Experimental Study on the Hygrothermal Performance of Zeolite-Based Humidity Control Building Materials

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ABSTRACT

The hygrothermal performances of three types of zeolite-based humidity control building materials (ZBHCMs) were experimentally studied. The distributions of the pores and matrix of the ZBHCMs were visually characterized by micrographs. The saturated solution method was applied to measure the humidity control performance of the ZBHCMs. The effects of porosity, ambient temperature and relative humidity (RH) on the humidity control performance of the ZBHCMs were studied. The experimental results indicated that the humidity control performance of ZBHCMs is strongly affected by the porosity and the pore diameter. The environmental temperature and the RH have considerable influence on the adsorption performance of ZBHCMs. The desorption performance of ZBHCMs is affected more strongly by the ambient RH.

Keywords: Zeolite-based humidity control building material, Adsorption, Desorption, Pore structure.

1. INTRODUCTION

Humidity is very important to human health, and living environment, such as relative humidity indoors being too high or too low and thus having an adverse effect on our living conditions [1-4]. Additionally, an excessively high or low relative humidity (RH) of the indoor air can cause various problems in the lives of people, such as the comfort of the occupants, the storage of goods, the durability of the outer structure of the building and energy consumption [5-10]. When indoor relative humidity is maintained at 30% ~ 60%, the body feels most comfortable [11-13]. The heating, ventilating and air conditioning system is typically used to control the RH of indoor air and is a primary component of building energy consumption. A humidity control material (HCM) can adsorb or release moisture automatically without any power source or mechanical equipment due to its sensitivity to the variations of ambient temperature and RH [14-16]. Thus, the use of a HCM is of great importance to the indoor environment, energy conservation and sustainable development of the ecological environment.

Since the use of a HCM was initially proposed by a Japanese scholar [17], it has received considerable interest globally. Various HCMs were developed, including biomass HCM [18-19], inorganic HCM [20-24], and organic HCM [25-26]. Horikawa [27] studied the humidity control capacity of activated carbon from bamboo. The optimal treatment for the highest humidity control capacity was obtained. Wang

[28] prepared an acrylate-based copolymer emulsion and investigated the humidity controlling characteristics in an interior wall coating.

The research studies described above experimentally characterized the HCM on the macroscopic scale, primarily examining the original material selection, the optimal treatment and the best mixed portions of raw materials, as well as the humidity control performance measurement of the HCM. As a typical porous media, the micro-structure of HCM might have an influence on the humidity control performance of the HCM [30-33]. To the best of our knowledge, there are only a small number of reports describing research in this area. Tomita [34] investigated the humidity control ability of monolithic bimodal porous silica gel. The humidity control performance of the gel was controlled by changing its mesopore size. The presence of macrospores enabled the gel to quickly respond to a change in humidity, due to the rapid gas diffusion in the monolithic gel body. Watanabe [35] investigated the humidity control abilities of allophone, silica gel and gibbsite, which had mesopores. The results indicated that the mixture of gibbsite and clay minerals is appropriate for use as interior wall building materials, and these materials exhibit superior performance in the removal of formaldehyde.

The above studies analyzed the humidity control abilities of HCM from the perspective of pore size. The results indicated that the humidity control performance of HCM varies with mesopore size. However, detailed micro-pore

structure parameters which affected the heat or moisture migration in HCM must be further studied. Therefore, this work will prepare ZBHCMs by mixing mineral zeolite, cement, pulverized fuel ash, poplar cellulosing, and mildew preventive and antimicrobial agents. The effects of the micro-pore structure on the heat and moisture migration in ZBHCMs will be studied."

2. PREPARATIONS OF ZBHCMS

### 2.1 Optimization of the zeolite performance by calcinations

Zeolite, a siliceous sedimentary rock abundant all over the world, is receiving significant attention due to its unique combination of physical and chemical properties. Figure.1 shows micrographs of raw and calcinated zeolite taken using a scanning electron microscope (SEM). As shown in Figure. 1(a), the raw zeolite is observed to have a round plate structure with loose pore distribution. Figure 1(b) shows the micro-pore morphology of zeolite changes after performing calcinations at 500 °C for 3 hours. The pores become smaller, and the number of small pores increases. As shown in Figure 1(c) and Figure 1(d), the energy spectra of impurity elements (Fe, Al) almost disappeared after calcinations at 500 °C for three hours, which implies that the purity of zeolite increases after calcinations.
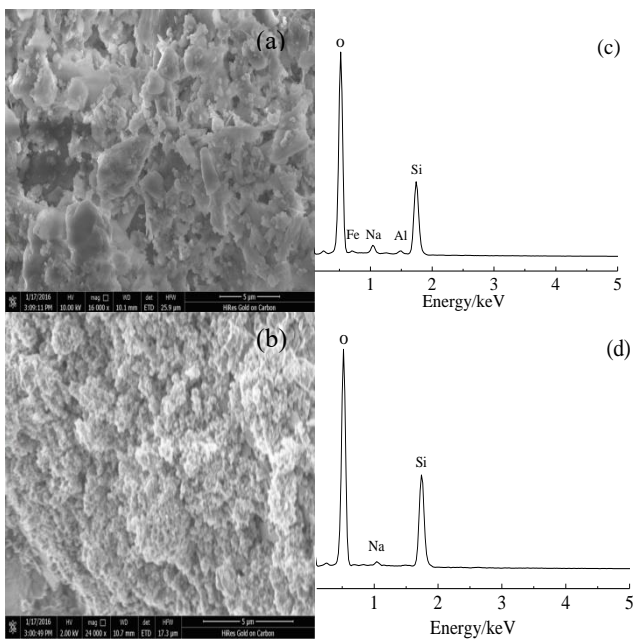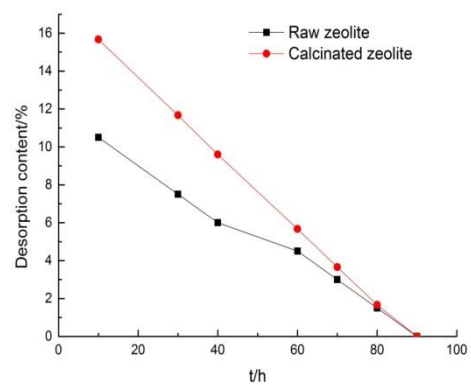

Figure 1. SEM and EDS of raw and calcinated zeolites

Zeolite that exhibits small and dense pores after calcination can improve the humidity control performance of zeolite. A comparison of the adsorption and desorption performances between raw zeolite and calcinated zeolite are shown in Figure. 2. It is evident that the adsorption content in calcinated zeolite is higher than that in the raw zeolite, which shows that calcinated zeolite has a superior ability to regulate and control the indoor RH for buildings. This ability is attributed to the fact that small and dense pores can provide higher capillary forces. Meanwhile, calcinated zeolite exhibits a slower speed in releasing excess moisture because small and dense pores promote water retention. The slow and

uniform desorption speed can create a more comfortable indoor environment for people. Therefore, calcination can improve the adsorption and desorption performances of zeolite.
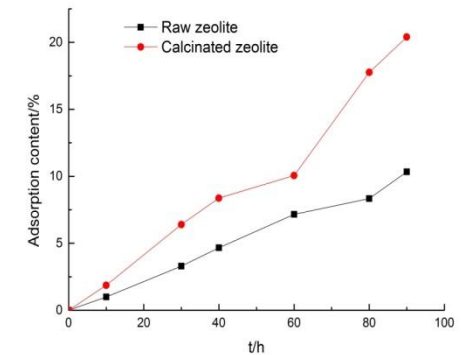
(a) Adsorption

(b) Desorption

Figure 2. Adsorption and desorption contents of raw and calcinated zeolites

2.2 Preparation of ZBHCMs

When preparing ZBHCMs, several raw materials were mixed at a certain ratio. Natural mineral zeolite was used as the main moisture modulating ingredient. It was important that the calcinations of zeolite should be performed at a certain temperature for a specific time to increase the porosity and pores connectivity of the ZBHCMs. Cement was applied for structural support. Pulverized fuel ash, an industrial waste from coal combustion, was used as a supplement in the composite. Because pulverized fuel ash is a product pollutant, using pulverized fuel can not only provide a way to utilize the waste and promote the sustainable development of ecological environment but also reduce the cement and water consumption, thereby lowering the cost of building materials. Poplar cellulosine was used as a suplemntary humidity control material. The well-developed fibrous microcellular structure of poplar cellulosine was able to maintain balance between the adsorption and desorption performances of ZBHCMs. Mildew preventive and antimicrobial substances were added to ZBHCMs for preservation. All of the abovementioned components were mixed, pulped and poured into molds. After the composites were shaped, the tested blocks were removed from the molds and maintained at a certain temperature and RH to achieve the best strength.

3. STRUCTURAL CHARACTERIZATIONS OF ZBHCBMS

The effects of micro pore structures (physical factor) on humidity control performance of ZBHCBMs were investigated. It was found that pore size distribution is directly relevant to capillary force of ZBHCBMs, while surface free energy directly affects the interaction force between water vapor in air and ZBHCBMs. So, the pore structures have a significant effect on humidity control performance of ZBHCBMs.

To elucidate the effect of the micro-structure of ZBHCBMs on its humidity control performance, micrographs and binary images of ZBHCBMs of different porosity levels in Figure 3 were obtained using microscope and image analysis methods. The proportion of the raw materials and the stirring speed were the same when preparing the blocks #1, #2, and #3, while the stirring times for the three blocks were 5 min, 10 min, and 15 min, respectively. Therefore, the micro-structure of each of the blocks was different. As shown in Figure. 3, the black portion represents the pores and the white portion represents the framework. The porosity level of the blocks decreases with the increasing of stirring time, as these raw materials become better mixed. The corresponding pore distributions were characterized using the statistical analysis method shown in Figure. 4. The pore diameter of the ZBHCBMs was found to become more uniform with an increase in stirring time.
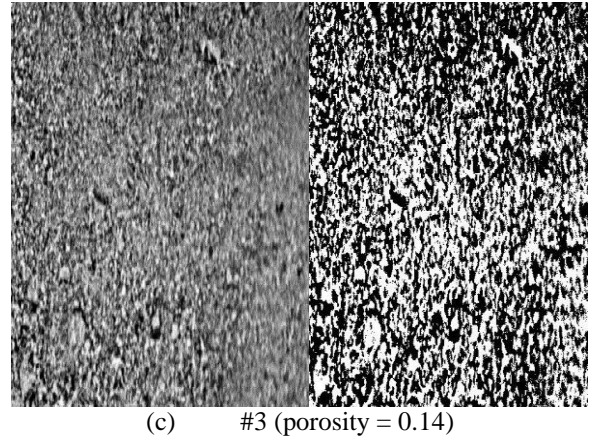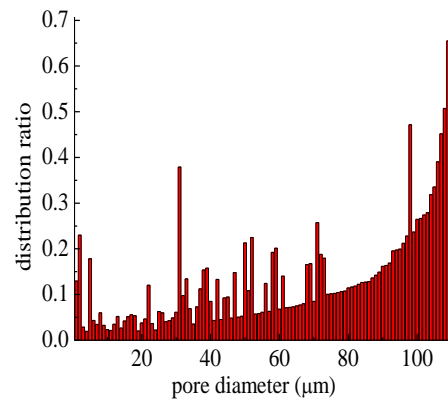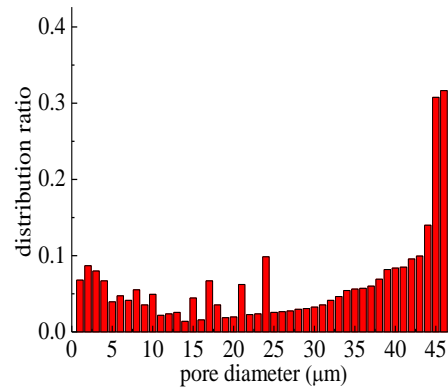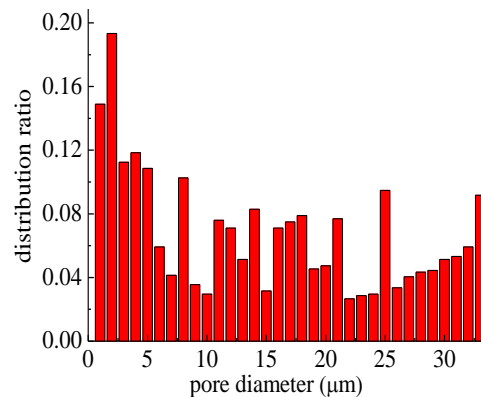

(c) #3 (porosity = 0.14)
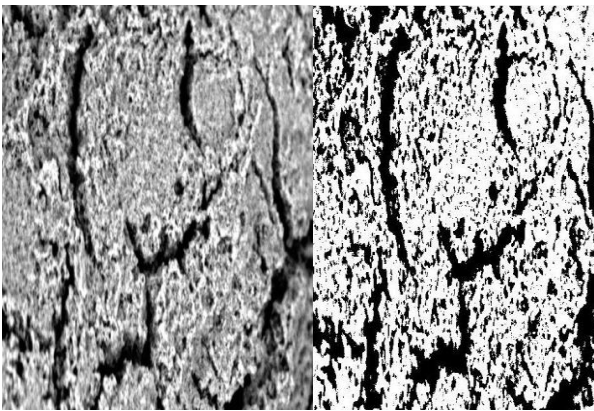
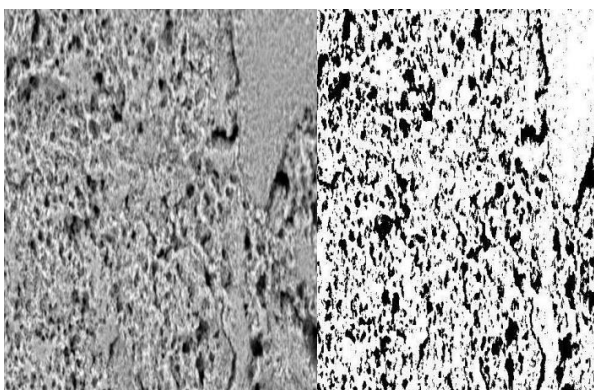**Figure 3.** Micrograph and binary image of the ZBHCBMs

(a) #1 (porosity = 0.83)

(b) #2 (porosity = 0.41)

(a) #1

(b) #2

(c) #3

Figure 4. Pore distribution diagrams of the ZBHCBMs

4. SURFACE FREE ENERGY

Surface free energy is influenced by the surface temperature, the surface shape and curvature, the surface dispersion force and the dipolar force. Therefore, the capabilities of adsorbing or desorbing moisture from ambient air are different among the different materials or the different parts of a material. The contact angles of the two blocks were measured by an optical contact angle measuring device, as shown in Figure. 5. A sample with a large porosity level exhibits a small contact angle for water. A sample with a small contact angle for water has a high surface free energy, resulting in a strong capability for ZBHCBMs to adsorb liquid water and a weak ability to absorb water.
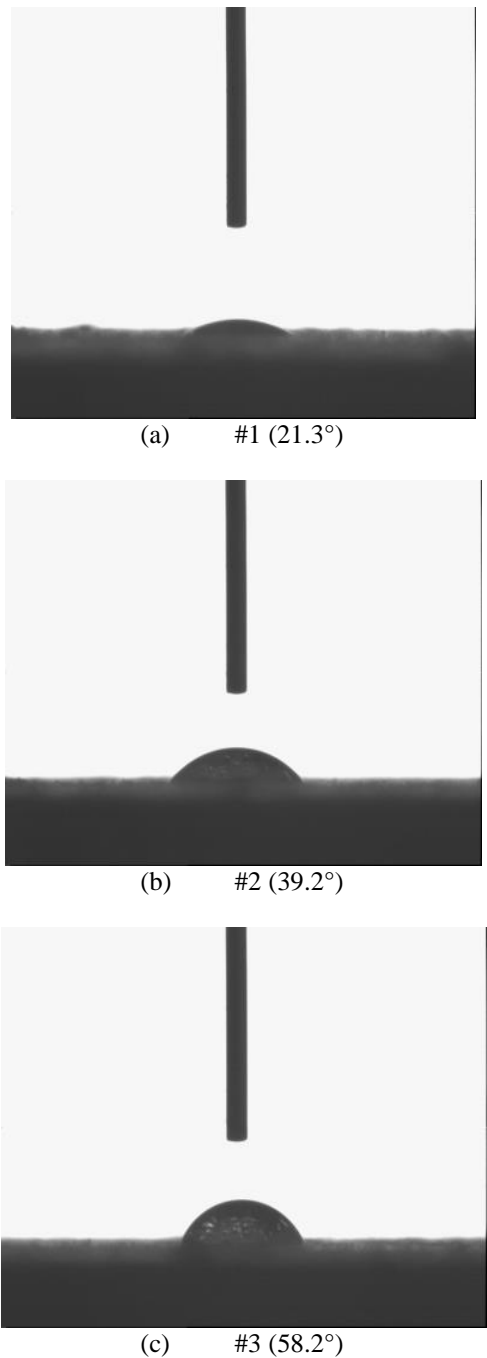

(a)   #1 (21.3°)

(b)   #2 (39.2°)

(c)   #3 (58.2°)

Figure 5. Surface contact angle of the ZBHCBMs

5. HUMIDITY CONTROL PERFORMANCE MEASUREMENTS OF ZBHCMS

5. 1 Experimental apparatus

The schematic diagram of the experimental apparatus used in the test is shown in Figure.6. The apparatus consisted of three systems: test chamber system, data acquisition system and blocks weighting system. The test chamber system consisted of a climatic chamber, testing blocks, and riser vent. The data acquisition system was comprised of a T&RH sensor (EE31-A), the data acquisition equipment and a recording computer. The T&RH sensor (with an accuracy of temperature of ± 1 °C and an accuracy of RH of $\pm 2\%$) was positioned above the blocks to monitor the temperature and humidity of the chamber. All the temperature and humidity data were recorded using an Agilent 34970A data acquisition system, which was connected to the recording computer. The weighing system for the blocks was a high precision electronic balance FA2004N with an accuracy of 0.0001 g.

The experimental setup was used to maintain the experimental temperature and RH within a certain range and to record the variations of the adsorption and desorption contents of the ZBHCMs at certain times.
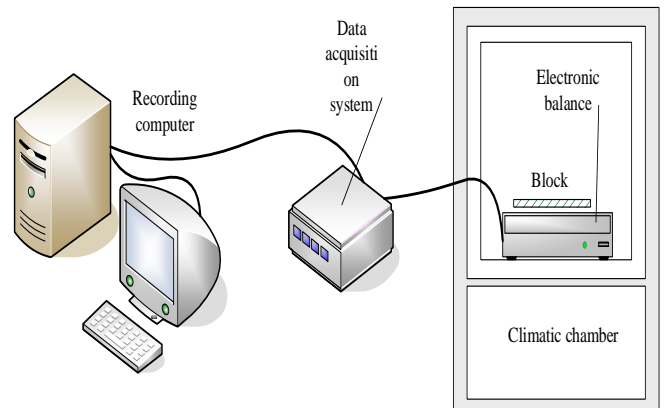

Figure 6. Schematic diagram of the experimental apparatus

5.2 Measurement of humidity controlling functions of zeolite

In order to measure the humidity controlling properties of the modified zeolite samples, increasing humidity and dehumidifying properties were measured in both dry and moist environments. Firstly, the zeolite samples were placed in glass plates or beakers, and dried. When the weight changes in the samples were less than 1% for approximately 12 hours, the blocks were considered and dried. Next, the testing samples were moved into a desiccator. The functions of these zeolite samples were measured in an artificial climate box as follows:

(a)   Dehumidifying properties: In an artificial climate box, the relative humidity (RH) was adjusted to below 30% at 25℃. Zeolite samples, previously saturated with water, were placed in the artificial climate box. During the first six hours, the testing samples were measured once every hour. Next, the samples were measured two times a day until the variations of their weights were within 1% for approximately 240 hours. The curve of RH vs. time was recorded.

(b) Increasing humidity properties: In an artificial climate box, the RH was increased to more than 85% at 25℃. After the drying process, Zeolite samples were placed in the artificial climate box. The testing samples were measured according to the same time schedule as that of the desorption experiment. However, the time when the variations of their weights were within 1% was approximately 180 hours. The curve of RH vs. time was recorded.

5.2 Testing protocol of the ZBHCMs

After the blocks were fabricated (as described in Part 2), they were placed into a drying oven (120 °C) to remove the moisture. When the weight changes of the blocks were less than 1% for approximately 12 hours, the blocks were considered as well dried. Next, the testing blocks were moved into a desiccators. After the temperature of the blocks reached ambient temperature, they were characterized in the adsorption experiment. It is important to perform waterproof treatment on the side and bottom surfaces other than the adsorption / desorption surface to model a one-dimensional heat and moisture migration environment. During the experiment, the temperature and RH of the test chamber were regulated to the required values. The initial weights of the testing blocks were measured before they were placed into the artificial environment. In the first six hours, the testing blocks were measured every hour. Next, the blocks were measured two times a day until the variations of their weights were within 1% for approximately 240 hours. In the desorption experiment, the saturated adsorption testing blocks were moved into the pre-set relatively low humidity environment. The testing blocks were measured according to the same time schedule as that of the adsorption experiment. However, the time when the variations of their weights were within 1% was approximately 180 hours.

The adsorption or desorption content of the ZBHCMs was calculated using the following formula:

$$C = \frac{W - W_0}{W_0} \times 100\% \tag{1}$$

where W is the moisture adsorption or desorption weight of the ZBHCM, and W_0 is the initial weight of the ZBHCM.

5.3 Characteristic values and properties of ZBHCM

Most Equilibrium isothermal moisture absorption relation of building material can be expressed as follows:

$$C = a\phi^b + c\phi^d \tag{2}$$

where, ϕ is ambient relative humidity, a, b, c, d are fitting coefficients.

The experimental facility is shown in Figure. 7, which consisted of a HS-150L constant temperature humidity chamber, high precision electronic balance FA2004N with an accuracy of 0.0001 g. The temperature was set as 20 ℃。 ZBHCM was placed into a drying oven (120 ℃) to remove the moisture. When the weight changes of ZBHCM was less than 1%, it was moved into a desiccator. After the temperature of the blocks reached ambient temperature, measuring the moisture content variation of ZBHCM can

started when the ambient relative humidity are 33% and 85%. Finally, the characteristic values and properties of ZBHCM were obtained by fitting these experimental data which is shown in Figure. 8.
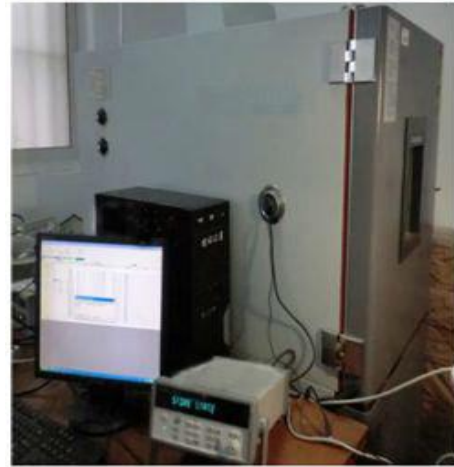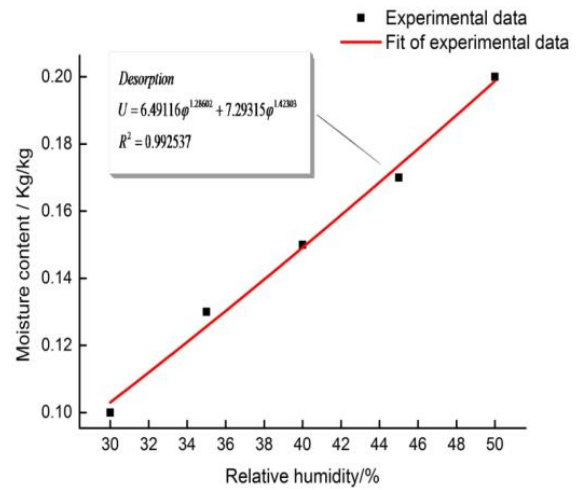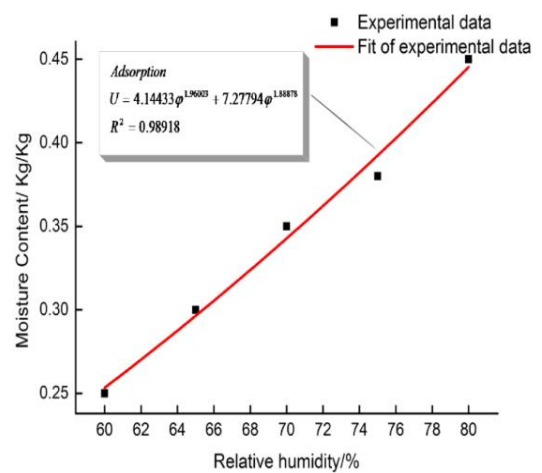

Figure 7. Moisture absorption test facility

(a) Adsorption

(b) Desorption

Figure 8. Hygroscopicity Figureure

6. EXPERIMENTAL RESULTS AND DISCUSSION

6.1 Effects of the pore structure and the surface free energy on the humidity control properties

Figure. 9 illustrated the adsorption and desorption contents of the ZBHCMs with different porosity levels. The temperature and RH of the adsorption process in the test chamber were set as 20 °C and 85%, respectively, while the temperature and RH of the desorption process in the test chamber were set as 20 °C and 33%, respectively. As shown in Figure 9, the adsorption speed curves in the first six hours exhibit sharp slopes due to the high concentration gradient of the moisture between the chamber environment and within the testing blocks. The adsorption speeds decrease with the increasing of time in the testing blocks. The ZBHCMs of lower porosity levels were observed to be able to adsorb more moisture compared with the other two types of ZBHCMs. The adsorption content of #3 (porosity = 0.14) is approximately two times higher than that of #1 (porosity = 0.83). During the desorption process, the rate of desorption decreases with an increase of time in the testing blocks. However, a higher amount of moisture remains in the testing blocks of relatively small porosity levels (#2, #3). The ZBHCM with small porosity and tiny pore diameter exhibits a small surface contact angle (large surface free energy), which can exhibit a large capillary force, thereby enabling water retention in the testing blocks. Hence, when designing ZBHCMs, there is a reasonable pore size for superior humidity control performance with reduced water retention.

6.2 Effect of the ambient temperature on the humidity control properties

Figure. 10 shows the effect of the chamber temperature on the humidity control performance of a ZBHCM. The porosity of the testing block is 0.21 (#2). The RH of the adsorption process in the test chamber was set to 85%, while the RH of the process of desorption in the test chamber was set to 33%. The trend of adsorption / desorption speed is similar to that in Figure 9. The humidity control performance increases with an increase in ambient temperature, decreases with a decrease in ambient temperature. As the chamber temperature increases, the saturated water vapor concentration in air increases, thereby causing the adsorption content of ZBHCM to increase; that is, the increase in the moisture concentration gradient between the chamber environment and within the testing block. Adsorption is a process of the condensation of water vapor in air which will release the condensation heat. However, chamber temperature has little influence on the desorption performance of the ZBHCM because of the pore capillary effect of ZBHCM. Kinetic energy of water vapor molecules adsorbed in ZBHCM increases in a low humidity environment with an increase in ambient temperature, so the ability to eliminate the water vapor intermolecular constraint increases. Moreover, desorption is a gasification process for water vapor to struggle to be released from ZBHCM which absorbs the ambient heat.
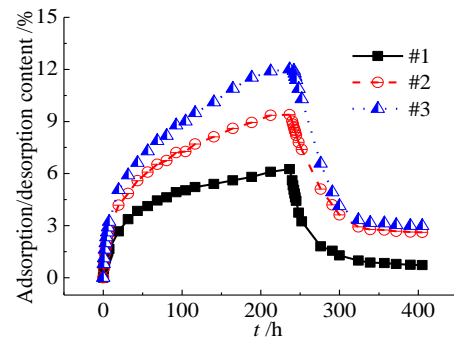

Figure 9. The effect of porosity on the humidity control performance of the ZBHCMs
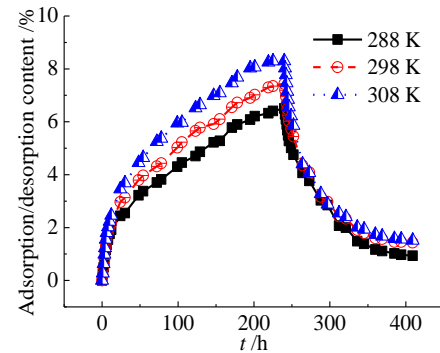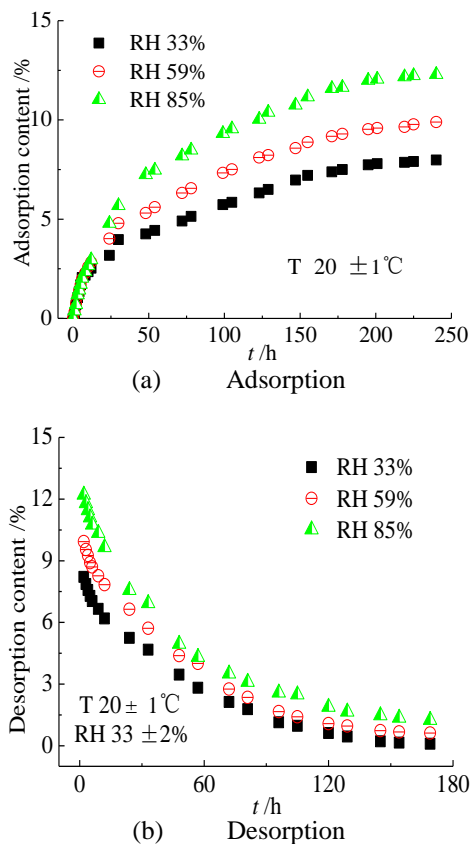

Figure 10. The effect of ambient temperature on the humidity control performance of the ZBHCMs

6.3 Effect of the ambient RH on humidity control properties

Figure 11 presents the effect of ambient RH on the humidity control properties of a ZBHCM. The porosity of the testing block is 0.11 (#3). The temperature of the adsorption and desorption processes in the test chamber were set to 20 °C. The maximum adsorption content was approximately 12% of its own weight when the RH was 85%, and the minimum adsorption content was approximately 7.5% of its own weight when the RH was 33%. The maximum desorption content was approximately 9% of its own weight when the RH was 85%, and the minimum desorption content was approximately 6.5% of its own weight when the RH is 33%. They are all caused by the RH gradients inside ZBHCMs and in air. A larger RH implies more moisture in the chamber environment. Therefore, a higher concentration gradient of moisture between the chamber environment and within the testing block can promote larger adsorption/desorption content in the testing block. The time that the adsorption becomes stable is always longer than the time it takes for the desorption to become stable, which indicates that the ZBHCM retains a certain amount of water or water vapor. Therefore, ZBHCMs must be treated to prevent the growth of bacteria and mildew. The adsorption content is always more than the desorption content, which agrees with the pore capillary effect of ZBHCM.

Figure 11. The effect of ambient RH on the humidity control performance of the ZBHCMs

7. CONCLUSIONS

The experiments were performed investigating the hydrothermal performance of ZBHCMs. The effects of porosity, ambient temperature and RH on the humidity control performance of ZBHCMs were studied. The results indicated that the adsorption performance of a ZBHCM is strongly affected by the environmental temperature and RH. The desorption performance of a ZBHCM is more affected by the ambient RH, rather than the temperature. In addition, the micro-structure of a ZBHCM has a considerable influence on its humidity control performance. The ZBHCMs with small porosity and tiny pore diameter has higher adsorption capability as well as greater water retention in the blocks. Therefore, there is a reasonable pore size for superior humidity control performance with reduced water retention.

ACKNOWLEDGMENT

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NOMENCLATURE

C	the adsorption or desorption content of the ZBHCMs , %
W	the moisture adsorption or desorption weight of the ZBHCM , kg
W_0	the initial weight of the ZBHCM, kg
a	fitting coefficient
b	fitting coefficient
c	fitting coefficient
d	fitting coefficient

Greek symbols

ϕ	ambient relative humidity thermal, %