cm^{-1} is composed of three different bands at 1211, 1076 (shoulder), and 1016 cm^{-1}. That band is very sensitive to acid attack and changed its form, which clearly shows the textural changes in the solids. These observations are in agreement with Lazarević et al.'s (2007) results. But it is observed that bands of calcite and dolomite at 2872, 2521, 1798, 1421 and 875 cm^{-1} are absent in the AS, because calcite and dolomite were almost removed by acid-treatment. The result is in agreement with the above XRD analysis.

3.2. Property of slurry

After sepiolite was added and effectively dispersed in slurries, the density and fluidity of slurries were measured, Table 1 shows that the fluidity of slurries was getting worse with the increase of sepiolite addition, although the density had no change. The slurry almost lost fluidity when sepiolite addition was above 4%, which can probably result from

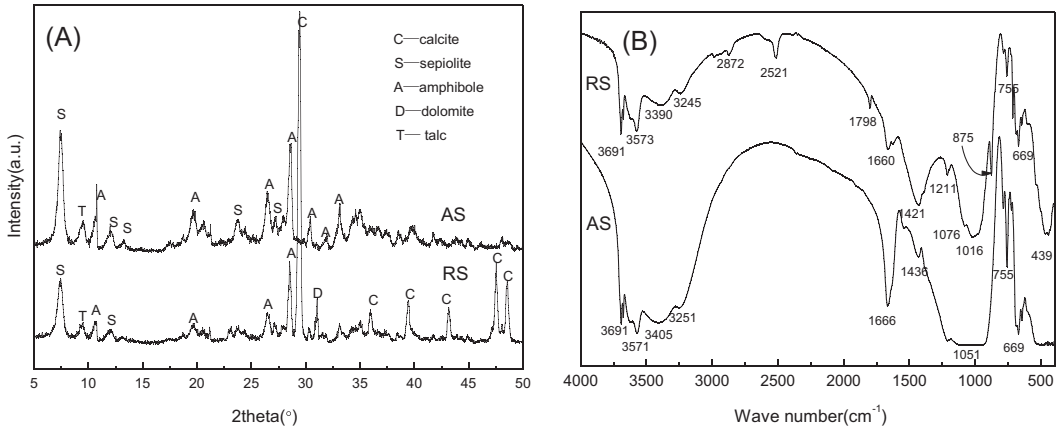

Fig. 1. XRD patterns (A) and FTIR spectra (B) of raw and acid-treated sepiolite.