Advanced Powder Technology xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Preparation and characterization of novel diatomite/ground calcium carbonate composite humidity control material

Zhibo Hu, Shuilin Zheng *, Mengzi Jia, Xiongbo Dong, Zhiming Sun

School of Chemical and Environmental Engineering, China University of Mining & Technology (Beijing), Beijing 100083, PR China

ARTICLE INFO

Article history:
Received 26 October 2016
Received in revised form 23 January 2017
Accepted 7 March 2017
Available online xxxx

Keywords:
Diatomite
Ground calcium carbonate
Calcination
Humidity control material
Mesoporous

ABSTRACT

Three kinds of novel diatomite/ground calcium carbonate composite humidity control materials were prepared with different calcination temperatures using diatomite and ground calcium carbonate (GCC) as raw materials. The microstructure and morphology properties of samples were studied by nitrogen gas adsorption, mercury intrusion porosimetry (MIP) and fractal dimensions on the basis of gas adsorption isotherms with FHH methods. Fourier transform infrared spectroscope (FTIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to characterize the mineral composition and surface properties. Furthermore, X-ray diffraction (XRD), thermo-gravimetry and differential scanning calorimetry (TG-DSC) were used to analyze the formation mechanism of composite material. It was indicated that DG750 showed the better moisture adsorption performance. The 72 h moisture adsorbed amount of DG750 reached 11.66%, 8.81% and 8.00% at 98%RH, 85%RH and 75%RH, respectively, which improved about 0.46, 0.54 and 0.53-fold as those of diatomite. The hydrophilic calcium silicate, calcium oxide and calcium hydroxide were formed in the DG750 during the calcinations process. As compared with the raw materials, the content of mesoporous component increases in the DG750, which is in favor of capillary condensation and improving moisture adsorption ability.

1. Introduction

The high-level indoor air quality is required to resident buildings as the increasing demands of energy conversation and comfortable living environment [1,2]. The indoor relative humidity is closely associated with human health and it also affects the building durability and energy consumption [3]. Some researchers have found that the appropriate relative humidity for human to live is 40% to 60% [4]. Both damp and dry environment have adverse influence on human health and livelihood. The house smells stuffy and microbial breed fast under high relative humidity. Moreover, large relative humidity fluctuations can also lead to construction damage such as dimensional changes and wooden building materials deformation. On the other hand, dry air makes furniture shrinkage, causes skin crack and respiratory diseases [3,5,6]. Humidity control is usually practiced by mechanical air-conditioning. However, this approach consumes a lot of energy [7]. The demand for energy saving and healthy living environment has promoted the development of novel indoor humidity control technologies.

Humidity control materials which are used on the walls of buildings have moisture-controllability and water vapor resistance to regulate indoor ambient air humidity [3,8]. The humidity control material was initially studied and developed in Japan from 80' in last century, which has been widely used on chemistry industry, textiles and cultural heritage protection [6]. The versatility of humidity control material is confirmed by some raw materials which include silica-gel [9], minerals [8,10,11], charcoals [12], inorganic salts [13], organic and bio-composites [14,15]. Due to zero-energy consumption, humidity control material has been regarded as one of most promising indoor air regulating technologies and attracted many researchers' attention during the past decades.

The diatomite, a natural porous nonmetallic mineral, is a kind of biogenic siliceous sedimentary rock which is mainly composed of diatoms' remains. The main chemical component of diatomite is amorphous silica derived from the deposition of single-celled aquatic algae [16,17]. Owing to excellent physicochemical properties, such as nontoxicity, light weight, unique pore structure, high porosity, excellent absorption capacity, chemical inertness, low-price and large reserves [18], diatomite is widely used as filter aid [19], adsorbent [20,21], industrial packing, catalyst carrier [22,23], porous ceramics[24,25] and environmental protection

* Corresponding author. Fax: +86 10 62339920.
E-mail address: zhengsl@cumtb.edu.cn (S. Zheng).

http://dx.doi.org/10.1016/j.apt.2017.03.005

building material [7]. Specifically, diatomite has been widely used as interior wall material as the development of diatom ooze in recent years.

Ground calcium carbonate (GCC) is processed by mechanical crush and grinding using natural carbonate minerals as raw material. Due to its superior performance in thermal stability, whiteness and rheological property, GCC is widely used in many industrial products such as paper, plastics, paint, chemical fiber, coatings, diatomite ooze, rubber and etc. [26–28]. However, the GCC does not have the humidity control capacity, and it has been used as common filler in the indoor decoration materials for improving the whiteness and hiding power.

Until now, there are few reports on the diatomite/ground calcium carbonate composite humidity control material. The purpose of this research is to prepare a novel diatomite/ground calcium carbonate composite humidity control material and investigate the influence of calcination temperature on the moisture adsorption, pore structure and surface characteristic of composite materials. The pore structure difference in these samples was studied by nitrogen gas adsorption, mercury intrusion porosimetry (MIP) and fractal dimensions on the basis of gas adsorption isotherms with FHH methods. Fourier transform infrared spectroscope (FTIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to characterize the mineral composition and surface morphology. Furthermore, X-ray diffraction (XRD), thermo-gravimetry and differential scanning calorimetry (TG-DSC) were applied to analyze the formation mechanism of composite materials.

2. Materials and methods

2.1. Materials

The diatomite (labeled as DE) used in this study was collected from Changbai deposit in Jiling Province of China and provided by Linjiang Beifeng diatomite Co., Ltd. Ground calcium carbonate (labeled as GCC) was obtained from Zhangjiajie Hengliang novel material Co., Ltd in Hunan province of China. The main chemical compositions of DE were as follows: SiO_2, 87.05%; Al_2O_3, 1.29%; Fe_2O_3, 0.42%; CaO, 0.01%. The main chemical compositions of GCC were as follows: $CaCO_3$, 98.24%.

2.2. Sample preparation

The DE and GCC were mechanically mixed homogeneously with mass ratio 3:2. The as-prepared samples were calcined under static air atmosphere at different calcination temperatures. The annealing was conducted in open ceramic crucible. The crucible furnace was programmed such that it provided heating from 25 °C to specified temperature (650 °C, 750 °C and 900 °C) by 8 °C/min rate and remained at the temperature for 3.5 h. The samples are labeled as DG650, DG750 and DG900, respectively.

2.3. Characterization and measurement

The surface microstructure of DE, GCC, DG650, DG750 and DG900 was observed using scanning electron microscopy (S-4800, Hitachi) at 3.0 KV, and the holistic morphology of samples was observed under low magnification, while the microstructure of pore channels was observed under high magnification. The transmission electron microscope (HT7700, Hitachi) at 100.0 KV was used to observe the internal pore structure. The XRD patterns were recorded via X-ray diffractometer D8 ADVANCE (Bruker, Germany) employing Cu Kα radiation at a scan speed of 4°/min in the 2θ range of 5–80°. The pore structure analysis based on nitrogen gas adsorption/desorption was carried out by a specific surface area analyzer Autosorb-iQ (Quantachrome, USA) with an automated gas adsorption. The surface area was measured using BET method. The pore volume and pore distribution of mesoporous were tested by BJH method using adsorption curve branch. The microporous surface area and microporous volume were calculated by t-plot method. Mercury intrusion porosimetry (MIP) was carried out with an AutoPore IV9500 (Micromeritics, USA), a porosimeter manufactured by Micromeritics and capable of providing a maximum mercury pressure of 414 MPa. Thermogravimetry and differential scanning calorimetry (TG-DSC) analysis were carried out on a METTLER TGA/DSC 1 SF/1382 (METTLER, Switzerland) instrument in an atmospheric environment with a heating rate of 8°C/min from room temperature to 1050 °C. Fourier transform infrared spectroscopy (FTIR) analysis was carried out on a Vertex70 infrared spectrophotometer (Bruker, Germany) using the KBr pellet technique. The FTIR spectral resolution was 4 cm^{-1}.

The main instrument used for humidity control performance experiment was high-low temperature testing chest (GDW-300, China), which could provide constant temperature environment. The humidity control experiments were conducted in the high-low temperature test chamber, and the relative humidity was controlled by saturated salt solution with potassium sulfate (98% RH), potassium chloride (85%RH) and sodium chloride (75%RH). The temperature was kept at 20 °C during the tests. Moisture adsorption performances were determined by the method of ISO 12571-2006. The samples were placed in the oven until the mass change was less than 0.001 g. All the samples adsorbed water vapor in different relative humidity for 72 h. The weighing bottles filled with samples were taken out of test chamber to record the mass change every 12 h, while during the first 6 h of adsorption experiment, the samples were weighted every 1 h due to the fast moisture adsorption rate. The moisture content of samples was calculated by the following equation:

$$M = \frac{m_t - m_0}{m_0} \times 100\% \quad (1)$$

where M is moisture content, m_0 is the initial weight of the dried sample, and m_t is the sample weight at time t.

3. Results and discussion

3.1. Moisture adsorption performance

The moisture adsorption performances of DE, GCC, DG650, DG750 and DG900 are shown in Fig. 1. The humidity control ability of the samples are represented by moisture content at any time and the maximum value of adsorbed moisture content at time of 72 h. It can be seen that GCC could hardly adsorb moisture. As shown in Fig. 1, the adsorbed moisture amount significantly increases as calcination temperature increases to 750 °C, and then, it reduces as the calcination temperature increases. The 72 h moisture adsorbed amount of DG750 reached 11.66%, 8.81% and 8.00% at 98%RH, 85%RH and 75%RH, respectively, while those of DE were only 7.96%, 5.71% and 5.24%. The moisture adsorption performances have improved about 0.46, 0.54 and 0.53-fold (Fig. 1(d)), respectively. Moreover, it should be noticed that most of diatomite/ground calcium carbonate composite humidity control materials (DG750 and DG900) have better moisture adsorption performance than that of DE.

3.2. Morphology analysis

Fig. 2 shows the scanning electron microscopy images of DE (Fig. 2(a)), GCC (Fig. 2(b)), DG650 (Fig. 2(c)), DG750 (Fig. 2(d)) and DG900 (Fig. 2(e)). As can be seen from Fig. 2, the diatom of dia-

Please cite this article in press as: Z. Hu et al., Preparation and characterization of novel diatomite/ground calcium carbonate composite humidity control material, Advanced Powder Technology (2017), http://dx.doi.org/10.1016/j.apt.2017.03.005