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Effect of pore size on the performance of composite adsorbent

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Abstract Three kinds of commercial silica gels with pore size of 2–3, 4–7 and 8–10 nm respectively are used for preparing composite adsorbents by soaking them into the aqueous solution of calcium chloride. The test result indicates that both the water uptake and adsorption rate of composite adsorbents prepared from 4–7 and 8–10 nm silica gels improve greatly compared to pure silica gels, but they do not for 2–3 nm silica gels. The silica gel with pore size of 2–3 nm is not suitable for preparing the composite adsorbent by impregnation method due to the pore blockage because of the small pore size. The SCP and COP of the adsorption chiller with sample SA50 are 128.3 Wkg^{-1} and 0.27 respectively at the hot source temperature of 90 °C, which are largely superior to that of SA0. Hence using the composite adsorbent instead of the pure silica gel can reduce the size of the adsorption chiller.

Keywords Refrigeration · Adsorption system · Composite · Adsorbent · Silica gel · Calcium chloride

1 Introduction

Heat driven adsorption refrigeration systems have drawn considerable attention due to the lower environmental impact and large energy saving potential as the systems neither use ozone depleting gases nor the fossil fuel and electricity as driving source. These systems allow utilization of the huge amount of industrial waste heat as well as the low grade solar energy. In addition, they have no moving parts, and are also less sensitive to shocks and the installation position. Although adsorption systems offer all the benefits listed above, they usually also have the drawbacks of low coefficient of performance (COP) and low specific cooling power (SCP). There are two major meanings for the improvement. One is to enhance heat and mass transfer mechanically between adsorbent bed and heat transfer surface, such as using the extended heat transfer surface. The other is to develop new adsorbent material having high adsorption rate.

The adsorption capacity of silica gel hardly exceeds 40 % of its own weight. Therefore there is a need for developing new adsorbents of high adsorption capacity and capable of being regenerated at low temperatures (Chen et al. 2010; Daou et al. 2008). Recently, a new family of composite sorbents called selective water sorbents has been presented for sorption cooling and heat pumping (Aristov et al. 2002). It is based on a porous host matrix (silica, alumina, etc.) and an inorganic salt ($CaCl_2$, LiBr, $MgCl_2$, etc.) impregnated inside pores (Maggio et al. 2009; Dawoud and Aristov 2003; Gordeeva et al. 2006; Saha et al. 2009; Simonova and Aristov 2006). Comprehensive experimental studies of the physicochemical properties and some application researches of the composite adsorbents have been reported by Aristov (2007a, b), Liu and Wang (2003), Zhang and Qiu (2007) and Zhang et al. (2005). All their studies show that the composite adsorbent has a higher adsorption capacity and can be regenerated with a lower temperature. Aristov has been the precursor in developing composite adsorbents by impregnating hygroscopic promoter salts (inorganic salts in general) into the pores cavities of host matrices. The experimental result showed that the water uptake of SWS-1L ($CaCl_2$-in-silica gel) was about 0.75 gg^{-1} at the temperature of 28°Cand the pressure of 23.4 mbar while for silica gel it was just 0.1 gg^{-1}. Daou et al. (2006) investigated the

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composite adsorbents of silica gel and calcium chloride. The experimental results showed that the composite adsorbents obtained with the solutions at concentration greater or equal to 40 % yielded a maximum capacity of adsorption approximately doubling their own weight and quadrupling that of the pure silica gel. Zhang and Qiu (2007) studied the moisture uptake curves and dynamic adsorption curves in silica gel–calcium composite adsorbents by using the thermal gravimetrical method in a relative vapor pressure range from 0.5 to 0.9 at different temperature levels of 25, 35 and 45 °C, respectively. The experimental study showed that the saturated water uptake increases with an increase in $CaCl_2$ content, however, the presence of salt inside the pores will slow the rate of water vapor transport, which results in a poor mass diffusion coefficient. Glaznev et al. (2011) reported a synthesis of novel composite sorbents “$CaCl_2$ confined to a meso-structured silicate SBA-15″ with variable properties which appear to depend on the SBA pore size (8.1 and 11.8 nm). One of the most important findings was a tuning of the hydration temperature of the confined salt as it appeared to depend on the pore size of the host matrix.

Although impregnation of inorganic hygroscopic salt into the porous matrices has been extensively investigated, the question of how big the size of the pore diameter of silica gel should be for the synthesized adsorbent to have significantly better adsorption capacity in comparison with that of the matrix host used still need further research. This paper is intended to answer this question by presenting a study of the sorption properties of composites obtained by impregnating silica gels of different pore diameter size with calcium chloride at various contents of $CaCl_2$ ranging from 0 up to 50 %. Three different commercial silica gels with pore size of 2–3, 4–7 and 8-10 nm respectively are chosen as the matrix host.

2 Experimental

2.1 Materials

Main raw materials, used in this study, included silica gels, $CaCl_2$ and deionized water. The commercial silica gels were purchased from Qingdao, China. The parameters of the silica gels were shown in Table 1.

2.2 Samples preparation process

The composite adsorbents were synthesized by filling the silica gel pores with aqueous solutions of calcium chloride with different concentrations (Daou et al. 2008; Gong et al. 2010; Ji et al. 2007; Tokarev et al. 2002). The silica gel was first dried at 120 °C until its mass remained constant and cooled down to ambient temperature in a vacuum chamber. Secondly, six different $CaCl_2$ aqueous solutions with 0, 10, 20, 30, 40 and 50 % in mass concentration were prepared by dissolving dry $CaCl_2$ into deionized water. The solutions were cooled down to ambient temperature under airproof condition for avoiding water absorption. The dry silica gel was then impregnated with an aqueous solution of calcium chloride at 25 °C for 48 h until all the pores were filled. Next the sample was taken out of the solution and laid on the shelves. After that, the sample was washed once with deionized water quickly in order to remove the salt on its surface (Daou et al. 2006). Finally the composite sample was dried at 120 °C in a vacuum drier until the sample weight remained constant (Zhu et al. 2006). Eighteen composite samples were prepared by the above method. Table 2 presents the contents of $CaCl_2$ in the composite samples obtained. The symbol SA, SB and SC denote mesoporous, B type and microporous silica gels respectively. In Table 1, the symbol SA0 denotes the composite adsorbent obtained by filling the microporous silica gel pores with aqueous solutions of calcium chloride with 0 % in mass concentration. The symbol SA10 denotes the composite adsorbent obtained by the impregnation of the aqueous solutions of $CaCl_2$ with 10 % in mass concentration into the pore volumes of the microporous silica gel, and so on.

2.3 Experimental section

The measurements of composite adsorbents performances including adsorption amounts and adsorption rate were conducted in thermostat–humidistat chamber (with uncertainties of ±0.5 °C for temperature, ±5 % for humidity) (Zhang and Qiu 2007; Gordeeva et al. 2002). The mass of every prepared adsorbent sample placing on the stainless steel screen in the thermostat–humidistat chamber was 50 g. The testing condition in thermostat–humidistat chamber was set at the temperature of 40 °C and relative

Table 1 Parameters of silica gels

Designation of silica gel	Particle diameter/mesh	Specific surface area/m^2g^{-1}	Pore size/nm	Pore volume/ ml g^{-1}
Mesoporous silica gel	20–28	300–400	8–10	0.75–1.0
B type silica gel	20–28	450–600	4–7	0.5–0.8
Microporous silica gel	20–28	≥600	2–3	0.35–0.45

Table 2 $CaCl_2$ content of composite adsorbents

Sample designation	$CaCl_2$ content/wt%	Sample designation	$CaCl_2$ content/wt%	Sample designation	$CaCl_2$ content/wt%
SA0	0	SB0	0	SC0	0
SA10	7.26	SB10	3.61	SC10	3.14
SA20	15.38	SB20	14.09	SC20	5.18
SA30	19.87	SB30	21.6	SC30	11.45
SA40	32.14	SB40	26.74	SC40	17.9
SA50	37.75	SB50	41.3	SC50	23.08

humidity from 20 to 60 %. The amount of water adsorbed by the samples was expressed as uptake per 100 g composite adsorbent. An electronic scale having an accuracy of ±0.001 g was used for weighting the mass variation. It usually took 2–5 h to reach the adsorption equilibrium, but for refrigeration, the adsorption time of 2 h was enough.

The experimental procedure was as follows

①. Put the sample into drier at the temperature of 90 °C for 2 h at desorption stage;

②. Set the temperature and humidity of thermostat–humidistat chamber to 40°Cand the needed value respectively;

③. Put the sample into thermostat–humidistat chamber for 2 h and record the weight variation at adsorption stage;

④. Change the humidity of thermostat–humidistat chamber and repeat procedure ①, ② and ③.

3 Results and discussion

3.1 Adsorption isotherms

Adsorption isotherms of 18 samples are displayed in Fig. 1a, b at the adsorption temperature of 40 °C and adsorption time of 2 h. Three conclusions can be drawn from Fig. 1a, b.

The first conclusion is that the water uptake of composite adsorbents increases monotonously with the increase of humidity for all samples. The water uptakes of samples SA50, SB50 and SC50 are 15.7, 15.64 and 7.06 g/100 g respectively at the humidity of 20 %, while for the humidity of 60 %, the water uptakes are 42.33, 47.33 and 28.02 g/100 g, increase nets of 26.63, 31.69 and 20.96 g respectively compared to that for the humidity of 20 %.

The second conclusion is that the water uptakes of composite samples prepared from the mesoporous and B type silica gels are always bigger than that of substrate silica gels, but for samples prepared from the microporous silica gels, they do not, as shown in Fig. 1a, b. The water uptakes of substrate silica gels SA0, SB0 and SC0 are 2.0, 1.94 and 4.73 g/100 g respectively at the humidity of 20 %. When impregnated with an aqueous solution of calcium chloride with 20 % in mass concentration, the water uptakes of composite samples SA20, SB20 and SC20 can be as high as 8.42, 6.97 and 4.69 g/100 g, net increase of 6.42 g, 5.03 g and −0.04 g respectively compared with substrate silica gels. The net increase of the water uptakes of composite samples SA20, SB20 and SC20 are 17.5 g, 15.95 g and −5.6 g respectively compared with substrate silica gels at the humidity of 50 %.

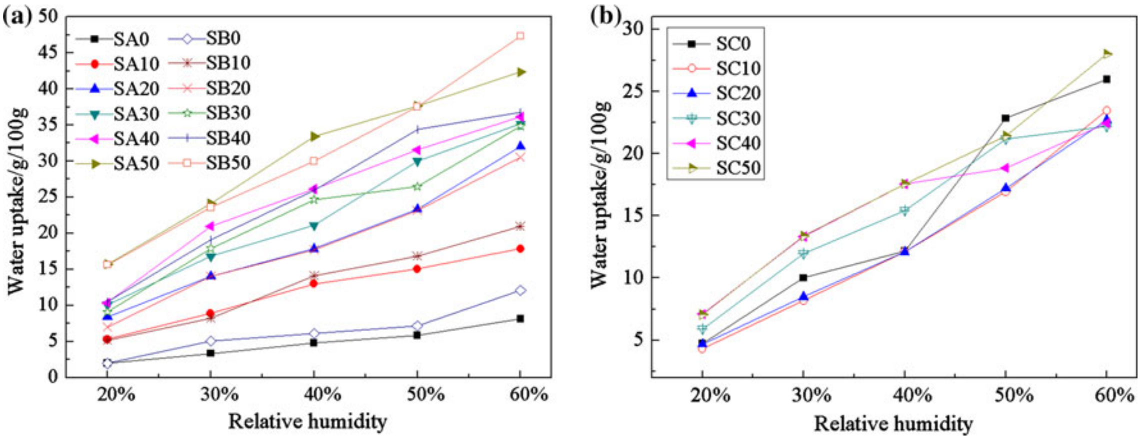

Fig. 1 Adsorption isotherms. **a** Mesoporous and B type composite adsorbents, **b** Microporous composite adsorbents

Apparently from Fig. 1 the water uptakes of sample SC0 are bigger than that of samples SA0 and SB0. But when impregnated with an aqueous solution of calcium chloride, the composite samples prepared from substrate silica gels SA0 and SB0 have a better adsorption capacity in comparison to the substrate silica gels, however, the composite samples from substrate silica gels SC0 do not show much better adsorption capacity than the substrate silica gels. This indicates that the commercial silica gel with pore size of 2–3 nm is not suitable for preparing the composite adsorbent by impregnation method, but the commercial silica gels with pore size of 4–7 and 8–10 nm do. This can be explained by the fact that the impregnation of the commercial gels with pore size of 4–7 and 8–10 nm with an aqueous solution of calcium chloride does not cause the pore blockage because of the bigger pore size. But for the commercial gels with pore size of 2–3 nm, the impregnation of calcium chloride may cause partial pore blockage due to the smaller pore size, thus this blocks mass transfer of vapor.

The third conclusion is that the water uptakes of composite samples prepared from the mesoporous and B type silica gels increase with the increase of concentration of calcium chloride used for the impregnation of silica gels, however, they do not for samples prepared from the microporous silica gels. The contents of $CaCl_2$ in the composite samples are listed in Table 2. The water uptakes of composite samples SA0, SA20 and SA50 are 5.86, 23.36 and 37.65 g/100 g respectively at the humidity of 50 %. It can be noticed that the sample SA50 is able to take up 6.42 times as much water vapor as the pure silica gel can take up. At the same humidity, the water uptakes are 7.2, 23.15 and 37.55 g/100 g respectively for samples SB0, SB20 and SB50. Observing the profiles of other samples in Fig. 1a it can be also noticed that the water uptakes increase with the increase of $CaCl_2$ contents in samples. The water uptakes are 22.83, 17.23 and 21.43 g/100 g for samples SC0, SC20 and SC50 respectively, which does not reveal the water uptakes increase with the increase of $CaCl_2$ contents in samples, as shown in Fig. 1b.

3.2 Adsorption rate

The high performance adsorbents demand not only big adsorption amounts but also fast adsorption rate. Figure 2 describes the water uptake as a function of time for different humidity. It can be seen obviously from Fig. 2 that the water uptakes increase with the increase of both time and humidity for all samples. A steep increase of water uptakes for all samples can be observed at this early stage of adsorption from Fig. 2.

The adsorption rate of microporous composite samples does not increase with the increase of $CaCl_2$ contents in the samples, while for mesoporous and B type composite samples the adsorption rate increase with the increase of $CaCl_2$ contents in the samples especially when the adsorption time is larger than 20 min. In Fig. 2a, the water uptakes of sample SA0 are 1.95, 1.96 and 1.99 g/100 g at the humidity of 20 % when the adsorption time are 30, 60 and 90 min, reaching 97.89, 98.17 and 99.67 % of the two hours water uptake respectively, while for sample SA50 the water uptakes are 10.29, 13.95 and 15.02 g/100 g at the same adsorption time and humidity, corresponding to the percentages of 65.57, 88.87 and 95.69 % of the two hours water uptakes respectively. This indicates that it almost takes 30 min for sample SA0 to reach adsorption equilibrium, while for sample SA50 it takes 90 min still not to reach adsorption equilibrium. In Fig. 2b, it takes 30, 60 and 90 min for sample SA0 to reach 93.18, 94.86 and 97.14 % of the two hours water uptake respectively at the humidity of 30 %, while for sample SA50, at the same adsorption time and humidity, the percentages of 58.31, 84.11 and 95.45 % of the two hours water uptake are reached. The above analyses show that it takes short time for pure silica gel to reach adsorption equilibrium. For composite adsorbents they adsorb faster than pure silica gels, but they still need long time to reach adsorption equilibrium due to the higher adsorption capacity resulting from the impregnation of $CaCl_2$, that is to say, the impregnation of $CaCl_2$ in silica gel extends the adsorption equilibrium time. This is likely to contribute to the fact that the water absorption by silica gel belongs to physical adsorption. The water uptake is small, so the temperature in the pore changes little due to small adsorption heat release, resulting in a short adsorption time for reaching adsorption equilibrium. While for the composite adsorbents, the water adsorption includes physical adsorption and chemical adsorption (the formation of salt hydration). The temperature in the pores has a great change due to large adsorption heat release (caused by a large water adsorption capacity) and poor thermal conductivity of the adsorbents, thus the further formation of hydrate is difficult, leading to the long adsorption time for reaching adsorption equilibrium. So, in the practical application of the composite adsorbents, the adsorption heat should be timely removed in order to increase adsorption rate and refrigeration power.

To sum up the above arguments, for composite adsorbents prepared from 2–3 nm silica gels neither the water uptake nor the adsorption rate improve in contrast with pure silica gels. While for composite adsorbents prepared from 4–7 and 8–10 nm silica gels, both the water uptake and the adsorption rate raise a great deal compared to substrate silica gels.

3.3 Refrigeration performance

The composite adsorbents are mainly used for adsorption refrigeration. A lab-scale adsorption chiller with a cooling

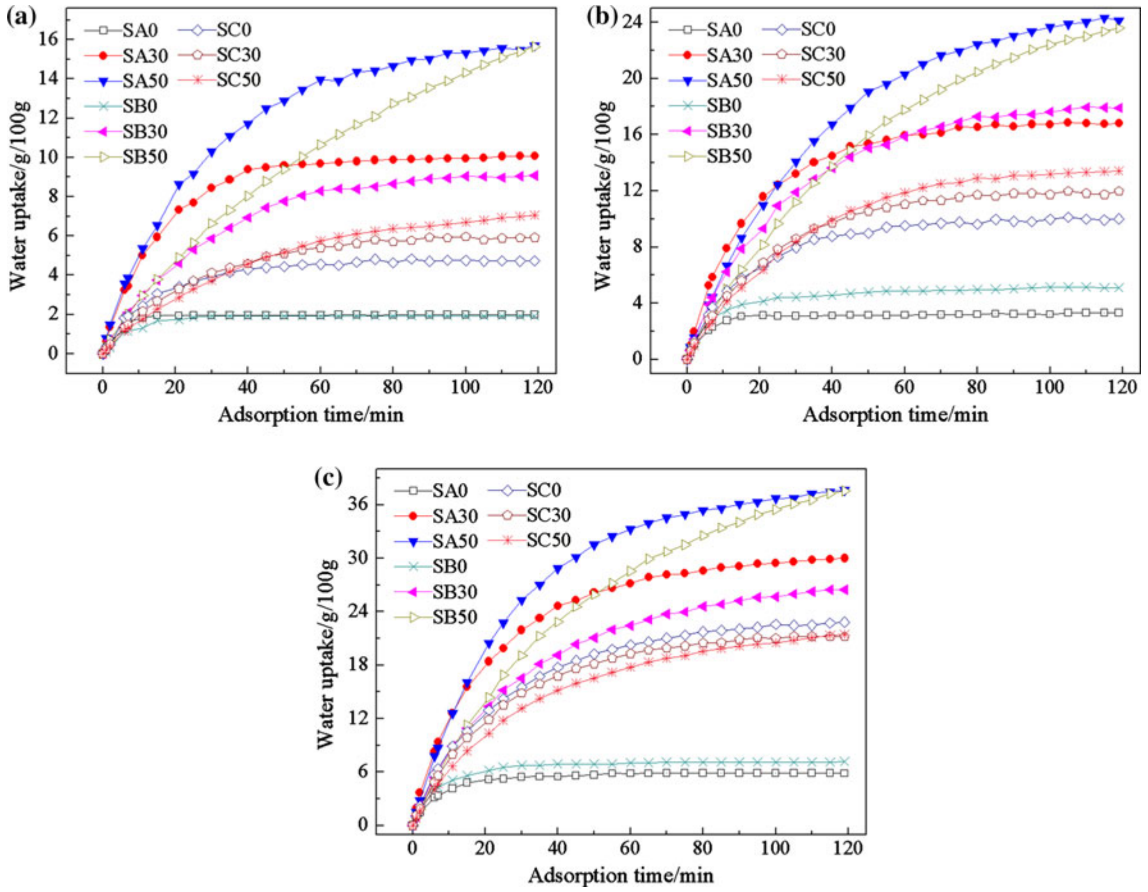

Fig. 2 Adsorption rate at different humidity. **a** Humidity = 20 %, **b** Humidity = 30 %, **c** Humidity = 50 %

power of 1 kW is manufactured in order to test the performance of composite adsorbents.

The typical work condition in the adsorption chiller is almost 20 % relative humidity and 5–20 min adsorption time for each cycle. The specific cooling power (SCP) is an important parameter of refrigeration performance of adsorbent. Figure 3 shows the variation of water uptake and SCP with time within twenty minutes. Observing the profiles in Fig. 3 it can be note that the SCP reaches the greatest value at about 2 min, and then decreases gradually with time. The adsorption time is generally no less than 5 min for practical refrigeration operation. Although the SCP is high for short adsorption time, the COP is low. This is due to the fact that the adsorption bed is in the process of cold and heat alternation, if the switch between adsorption operation and desorption operation is too frequent, the heat loss of the adsorption bed will be large, thus resulting in the low COP. The SCP for sample SA50 is 245, 222.7, 189 and 175 Wkg^{-1} at adsorption time of 5, 10, 15 and 20 min with relative humidity of 20 %. While for sample SA0, the SCP is 113.4, 73.1, 56.3 and 42.6 Wkg^{-1} respectively at the same adsorption time and same humidity, as shown in Fig. 3. The SCP for sample SB50 is 125.92, 124.43, 109.88 and 105.89 Wkg^{-1} at adsorption time of 5, 10, 15 and 20 min, while for sample SB0 the SCP is 92.67, 56.62, 49.41 and 37.6 Wkg^{-1} respectively. As evident in Fig. 3, the composite adsorbents of SA10 ∼ SA50 have better water uptake capacity compared to SB10 ∼ SB50, the sample SA50 has the highest SCP.

Based on the above analysis, a lab-scale adsorption chiller is manufactured using the composite adsorbent SA50, the photograph of the adsorption chiller is shown in Fig. 4. The chiller runs intermittently, which includes one adsorption bed, one condenser and one evaporator. The adsorption bed is a finned tube heat exchanger, and the adsorbents with the weight of 8 kg are packed between the fins. The design cooling power of the adsorption chiller is 1 kW with the size of 600 mm × 500 mm × 500 mm. The hot water and cooling water are supplied by two thermostats.

The test result shows that when the temperatures of hot water inlet, cooling water inlet, and chilled water inlet and outlet are 90, 35, 16.5 and 14.4 °C, the cooling power, SCP and COP are 1.03 kW, 128.3 Wkg^{-1} and 0.27 respectively with adsorption time of 10 min and desorption time of 5 min (cycle time of 15 min). While for adsorption chiller manufactured by the sample SA0, the cooling power, SCP

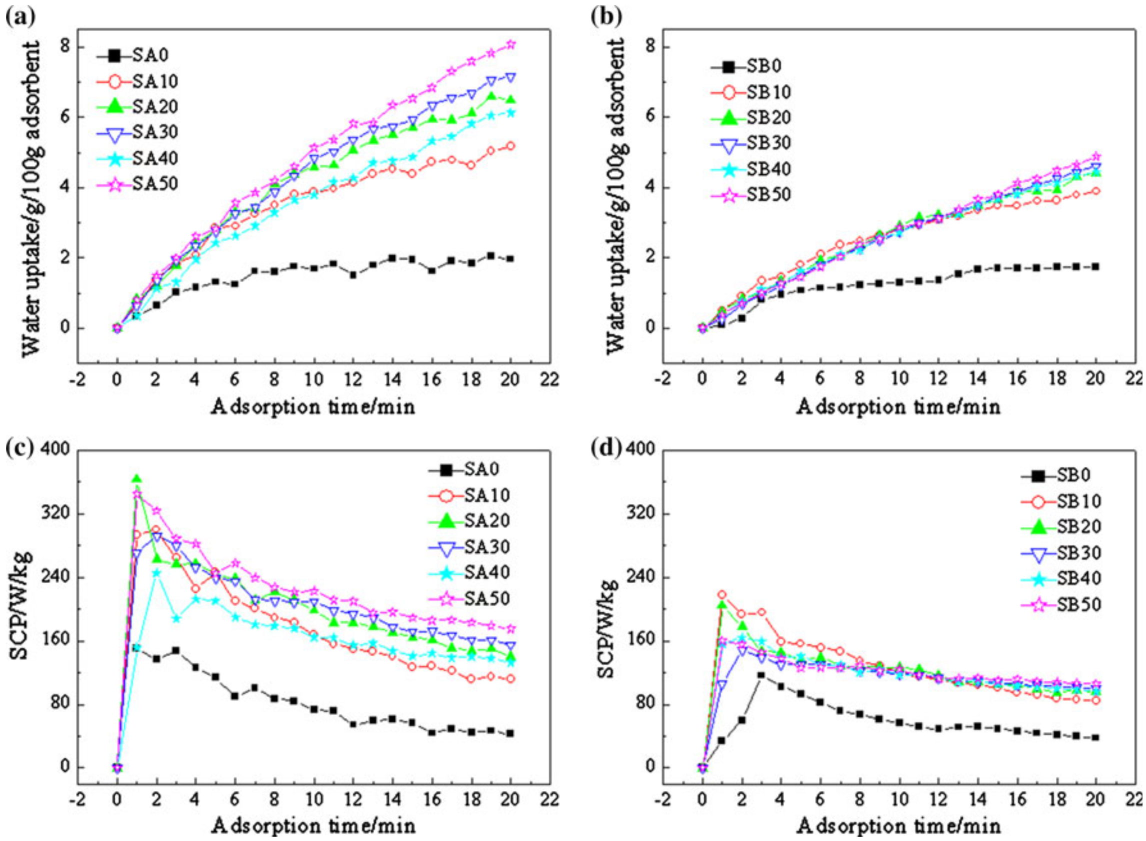

Fig. 3 Variation of adsorption amounts and SCP with time at humidity of 20 %

Fig. 4 Photograph of adsorption chiller

and COP are 0.33 kW, 41.5 Wkg^{-1} and 0.22 respectively under the same temperatures of hot water inlet, cooling water inlet and chilled water inlet, and the same cycle time.

The experimental result indicates that the SCP and COP of the adsorption chiller with the composite adsorbent SA50 are largely superior to that of SA0, hence it can be concluded that the manufacture of the adsorption chiller using the composite adsorbent, instead of the pure silica gel, makes the size of adsorption equipments small.

4 Conclusions

Three kinds of commercial silica gels with pore size of 2–3, 4–7 and 8–10 nm respectively are used for preparing composite adsorbents by soaking them into the aqueous solution of calcium chloride, and then the performances of the composite adsorbents are tested by experimental method. The conclusions are shown below:

(1) For composite adsorbents consisting of $CaCl_2$ impregnated inside the 2–3 nm silica gels neither the water uptake nor the adsorption rate improve in contrast with pure silica gels. While for composite adsorbents prepared from 4–7 and 8–10 nm silica gels, both the water uptake and the adsorption rate raise a great deal compared to the substrate silica gels. The water uptake of composite sample SA50 is 8.08 g/100 g at the humidity of 20 % and adsorption time of 20 min, while for sample SA0 the water uptake is only 1.96 g/100 g under the same operating conditions. The composite adsorbents have much better adsorption capacity than the substrate silica gels.

(2) The water uptakes of composite samples consisting of $CaCl_2$ impregnated inside the mesoporous and B type

silica gels increase with the increase of $CaCl_2$ content, however, they do not for samples prepared from the microporous silica gels. This is likely to due to the fact that the impregnation of calcium chloride may cause partial pore blockage due to the smaller pore size of 2–3 nm silica gel thus blocking mass transfer of vapor, but for 4–7 and 8–10 nm silica gels they do not.

(3) The SCP for sample SA50 is 245, 222.7, 189 and 175 Wkg^{-1} at adsorption time of 5, 10, 15 and 20 min with relative humidity of 20 %. The composite adsorbents of SA10 $\sim$ SA50 have better water uptake capacity compared to SB10 $\sim$ SB50, among them the sample SA50 has the highest SCP. Thus the sample SA50 is chosen as adsorbents to manufacture the adsorption chiller.

(4) The test result of an adsorption chiller manufactured using the composite adsorbent SA50 shows that when the temperatures of hot water inlet, cooling water inlet, and chilled water inlet and outlet are 90, 35, 16.5 and 14.4 °C, the cooling power, SCP and COP are 1.03 kW, 128.3 Wkg^{-1} and 0.27 respectively with adsorption time of 10 min and desorption time of 5 min (cycle time of 15 min). The SCP and COP of the chiller with the composite adsorbent SA50 are largely superior to that of SA0. Hence using the composite adsorbent instead of the pure silica gel can reduce the size of the adsorption chiller.

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