ADSORPTION PROPERTIES OF SEVERAL MATERIALS FOR SOLAR ADSORPTION COOLING SYSTEM

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ABSTRACT

It is important for an adsorption refrigeration system to use an appropriate adsorption material and the matched refrigerant. In the present study, the adsorption characteristic of the allochroic silica gel and three kinds of zeolite material to water-vapor have been investigated experimentally. Upon analyzing the variation of the material adsorption capacity exposed in moist air atmosphere of controlled temperature and humidity, the adsorption characteristic to water-vapor is verified, and the influence of the temperature and the pressure are correlated. The results show that the dynamic adsorption characteristic and the adsorption isotherm of the tested material is closely related to its microstructure such as the BET surface area, the distribution of the pore diameter, the porosity, and the bulk density, etc. In view of the application in the solar adsorption air conditioning, 5A-II and 13X-II zeolite shows relatively fast adsorption rate and their time to adsorption saturation state is short. The allochroic silica gel, 5A-II and 13X-II zeolite is of higher adsorption capacity, while the dynamic adsorption characteristic of 5A-I and ZSM-5 zeolite is more sensitive to the change of pressure. The adsorption isotherm of allochroic silica gel follows an exponential function and ZSM-5 and 5A-I zeolites shows a "S" curve, The latter may be more suitable for the solar adsorption cooling system in summer.

KEYWORDS

zeolites, adsorption capacity, dynamic adsorption rate, microstructure, solar energy

INTRODUCTION

Using solar energy to drive air conditioning system in summer, so as to reduce the electricity power consumption of the building, is always a technological goal for researchers. Compared to absorption refrigeration, which is usually large in system size, there are preferable advantages of adsorption refrigeration, such as lower temperature requirement of heat source, smaller system, flexible application, etc. The research on the adsorption refrigeration has been paid wide attention in recent years [1-3].

Generally, the application of adsorption refrigeration strongly depends on the working pair, which significantly determines the refrigeration coefficient, the preliminary investment of the system and the devices [4]. Currently, the performance of working pairs for adsorption cooling such as the activated carbon-methanol, zeolite-water, silica gel-water, calcium chloride-ammonia has been widely reported. In the study of Dawoud and Aristov [5] , the adsorption properties of the composite adsorbent that is made of silica gel and alumina have been investigated. The results suggested that the composite adsorbent had higher adsorption capacity but lower adsorption rate compared to the original adsorbent. In the study of Choma et al [6], the method of nitrogen adsorption was invoked to identify the difference of the adsorption characteristic between the unmodified silica gel and the modified silica gel. Jentys et al. [7] studied the adsorption characteristic of ZSM-5 zeolite to water-vapor using chromatographic analysis. They found that the cations in the working pair signigicantly affect the adsorption characteristic. Wang et al [8]. investigated the equilibrium adsorption capacity of the compound adsorbent of $CaCl_2/CaSO_4$. The results showed that the refrigeration capacity of the compound adsorbent of $CaCl_2/CaSO_4$ is 1.26 times of the pure $CaCl_2$.

NOMENCLATURE

a	[g/g]	Correlation constant in Eq.(3)
A_1	[g/g]	Correlation constant in Eq.(2)
A_2	[g/g]	Correlation constant in Eq.(2)
b	[g/g]	Correlation constant in Eq.(3)
B_1	[g/g]	Correlation constant in Eq.(4)
B_2	[g/g]	Correlation constant in Eq.(4)
c	[-]	Correlation constant in Eq.(3)
d	[nm]	Pore diameter in the material
dp	[Pa]	Correlation constant in Eq.(4)
K	[-]	Prameter in D-A Eq.(9)
m_0	[g]	Mass of the material at dry state
m_1	[g]	Mass of the material after adsorption
n	[-]	Prameter in D-A Eq.(9)
P_0	[Pa]	Correlation constant in Eq.(4)
P_{H2O}	[Pa]	Partial pressure of the water vapour
P_s	[Pa]	Vapour saturation pressure for a given temperature
Q_0	[g/g]	Correlation constant in Eq.(2)

Q	[g/g]	Adsorption amount of the material
Q_{max}	[g/g]	Equilibrium adsorption amount for cases φ<100%
Q_{sat}	[g/g]	Equilibrium adsorption amount at the saturation temperature T_s
t_1	[min]	Correlation constant in Eq.(2)
t_2	[min]	Correlation constant in Eq.(2)
T	[℃]	Temperature
T_s	[℃]	Saturation temperature to the vapor's partial pressure
φ	[-]	Relative humidity of the air

Mostly, the research on the adsorption properties of various materials have been conducted from the micro perspective. However, in the actual system, the sorbent bed is loaded with a large amount of absorbent material. The adsorption properties in practical system are influenced by many factors, such as the stack mode of the adsorbent material, the method of enhancing the heat-transfer, and the form of the heat collector, etc. Therefore, conclusions that were drawn from the micro perspective can not be directly applied to the actual adsorption system. To get insight into the adsorption characteristics of the material in the bulk status, the current study will aim to the allochroic silicagel and three kinds of zeolite for their adsorption characteristic to water-vapor. The experiment is focused on the adsorption characteristic of the material at a small stacking state. In addition to the adsorption rate and the equilibrium adsorption capacity, the impact of the temperature and the pressure will be also paid attention.

EXPERIMENTAL METHOD

The allochroic silica gel and the ZSM-5 zeolite used in the study are provided by Xilong Chemical co.and Tianjin Shenneng Technology co., respectively. 5A-I, 13X-I, 5A-II, 13X-II zeolite are provided by Langfang Peng Cai Fine Chemical co., LTD., in which I and II represents different production batches. For the experiment the test sample is put into a container. The container is a small sieve of stainless steel with 10 mesh in hole, and 1.5cm high and 6cm in diamter. After being weighted, the test sample, together with the small sieve, was put into the test chamber of constant temperature and humidity and then experienced a time period of about 400 minutes. The experimental atmosphere in the chamber can be controlled in terms of temperature and humidity, with the fluctuation of the temperature ± 0.5 ℃ , and the relative

humidity ± 2.5%. However, the total pressure of the experimental atmosphere is not adjustable and ever kept at 1 atm. To know the weight change of the test sample with the time, it was taken out of the chamber and weighted in an electronic balance at certain time interval. The electronic balance is of the precision of 0.1mg uncertainy.

Prior to the adsorption experiment, the test sample was firstly heated for two hours at 120℃ to ensure it was in complete dry state. For most cases of the adsorption test, the temperature was set to T = 20 ℃, 30 ℃, 40 ℃, 50 ℃, and the humidity φ = 40%, 50%, 60%, 70%, 80%, 85% for each temperature. Seeing that the dynamic adsorption process possesses a feature of fast rate at begining and then slowing down gradually, the time interval adopted in the test has been taken as changeable, with shorter interval within 100 minutes, and longer afterwards. On the other hand, the fluctuation of the relative humidity would affect the smooth and continuity of the dynamic adsorption curve. To know the effect of the relative humidity fluctuation on the adsorption process, the relative humidity in different time was recorded, too.

The microscopic characteristics of the material, such as the specific surface area (by multi-point BET method), and the pore structure of the adsorption materials, were determined by 3H-2000PS2 chemical analyzer. The samples were degassed by heating to 200℃ in a vacuum lasting 12h to remove impurity molecules and non-adsorbate molecules before analysis. As shown in Table 1, the specific surface area, bulk density, porosity, etc. are different one another. The same kinds of material that are from the same company but from different product batches are also different of the structure. In the present study, the adsorption material contains both the micropore($d < 2nm$) and the mesopore ($2nm < d < 50nm$), but the proportion is different. The total pore volume in the table refers to the sum of the micropore and the mesopore volumes. The analyzer results of the microstructure tell us that the micropore volume and the specific surface area of ZSM-5 and 5A-I zeolite are lower than the others. Such a difference is of much significance and their impact will be reflected in the following adsorption isotherm.

Table 1 Microstructure characterization of the tested adsorption material

	particle size （mm）	porosity （%）	micropore volume （ml/g）	total pore volume （ml/g）	Aperture (nm)		specific surface area (m²/g)	bulk density （g/L）
					mesopore	micropore		
Allochroic silicagel	4.1	59.8	0.289	0.336	2.29	0.93	535.8	821.8
ZSM-5 zeolite	2.2	72.8	0.071	0.590	4.41	0.83	282.5	593.7
5A-I zeolite	4.8	41.8	0.160	0.329	3.47	0.81	474.1	638.9
5A-II zeolite	4.3	45.6	0.246	0.375	2.00	0.80	655.8	599.6
13X-I zeolite	2.3	34.9	0.239	0.358	2.00	0.82	638.7	659.9
13X-II zeolite	4.2	47.6	0.243	0.372	2.00	0.86	652.3	586.3

Note：ZSM-5 zeolite is the form of strip molecular sieve with diameter of 2.2 mm and 20 mm in average length.

DYNAMIC ADSORPTION CHARACTERISTICS

Adsorption amount Q of the material is defined as

$$Q = \frac{m_1 - m_0}{m_0} \times 100\% \qquad (1)$$

where m_0 is the mass of the material in the dry state, m_1 is the mass after adsorbing water. With the recorded data of the weight change of the test sample, the Q-t dynamic adsorption curve were obtained. Due to the performance limitation of the constant temperature and humidity test chamber, it is hard to reach such cases as the high temperature - high humidity, and nor the low temperature-low humidity. Therefore the adsorption amount in the saturated air Q_{sat}, which is referred as the saturated adsorption amount for a given temperature, can not be obtained directly from the experiment data. Thus, in Eq. (1) Q is actually the "unsaturated adsorption amount" in the case of $\varphi < 100\%$, while the Q_{sat} can only be obtained indirectly from fitting the data.

The dynamic adsorption curve of the material at the temperature of 40 ℃ and the relative humidity of 70% (corresponding to the partial pressure of water vapor $\overline{P}_{H_2O} = 5167$ Pa) is presented in Figure 1. Connecting lines in the figure are drawn as the result of the asymptotic fitting formula, which is

$$Q = Q_0 + A_1 \times \left[1 - e^{\left(-\frac{t}{t_1} \right)} \right] + A_2 \times \left[1 - e^{\left(-\frac{t}{t_2} \right)} \right] \qquad (2)$$

where t is the time, and Q_0, A_1, A_2, t_1, t_2 are the fitting parameters. When the time t tends to infinity, the $Q_0 + A_1 + A_2$ is the unsaturated asymptotic value Q_{max}, which represents the maximum amount of adsorption under this condition. Fitting parameters for the different adsorption materials, together with the asymptotic value Q_{max}, are shown in Table 2.

Table 2 The fitting parameters for Eq.(2)

	allochroic silicagel	ZSM-5	5A-I	5A-II	13X-I	13X-II
Q_0	0.005	0.000	0.000	0.000	-0.003	-0.001
A_1	0.142	0.137	0.099	0.045	0.110	0.258
t_1	153.6	69.41	13.89	90.47	38.71	19.85
A_2	0.142	0.042	0.099	0.234	0.110	0.016
t_2	153.6	0.125	93.31	11.85	38.71	133.8
Q_{max}	0.288	0.180	0.198	0.279	0.217	0.273

As shown in Table 2, allochroic silicagel, 5A-II, and 13X-II possesses higher Q_{max} than the others. Its reason may be interpreted in analysis of the microstructure of the adsorption material. Upon analyzing the relationship between Q_{max} and the specific surface area and the porosity, it is revealed that the adsorption material with higher Q_{max} such as allochroic silicagel, 5A-II, 13X-II and 13X-I zeolite all possess higher specific surface area. The asymptotic adsorption amount of the silicagel is the highest among the testing materials because it possesses a high specific surface area as well as a high porosity. On the other hand, though the ZSM-5 zeolite possesses the highest porosity, its Q_{max} is the lowest. This is mainly attributed to its least specific surface area (282.5 m²/g). It is inferred that appropriate specific surface area acts as the basis of adsorption phenomena, while the porosity does not improve the adsorption ability of the material necessarily. As is known, there are two kinds of pore, the connected and the disconnected. The former promote the adsorption ability, and the latter does not contribute anything.
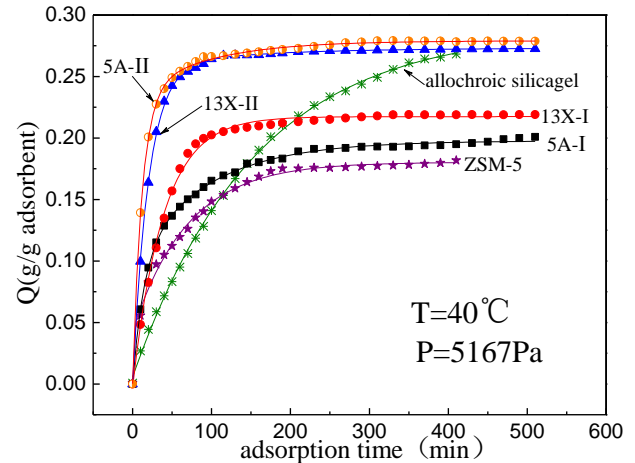

Figure 1 Dynamic adsorption curve of the test materials under the same condition

In solar adsorption air conditioning system, the adsorbent is required to possess not only a high adsorption capacity but also a fast absorption rate. By differentiating the adsorption curve in Figure 1 to the time, the related adsorption rate of the test material is obtained and the result is presented in Figure 2. The results suggest that the adsorption rate for all the sample is the highest in the initial period and then decreases gradually. By the theory of adsorption dynamics, the adsorption phenomenon happens only on the potential caveolaes. At beginning of the process all the caveolaes are empty so that the collision of the water molecule and the caveolae is readily to happen. As the adsorption proceeds more potential caveolaes are occupied, so that the molecule collisions become less and the adsorption rate slow down correspondingly. It is clear that the initial adsorption rate of 5A-II and 13X-II zeolite is the highest, while those of ZSM-5 zeolite and the allochroic silicagel are much lower. In addition, the adsorption rate of the allochroic silicagel and ZSM-5 zeolite in the test period are almost invariable because of their low initial adsorption rate.
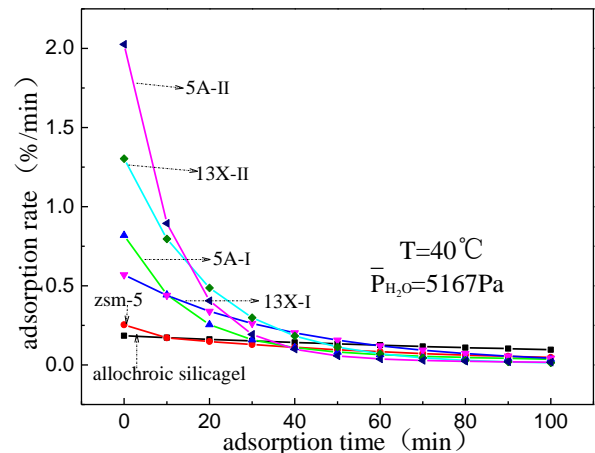

Figure 2 Adsorption rate comparison of the tested materials

During the experiment it is found that the humidity fluctuation inside the test chamber strongly affects the dynamic curve of the ZSM-5, 5A-I, and 13X-I zeolites, but less to the other three. This implies that the different adsorption material is of different sensitivity to the vapor pressure. The relevance between the adsorption capacity and the fluctuation of the vapor pressure in the condition of T = 40 ℃, φ= 70% is shown in Figure 3. If an adsorbent material is sensitive to the pressure change, it will be affected jointly by the pressure and the temperature in the adsorption performance. Generally, we hope that the adsorption capacity of material is a strong function of the temperature but a weak function of the pressure. Since the pressure in the adsorption bed will rise up in the phase of desorption as being heated, the strong dependence of the adsorption on the pressure will result in poor desorption effect. On the other hand, the weak dependence to pressure implies that adsorbent still maintains relatively high adsorption capacity in the phase of adsorption cooling.
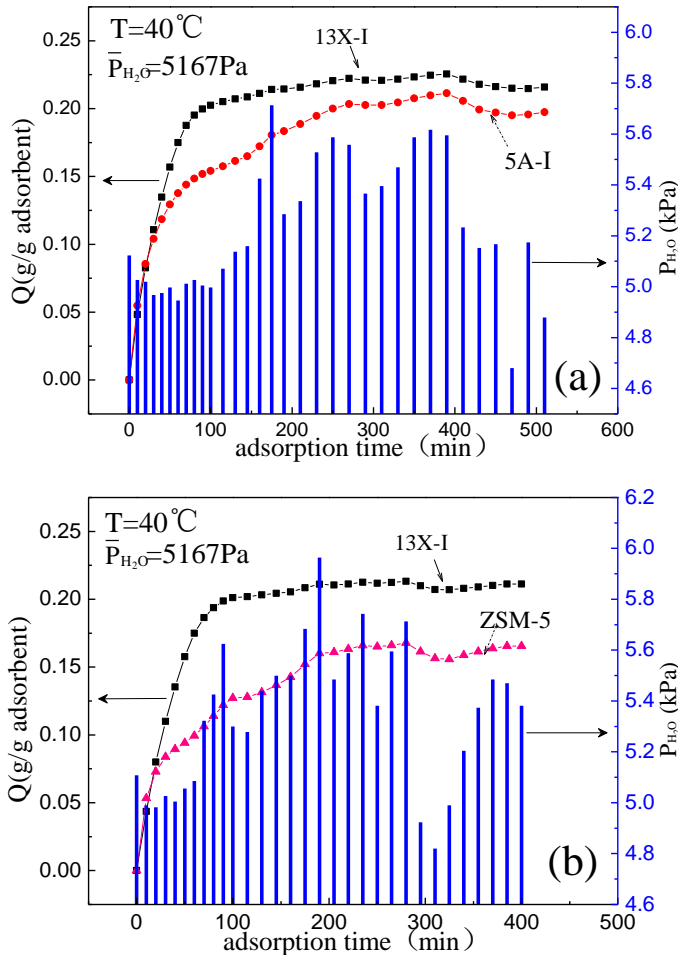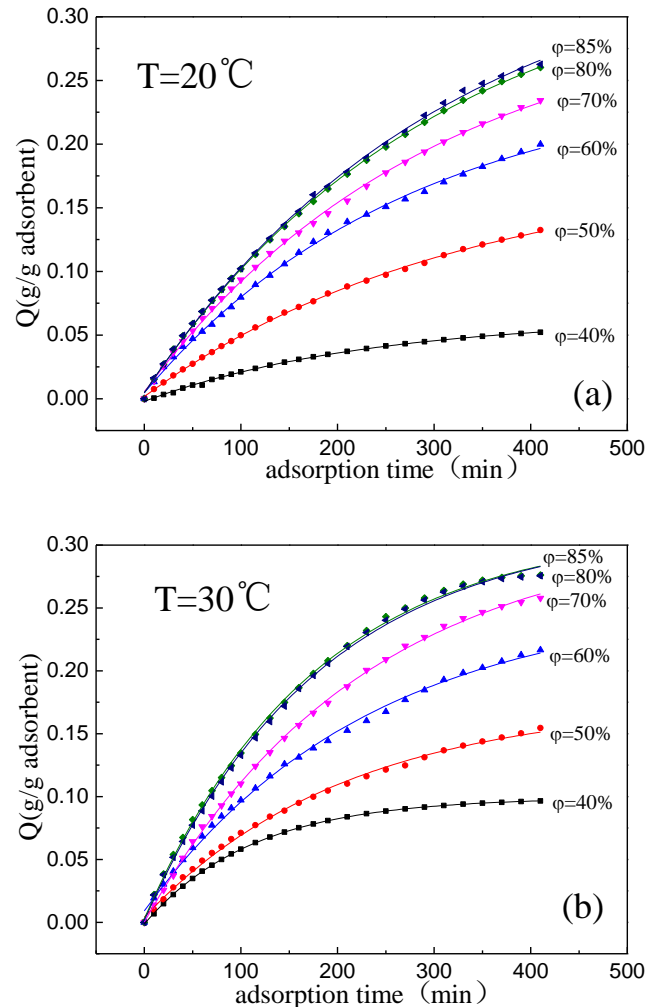
As shown in Figure 3, ZSM-5, 5A-I zeolite are more sensitive to pressure changes compared to 13X-I zeolite, and their adsorption capacities are lower than 13X-I zeolite. In comparison under the given experimental condition, the ZSM-5 zeolite shows inferior performance feature in terms of the dynamics as well as the equilibrium adsorption capacity.

To facilitate understanding the variation of the dynamic adsorption process at different temperatures, four cases of the allochroic silicagel are selected here as shown in Figure 4. Obviously, as the temperature increases the adsorption rate in the initial period becomes more and more rapid. As the result the time to reach the equilibrium state decreases correspondingly. At the same time, the equilibrium adsorption capacity decreases with the increase of the temperature, which is the most important characteristic to fulfill the cooling cycle as driven by the solar energy. The influence of the temperature and the humidity for the other tested materials is similar to the situation of the allochroic silicagel, though every kind of material has its own different equilibrium value of the adsorption capacity.
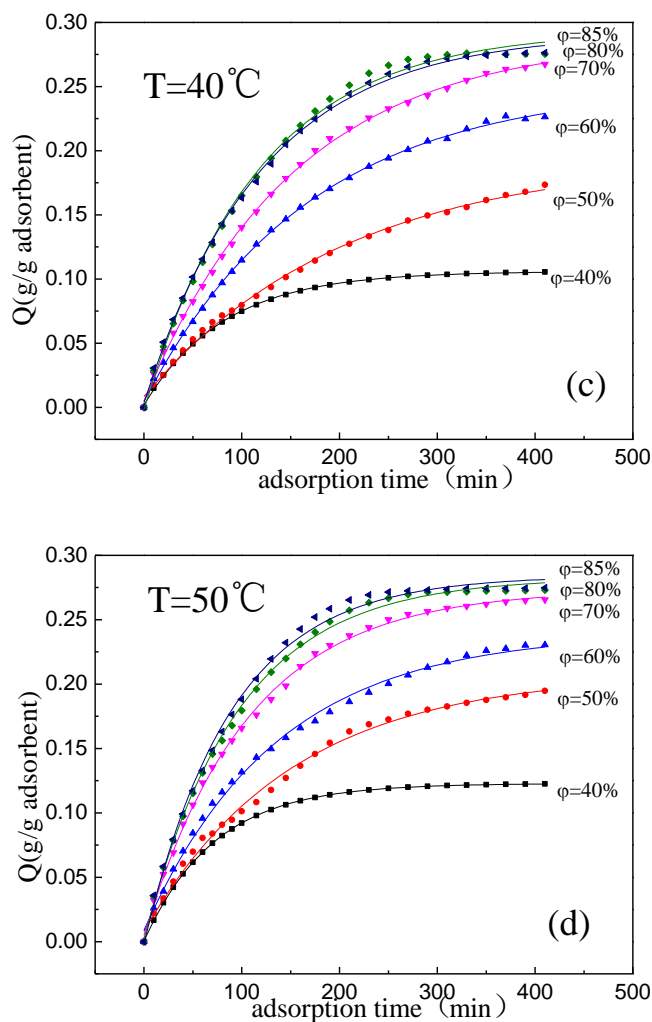

Figure 3 Comparison of the adsorption sensibility to the vapor pressure fluctuation for 5A-I, 13X-I, and ZSM-5

Figure 4. Dynamic adsorption curves of allochroic silicagel to water-vapor at different temperatures

UNSATURATED ASYMPTOTIC ADSORPTION CAPACITY

Now the attention is turned to the discussion of the unsaturated asymptotic adsorption capacity of the material. Here the ZSM-5, the 5A-I, and the allochroic silicagel will be taken as the representative materials in the disscusion. At the first step, the unsaturated asymptotic adsorption Q_{max} of the three kinds of material for a relative humidity is fitted according to Eq. (2). The Q_{max} increases with the increase of the partial pressure of the water vapor. Then the change of the Q_{max} with the water vapor pressure is fitted. For the allochroic silicagel, the asymptote fitting model is applied, which is

$$Q = a - b \times c^P \qquad (3)$$

where a, b, c are fitting parameters, P is the partial pressure of the water vapor. The "S" fitting model is more suitable for ZSM-5 and 5A-I zeolite, and the formula is

$$Q = \frac{B_1 - B_2}{1 + e^{(P-P_0)/dp}} + B_2 \qquad (4)$$

where B_1, B_2, P_0, dp are fitting parameters. The water vapor pressure P is determined by the relative humidity φ and the vapor saturation pressure P_s, which corresponds to the experimental temperature. The transformation formula from φ to P is as

$$P = \varphi P_s \qquad (5)$$

The correlating result of the three materials at T = 40 ℃ are shown in Figure 5. The correlation for the silica gel in the figure is as

$$Q = 0.31 - 2.48 \times 0.43^P \qquad (6)$$

While the correlation of 5A-I and ZSM-5 zeolite takes the different form

$$Q = \frac{0.14 - 0.30}{1 + e^{(P-5.47)/0.64}} + 0.30 \qquad (7)$$

$$Q = \frac{0.08 - 0.20}{1 + e^{(P-4.69)/0.33}} + 0.20 \qquad (8)$$

In comparison of the curves in Figure 5, it is revealed that the adsorption capacity of the allochroic silicagel increases quickly at low pressure, but slow down with the increase of the pressure. This phenomenon is closely related to the pore structure of the allochroic silicagel. As shown in Table 1, the micropore volume of the allochroic silicagel (0.289 ml/g) is obviously greater than those of ZSM-5 zeolite (0.071 ml/g) and 5A-I zeolite (0.160 ml/g). If the pore diameter distributions of the adsorbent material are all in the molecular scale, the adsorption capacity will increase quickly and reach to saturation at low pressure in accordance with the adsorption theory. Nevertheless, in Figure 5 the allochroic silicagel experienced a rather wide range of the pressure to reach the equilibrium adsorption status. It is speculated that the pore diameter distributions of the allochroic silicagel are in a wide range rather than solely in the molecular scale, and there should be larger pores besides the microporous channels. In contrast, the S shape of the ZSM-5 and 5A-I zeolites implies their adsorption ability at low pressure is weak. This must correspond to the fact of lacking micropores in the material. On the other hand, the adsorption capacity increased quickly when the pressure reaches to the medium stage. This suggests that the capillary condensation phenomenon occurs when the pressure reaches the critical pressure that corresponds to some pore diameter [9]. After the capillary condensation finishes in all mesopores, the adsorption appears only on the outside surface of the material. Since the adsorption capacity of the outside surface of the material is much less than the inner surface, the adsorption gradually slows down and the upper platform of the curve forms. The inference about the pore diameter distribution of the material above can basically be confirmed by the microstructure data listed in Table 1.
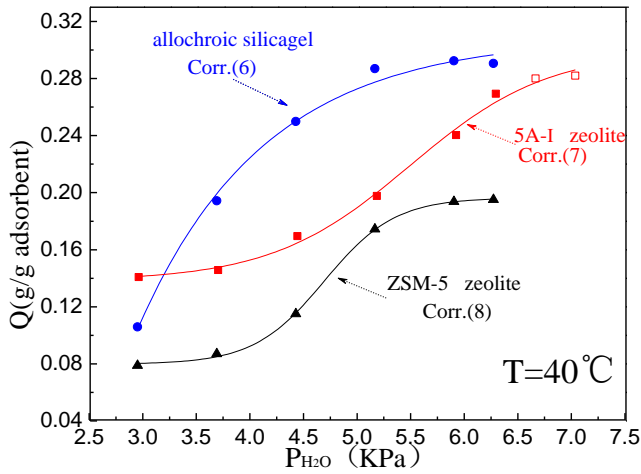
Figure 5. The unsaturated asymptotic adsorption capacity of the three materials vs the water vapor pressure. The two hollow points of 5A-I curve were obtained by extrapolating Eq.(7)

ADSORPTION ISOTHERM AND SATURATION ADSORPTION CAPACITY

In design of an adsorption cooling system it is essential to know the relevance of the adsorption capacity of the adsorbent with the pressure at different temperatures. The variation of the adsorption capacity at high and low temperature in a cycle determines how much adsorbent is needed and how big is the system. Figure 6 presents the tendency of the adsorption capacity with the partial pressure of the water vapor at different temperatures. These curves are known as the adsorption isotherm, which describes the dependence of the adsorption capacity on the temperature and the pressure.

As shown in Figure 6(a), the adsorption capacity of the allochroic silicagel increases with the pressure monotonically. Its adsorption capacity is more sensitive to the change of the pressure at low temperature. For the allochroic silicagel the adsorption isotherm is a kind of exponential function. In contrast, those isotherms of ZSM- 5 and 5A- I zeolite show typical "S" type. For the "S" type there are two platforms at high pressure and low pressure, with a rapid increasing section in between the two platforms. The "S" type of the adsorption isotherm is favorable for improving the performance of adsorption refrigeration system [10]. If the material is of an adsorption isotherm of "S" type, the adsorption bed may rapidly achieve desorption without need a rather low ambient temperature. In contrast, if the material's adsorption isotherm is exponential, a lower condensation temperature may be needed to obtain deep vacuum pressure in the adsorption bed.
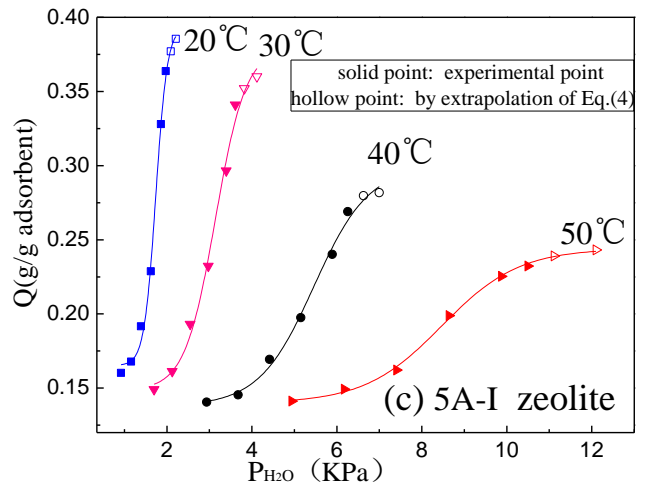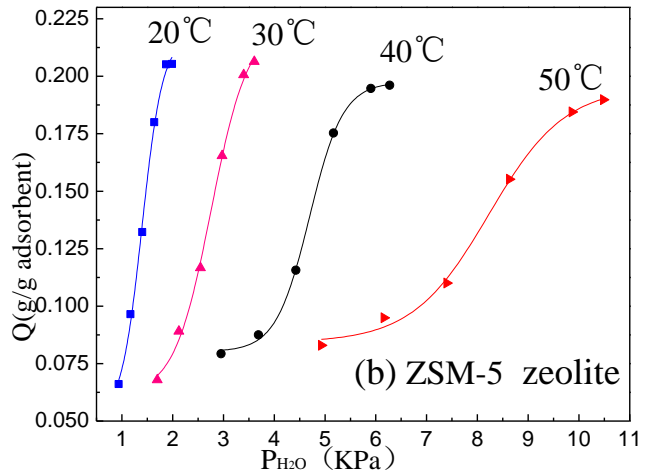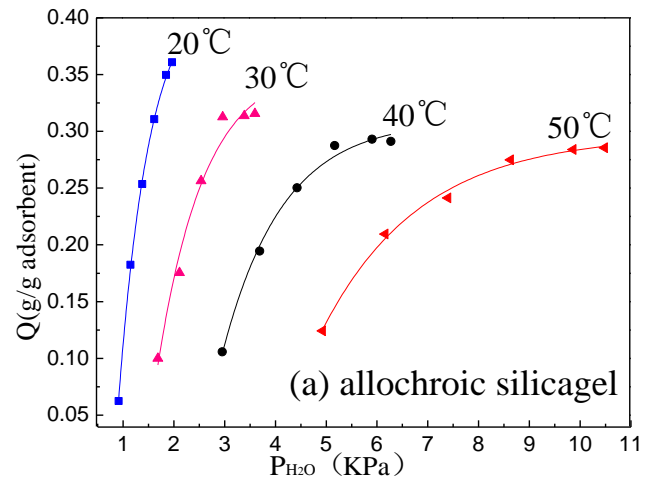

Figure 6 Adsorption isotherms of the three tested material

Although the adsorption capacity increases with the water vapor pressure as seen in Figure 6, there is a limited value of it. The limited adsorption capacity is the maximum adsorption capacity under the saturation pressure of the test temperature. More specifically, the saturation adsorption capacity corresponds to the top asymptotic value of the curve,

which is basically equal to the fitting constant "a" in Eq. (3), or the constant "B_2" in Eq.(4). As shown in Figure 7, the saturation adsorption capacity of the material obviously decreases with the temperature. In general, the saturation adsorption capacity of the allochroic silicagel and the 5A-I zeolite changes with the temperature strongly. For 5A-I zeolite the Q_{sat} decreases even to 40% with the temperature change from 20℃ to 50℃. In contrast, the saturation adsorption capacity of the ZSM-5 zeolite is in low level, and it is not so sensitive to the change of the temperature.
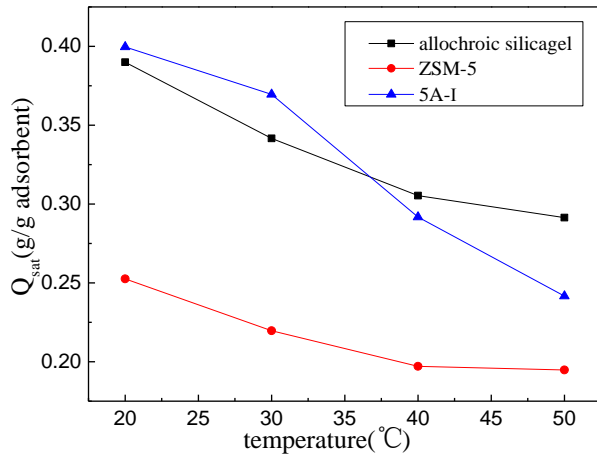

Figure 7 The change of the saturation adsorption capacity with the temperature for the three tested material

THEORETICAL PREDICTION OF ADSORPTION CAPACITY OF ALLOCHROIC SILICAGEL

Since different adsorption material shows different feature of microstructure, it is hard to describe the adsorption process for all of them in a uniform equation. There are three basically equations for the physical adsorption, i.e. the equation based on the speed theory, the thermodynamic adsorption equation, and the equation based on the capillary condensation theory [11]. Among them, the second category equation is most widely used. A representative of it is the Dubinin-Astakhov equation, which was proposed according to the theory of micropore filling driven by the thermodynamic potential. The simplified D-A equation is the form as

$$Q = Q_{sat} \exp\left[-K\left(\frac{T}{T_s}-1\right)^n\right] \qquad (9)$$

In Eq.(9) T_s corresponds to the vapor's partial pressure P. The constant K is jointly determined by the characteristics of the adsorbent and the adsorbate. n is associated with the pore diameter distribution in the adsorbent. $n = 2$ means that the pore of $d = 1.8\sim2.0nm$ is the dominant, $n < 2$ for the pores of $d > 2.0nm$ mostly, and $n > 2$ for $d < 1.8nm$ mostly.

Basically, the relationship of the adsorption capacity and the temperature in Eq.(9) is an exponential function, so it is not suitable to predict the performance of the material with

the adsorption isotherm of "S" type. On the other hand, the adsorption capacity of the allochroic silicagel could be well predicted by this equation, see Figure 8 and Table 3. Seeing that both the Corr.(3) and the D-A Eq.(9) belongs to the category of exponential function, the consistency of the point and the line in Figure 8 is reasonable. What is the difference here from the Corr.(3) is that the physical meaning of each parameter in D-A equation is more clearly, because it is obtained from the theoretical analysis. Moreover, the predicted values of n are all beyond 2 in Table 3. This suggests that the pore structure of the allochroic silicagel is mainly consist of micropore of $d < 1.8nm$. This is consistent with the results presented in Table 1, in which the micropore is clearly dominant to the mesopore for the allochroic silicagel. Also, the experimental result of the Q_{sat} is presented in Table 3 to facilitate the comparison. It can be seen that not only the tendency of the change is consistent one another, but also the adsorption amounts itself are well comparable.
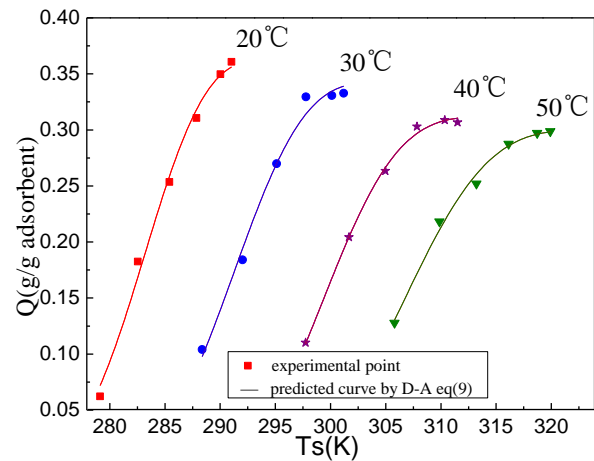

Figure 8 Comparision between the predicted and the experimental adsorption capacity of the allochroic silicagel

Table 3 Fitting parameters of the allochroic silicagel adsorption isotherm in Figure 8

	20℃	30℃	40℃	50℃
K	5207.9	5629.7	13586.1	5432.8
n	2.699	2.854	3.261	3.066
Q_{sat}(by Eq.(9))	0.362	0.325	0.296	0.285
Q_{sat}(experimental)	0.390	0.342	0.305	0.291

CONCLUSIONS

In the study the adsorption characteristics to water-vapor for allochroic silicagel, ZSM-5, 5A, and 13X zeolite in stack state have been investigated experimentally. Their microstructure features are also characterized. The results show that the allochroic silicagel is of higher adsorption capacity at the same condition, and ZSM-5 zeolite is inferior to the others. As to the 5A and 13X zeolites, they have shown variable equilibrium adsorption capacity since the test

samples were taken from different product batches. The initial adsorption rate of 5A-II, 13X-II, 5A-I is superior. The adsorption rate increases strongly with the temperature and the relative humidity. The dynamic adsorption curve of the ZSM-5 and the 5A-I zeolite are more sensitive to the pressure change than the 13X-I zeolite. The adsorption isotherm of the allochroic silica gel belongs to an exponential form and the ZSM-5 and 5A-I is of "S" shape. Finally, a discussion is conducted on the suitability of the Dubini-Astakhov equation to predict the adsorption capacity of the material. The analysis revealed that the D-A equation is more suitable to predict the adsorption capacity of the allochroic silicagel rather than those materials with a "S" type adsorption isotherm.

ACKNOWLEDGMENT

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