ISSN 0023 1584, Kinetics and Catalysis, 2010, Vol. 51, No. 5, pp. 754...761. © Pleiades Publishing, Ltd., 2010.

Dynamics and Isotherms of Water Vapor Sorption on Mesoporous Silica Gels Modified by Different Salts[1]

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Received October 9, 2009

Abstract—The mesoporous silica gels impregnated with different metal salts were prepared and studied. The pore structure and specific surface area of adsorbents were evaluated using nitrogen adsorption. Then, the sorption isotherms and dynamics of water vapor were carried out at 303 K and different relative humidity (RH). The temperature programmed desorption experiments were conducted to estimate the activation energy (E_d) of water desorption on the silica gels. The results showed that the sorption capacity for water decreased with the increase of the ionic radius (except the calcium ion) and that $CaCl_2$ and LiCl were particularly suitable for use in modification of the mesoporous silica gel to improve their sorption rates and capacities for water vapor at the lower and medium RH (RH < 80%). The larger the average pore diameter and pore volume of the initial silica gels, higher the accrual rates of the water vapor sorption rate and capacity were after modification with hygroscopic salts. The activation energy of the water desorption on the mesoporous silica gel modified by $CaCl_2$ were much higher than that on the silica gel modified by LiCl, because the polarizability of the Ca^{2+} was higher than that of Li^+.

DOI: 10.1134/S0023158410050186

Silica gels are widely used in dehumidification processes because their good moisture sorption capacities [1] which mainly depend on its larger surface area and pore distribution. It is well known that the pore structure and surface chemistry of silica gels also play important roles in the sorption of water vapor besides their developed surface area.

The sorption performance of silica gels was improved by different methods such as irradiation by neutron flux [2], manipulation of preparation parameters [3], and modification by plasma treatment [4]. Another way to increase moisture sorption capacity is the impregnation of silica gels with hygroscopic salts [5–11] such as $CaCl_2$, LiBr, LiCl, $Ca(NO_3)_2$, and $MgSO_4$, which can adsorb water vapor as the solid crystalline and/or salt solution. All of these methods are good for the improvement of sorption performance of adsorbents through improving the pore volume and/or surface chemistry. However, the effects of different salts impregnation and porous structure of the host matrix on the water sorption properties of initial and modified silica gels have not been investigated systematically.

In this paper, the sorption dynamics and desorption activation energy of water vapor on modified silica gels were investigated. The object of study was to search for the influencing factors that can greatly improve the sorption capacity, sorption rates and the activation energy of water vapor desorption on the silica gel in order to develop highly effective dehumidification materials with lower energy consumed in the regeneration process for industrial and residential applications.

EXPERIMENTAL

Materials

The three commercial silica gels used in this study were purchased from Qingdao Haiyang Chemical Co., Ltd and Spegial Silica Gel Factory (Qingdao, China). Their particle sizes ranged from 3 to 4 mm. They will be referred in the text as A_{sg}, B_{sg}, and C_{sg}, respectively (Table 1).

Table 1. Porous structure parameters of silica gels

Silica gel	BET surface area, m^2/g	Volume of pores, cm^3/g	Average pore diameter, nm	Salt content, wt %
A_{sg}	690.5	0.36	2.0	...
B_{sg}	591.4	0.82	5.3	...
C_{sg}	349.2	0.96	10.7	...
Li/C_{sg}	242.5	0.84	13.8	3.9
Ca/C_{sg}	212.1	0.84	15.3	9.5

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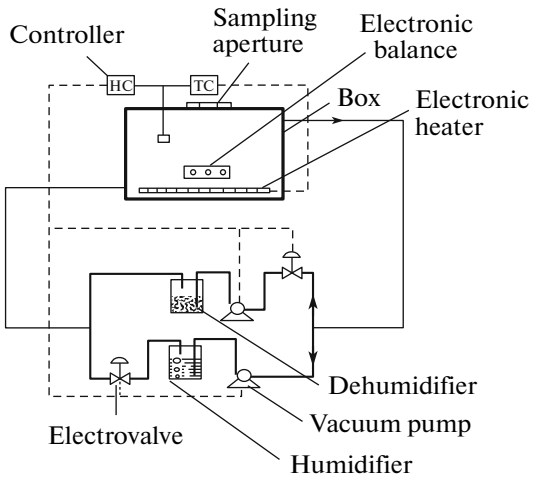

Fig. 1. Dynamic adsorption apparatus.

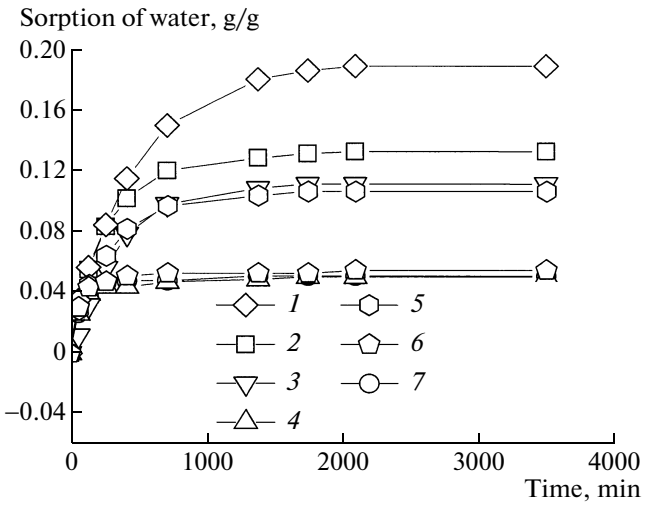

Fig. 2. The sorption kinetics of water vapor on C_{sg} and modified silica gels at 303 K and RH = 30%: *1*—Ca/C_{sg}, *2*—Li/C_{sg}, *3*—Mg/C_{sg}, *4*—K/C_{sg}, *5*—Zn/C_{sg}, *6*—C_{sg}, *7*—Na/C_{sg}.

Prior to the synthesis of the composites, silica gels were dried at 413 K in a vacuum for 4 h. A 10 g of host silica gel was immersed and kept in the 20 ml of 1M solutions of different salts for 8 h. Then the samples were taken out of the solution and laid at 140°C in oven for 4 h. At last, the dry composite adsorbents were placed in a vacuum desiccators for use. The composite adsorbents of C_{sg} treated by 1 M solutions of $CaCl_2$, $ZnCl_2$, $MgCl_2$, LiCl, KCl, or NaCl will be referred in the text as Ca/C_{sg}, Zn/C_{sg}, Mg/C_{sg}, Li/C_{sg}, K/C_{sg}, and Na/C_{sg}, respectively, and A_{sg} or B_{sg} treated by 1 M solution of $CaCl_2$ will also be referred as Ca/A_{sg} or Ca/B_{sg}.

Methods

Sorption of nitrogen. The specific surface area, the pore volume, and the average pore diameter of the samples were measured by nitrogen sorption at the liquid nitrogen temperature 77 K in a "Micromeritics" gas sorption analyzer ASAP 2010. The silica gel sample was degassed at 573 K for 3 h in the vacuum prior to nitrogen sorption measurements. The surface area was calculated from the sorption isotherms using the standard Brunauer–Emmett–Teller (BET) equation. The pore size distributions were determined using Density Functional Theory (DFT) based on statistical mechanics. The average pore diameter $D_p = 4V_p/S_{BET}$ (assuming a cylindrical shape of pores) was calculated from the BET surface area S_{BET} and pore volume V_p.

Sorption of water. The adsorption isotherms and kinetic curves of the water vapour on the silica gels at 303 K were separately determined by means of the homemade apparatus with the function of keeping constant temperature and humidity (Fig. 1). The apparatus consisted of an adiabatic sorption chamber and the temperature and humidity controlling system. A microelectronic balance was located within the adsorption chamber. Its accuracy was of 0.0001 g. The temperature and humidity of the sorption chamber can be adjusted and maintained constant by using the cycle of gas whose temperature and humidity can be adjusted. Relative humidity (RH) values were controlled by means of a dehumidifier and a humidifier, with accuracy of ±3%, and temperature can be controlled with accuracy of ±0.5°C.

Determination of Adsorption Kinetic Curve of the Water Vapor on the Silica Gels

Adsorption kinetic experiments were performed at atmospheric pressure by using the experimental apparatus shown in Fig. 1. Firstly, 2 g of fresh silica gel was introduced in airtight flask with a cover, and then the flask was placed on the microelectronic balance located in the sorption chamber. Secondly, after the temperature and RH within the sorption chamber was maintained onto some designed values by means of temperature and humidity controllers, the flask was opened and thus the adsorption experiment was start, i.e., the weight of the sample silica gel would be periodically recorded with the help of the electronic micro-balance as the adsorption occurred, and a adsorption kinetic curve giving the sorbed amounts of the water vapor on the silica gel as a function of time was measured (Fig. 2). The adsorption kinetic experiment ended when the weight of the sample got unvaried, meaning that equilibrium had been achieved (the silica gel was saturated with the water vapor). The adsorption kinetic curves were carried out at different relative humidity and on different silica gels.

Determination of Isotherm of the Water Vapor on the Silica Gels

Adsorption equilibrium experiments were carried out at atmospheric pressure in the similar way stated above. Firstly, 2 g of fresh silica gel was introduced in airtight flask with a cover, and then the flask was placed on electronic microbalance located in the sorption chamber. Secondly, after the temperature and relative humidity within the sorption chamber was maintained onto some designed values by means of temperature and humidity controllers, the flask was opened using internal mechanism and thus the adsorption of the water vapor on the silica gel began. The weight of the sample increased uninterruptedly as the adsorption carried out. When the time of the adsorption was long enough (generally, about 200 h), the weight of the sample hardly varied, which indicated the sample silica gel had been saturated with the water vapor, and hence the equilibrium sorption amount of the water vapor on the unit silica gel corresponding to some relative humidity was obtained. After finishing of adsorption equilibrium experiments under the condition of different RH, the corresponding equilibrium data of the water vapor adsorption on the unit silica gel can be obtained, and thus the plot of the equilibrium adsorbed amounts of the water vapor on silica gel versus corresponding relative humidity yielded an isotherm of the water vapor adsorption on the silica gel.

Thermal analysis. Thermal analysis was carried out using by NETZSCH STA 449C Simultaneous Thermal Analyzer. The heating rate was 20 K/min in air or nitrogen atmosphere with 25 ml/min flow rate and the temperature ranged from 303 to 523 K.

The temperature programmed desorption (TPD) experiments. The TPD experiments were conducted at different heating rates from 4 to 8 K/min. In each experiment, the sample that had saturated with the water vapour was packed in a stainless reaction tube whose inner diameter was 0.3 cm and whose packed length was 0.5 cm. Subsequently, the stainless tube was placed in a reaction furnace and then heated in the high-purity N_2 flow at a constant rate of 46.9 ml/min. The desorbed water vapour was measured by using the GC-9501 gas chromatograph with a thermal conductivity detector at the outlet of the stainless reaction tube, and effluent curves were recorded, which were called the TPD curves. According to the experimental TPD curves, the activation energy for desorption of water from three kinds of silica gels was estimated.

RESULTS AND DISCUSSION

Textural Properties of Silica Gels

The structure parameters of the silica gels derived from the basis of the nitrogen sorption data are summarized in Table 1. The table clearly indicated that A_{sg}, B_{sg}, and C_{sg} were all mesoporous silica gel. Additionally, surface modification with $CaCl_2$ and LiCl increased the average pore diameter and decreased significantly the pore volume. This was because the formation of the uniform salt layer on the silica surface probably resulted, on the one hand, in the complete filling of the small pores, and enlarging of the average pore diameter and, on the other hand, in partial filling and, hence, narrowing the large pores. It seemed that the first effect hold dominant position, and this resulted in an increase in the average pore size [10].

The Effect of Different Impregnating Salts on the Hygroscopic Properties of Silica Gels

Figure 2 showed the sorption kinetics of water vapor on C_{sg} and modified samples at 303 K and RH = 30%. It is seen that the hygroscopic abilities of the silica gels modified by $CaCl_2$, LiCl, $MgCl_2$, and $ZnCl_2$ were improved. The sorption capacity of the silica gel modified by $CaCl_2$ was the highest among the modified mesoporous silica gels and was three times higher than that of the unmodified silica gel. But the hygroscopic abilities of the silica gel modified by KCl and NaCl decreased slightly. However, their sorption rates and sorbed amounts were both lower compared with the reported for $CaCl_2$/silica gel [9] and LiBr/silica gel [6], due to the lower salt content in our work. Also others modified samples were compared with those in the [10, 11], and the similar results were obtained. From these results, it is seen that the hygroscopic abilities of $CaCl_2$ and LiCl are much stronger than those of others. Therefore, $CaCl_2$ and LiCl particularly suitable for use in modification of the mesoporous silica gels to improve their sorption capacity.

The effect of ionic radius on the water sorption at 303 K and RH = 30% is shown in Fig. 3. A tendency can be seen that the sorption capacity decreased with the increase of the ionic radius except the calcium ion. This was because that the acting forces between water and the ion with smaller ionic radius were stronger than those between water and the ion with larger ionic radius. But for the calcium ion, it was different from the other ions because the $CaCl_2$ loaded in the passageway of the silica gel more easily formed the $CaCl_2 \cdot 6H_2O$ complex, and Ca/C_{sg} had larger average pore diameter and salt content.

The Effect of $CaCl_2$ and LiCl on Sorption Isotherms and Dynamics of Water Vapor Sorption

The kinetics of water vapor sorption on modified silica gels at 303 K and RH = 10, 30, 45, and 65% are shown in Figs. 4, 2, 5, and 6, respectively. The sorption capacity and the sorption rate on C_{sg} were both improved greatly through modification by $CaCl_2$ and LiCl, which were even larger than those on A_{sg} or B_{sg} with smaller pore diameter. Especially, the sorption capacity of C_{sg} modified by $CaCl_2$ at different RH had been up to or exceeded that of A_{sg} and its sorption rate was larger than that of A_{sg} significantly. So $CaCl_2$ and

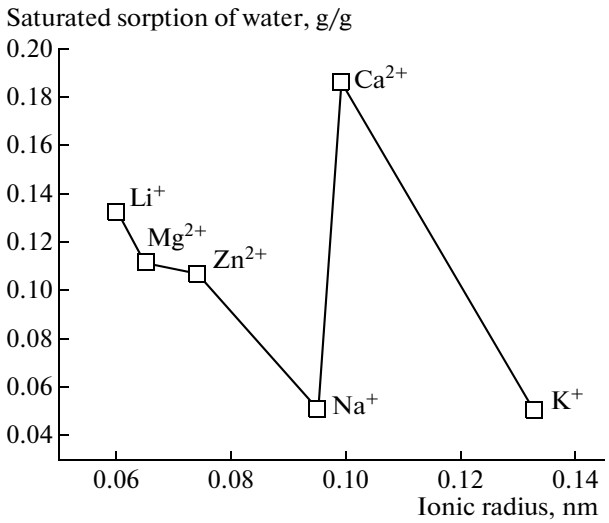

Fig. 3. The effect of ionic radius on the saturated sorption water at 303 K and RH = 30%.

LiCl are good modification agents for improving of sorption capacity and sorption rate of mesoporous silica gel at RH < 80%.

Termogravimetric (TG) curves of Li/C_{sg} and Ca/C_{sg} saturated with moisture at 303 K and RH = 30% are shown in Fig. 7. The weight loss of Li/C_{sg} and Ca/C_{sg} is equal to 15.99 and 12.70%, respectively. Then, the sorption capacity for water on the Li/C_{sg} and Ca/C_{sg} at 303 K and RH = 30% calculated from the weight loss was 19.03 and 14.94%, respectively, which were very close to the experimental values 18.69 and 14.55%. This indicated that the Li/C_{sg} and Ca/C_{sg} saturated with moisture can be regenerated completely at 413 K for 4 h.

Figure 8 shows the water vapour sorption isotherms on C_{sg}, Li/C_{sg}, and Ca/C_{sg} at 303 K. The sorption capacities of Li/C_{sg} and Ca/C_{sg} are larger than those of C_{sg} when the RH is below 80%. These results also indicated that the hygroscopic abilities of $CaCl_2$ and LiCl were great and Li/C_{sg} and Ca/C_{sg} can be used at the lower and medium RH as dehumidification materials with better sorption capacity for water vapor.

The Effect of $CaCl_2$ and LiCl on the Activation Energies of Water Vapor Desorption

TPD is a technique of surface analysis [12]. It is usually used to estimate binding energy between the adsorbate and the adsorbent, and desorption activation energy [13–16], which can be used to choose the adsorbents and estimate adsorption isotherms [15]. Basically, some kind of adsorbate is adsorbed onto the adsorbent, and then the adsorbent is heated in conditions of an inert gas flowing when the adsorbent temperature varies linearly with time. As the temperature becomes high enough, the adsorbate will desorb gradually. In a TPD experiment, a gas chromatograph (or a mass spectrometer) is used to measure the rate at which the adsorbate desorbs from the adsorbent. Using the TPD, one can know how strongly molecules are bound to the surface of the adsorbent. The TPD spectrum was a plot of desorption the rate of the adsor-

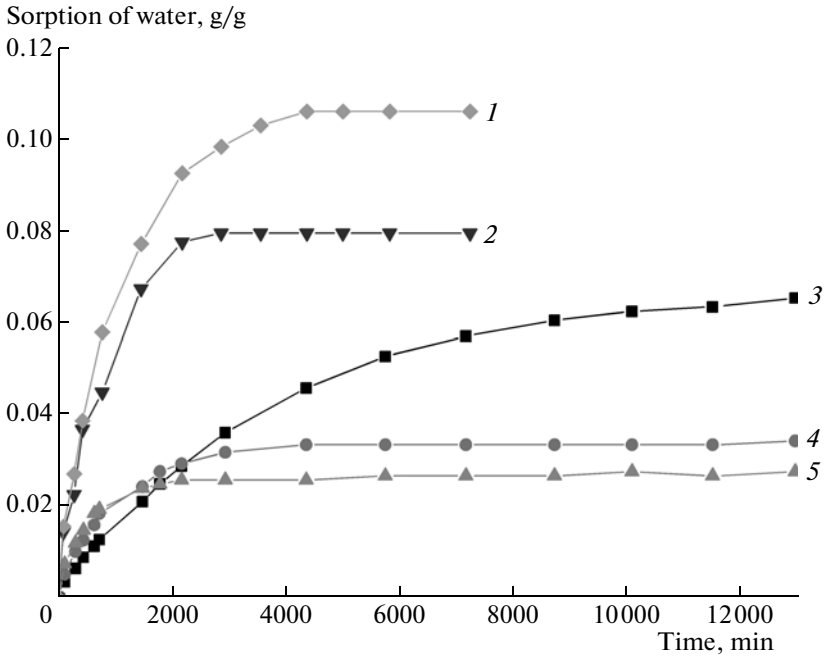

Fig. 4. The kinetics of water vapor sorption on the initial and modified silica gels at 303 K and RH = 10%: *1*—Ca/C_{sg}, *2*—Li/C_{sg}, *3*—A_{sg}, *4*—B_{sg}, *5*—C_{sg}.

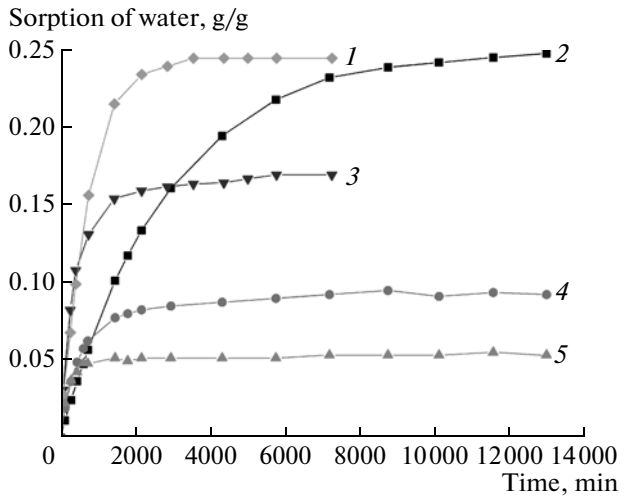

Fig. 5. The kinetics of water vapor sorption on the initial and modified silica gels at 303 K and RH = 45%: *1*—Ca/C_{sg}, *2*—A_{sg}, *3*—Li/C_{sg}, *4*—B_{sg}, *5*—C_{sg}.

bate as a function of the sample temperature [13–16]. Desorption from a surface in a TPD experiment is usually described by Polanyi–Wigner equation:

$$r_d = -\frac{d\theta_A}{dt} = k_0\theta_A^n \exp(-E_A/T). \quad (1)$$

The desorption reaction is assumed to follow first order kinetics, the activation energy for desorption can be calculated by equation

$$\ln\left(\frac{\beta_H}{RT_p^2}\right) = -\left(\frac{E_d}{RT_p}\right) - \ln\left(\frac{E_d}{K_0}\right), \quad (2)$$

where n is the order of the desorption reaction, θ—the fractional surface coverage, T_P—the peak desorption temperature, β_H—the heating rate, E_d—the activation energy for desorption, R—gas constant, and K_0—a constant that depends on the desorption kinetics.

TPD spectra of water desorption from the Li/C_{sg}, Ca/C_{sg}, and C_{sg} at different heating rates are shown in Figs. 9–11. It can be seen that there is an obvious peak in each TPD spectrum due to desorption of the water vapor from the silica gels. A gradual increase in the heating rate β_H led to an increase in the peak temperature T_P. T_P can be obtained from the TPD spectra depicted in Figs. 9–11. Knowing the values of T_P for different heating rates, it was possible to employ Eq. (2) to estimate the activation energy of water desorption from the silica gels studied. Figure 12 depicted the corresponding linear dependencies of $1/T_P$ versus $\ln(\beta_H/RT_P^2)$ for these silica gels. From the slop of these lines, activation energy E_d can be found out, and then k_0 can be also obtained from the intercept of these lines with y axis. The calculation results of the activation energy of water desorption from Li/C_{sg}, Ca/C_{sg}, and C_{sg} are listed in Table 2. The activation energies of water desorption from Li/C_{sg}, Ca/C_{sg}, and C_{sg} were 36.4, 43.1, and 26.2 kJ/mol, respectively. It means that

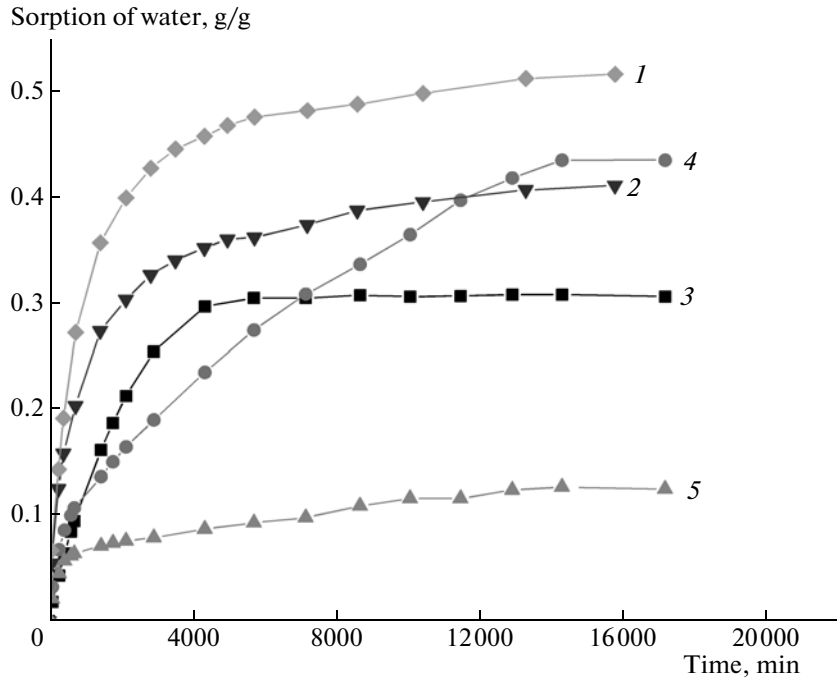

Fig. 6. The kinetics of water vapor sorption on the initial and modified silica gels at 303 K and RH = 65%: *1*—Ca/C_{sg}; *2*—Li/C_{sg}, *3*—A_{sg}, *4*—B_{sg}, *5*—C_{sg}.

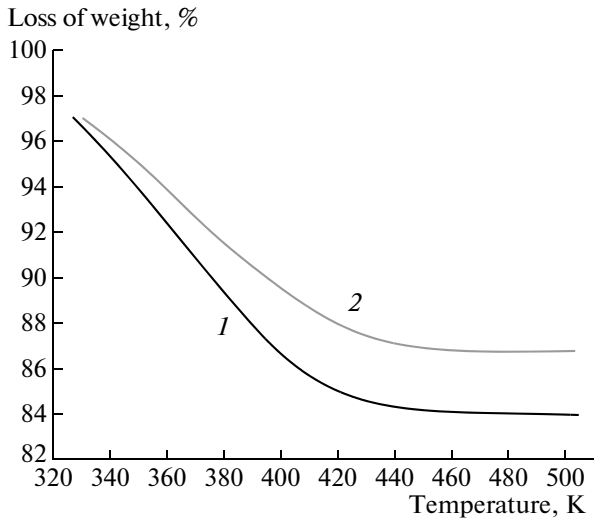

Fig. 7. TG curves of Ca/C_{sg} (*1*) and Li/C_{sg} (*2*).

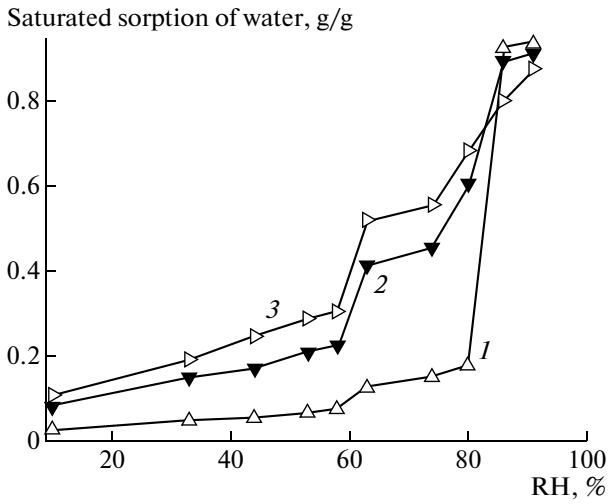

Fig. 8. The water vapour adsorption isotherms on C_{sg} (*1*), Li/C_{sg} (*2*), and Ca/C_{sg} (*3*) at 303 K.

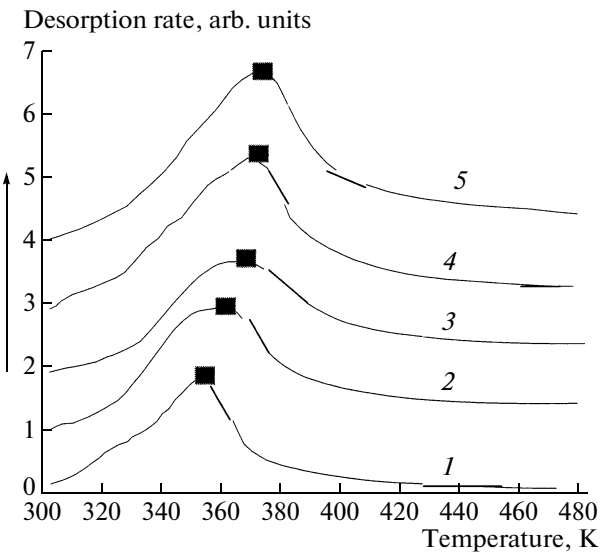

Fig. 9. TPD spectra of water on the $LiCl/C_{sg}$ at heating rates, K/min: *1*—4, *2*—5, *3*—6, *4*—7, *5*—8. T_p = 358.2, 362.2, 365.8, 369.0 and 371.8, respectively.

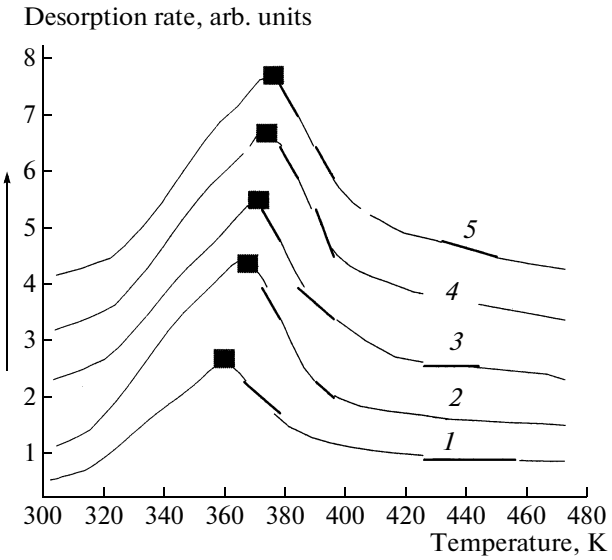

Fig. 10. TPD spectra of water on the $CaCl_2/C_{sg}$ at heating rates, K/min: *1*—4, *2*—5, *3*—6, *4*—7, *5*—8. T_p = 360.8, 366.0, 370.2, 373.9, and 376.6, respectively.

E_d on Li/C_{sg} and Ca/C_{sg} is more than that on the C_{sg}. In other words, the modification by $CaCl_2$ and LiCl not only increased the hygroscopic abilities of C_{sg}, but also increased the activation energy of water desorption from C_{sg}. This was because acting forces between water molecules and the surface of adsorbents became stronger if $CaCl_2$ and LiCl were inserted into the pore of C_{sg}. It was also seen that the E_d value on the mesoporous silica gel modified by $CaCl_2$ was much higher than that on the silica gel modified by LiCl, which was because the water molecule was polar, and the static electricity action between adsorbate and absorbent increased with the increase in the polarizabilities of the atoms on the surface of absorbent, and the polarizability of the Ca^{2+} ($\alpha_{Ca^{2+}} = 0.471$) was higher than that of Li^+ ($\alpha_{Li^+} = 0.029$) [16]. That is to say, the higher the polarizability of the ion, the stronger is the attraction forces between the ion and water. Hence the activation energy of water desorption from the silica gel modified by the ion with higher polarizability was increased. Therefore, comparing the dehumidification amounts of unit energy consumption, Li/C_{sg} was superior to Ca/C_{sg} for the practical application.

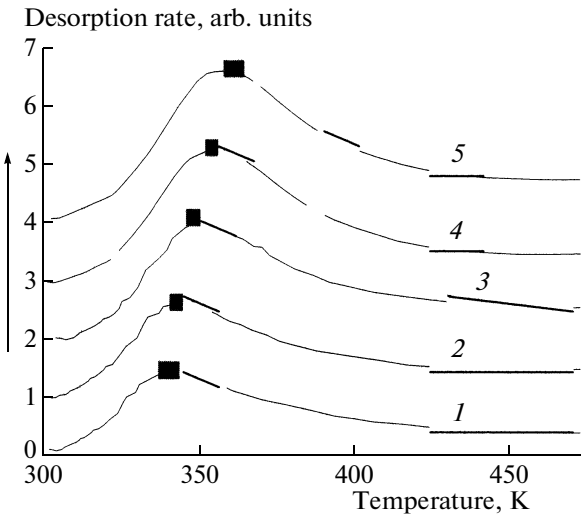

Fig. 11. TPD spectra of water on the C_{sg} at heating rates: *1*—4, *2*—5, *3*—6, *4*—7, *5*—8. T_p = 337.8, 343.7, 349.0, 355.2 and 359.3, respectively.

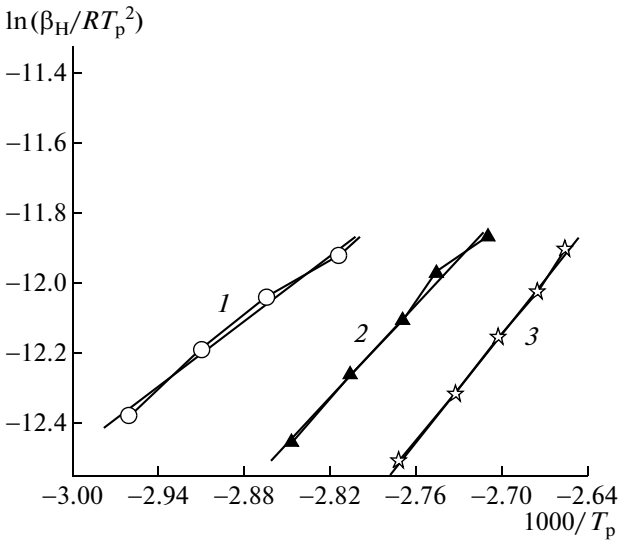

Fig. 12. Linear dependence between $\ln(\beta_H/RT_P^2)$ and $1/T_P$ for TPD of water from the silica gels: *1*—C_{sg} (E_d = 26.2 kJ/mol), *2*—$LiCl/C_{sg}$ (E_d = 36.4 kJ/mol), *3*—$CaCl_2/C_{sg}$ (E_d = 43.1 kJ/mol).

The Combination Effect of Pore Structure and Impregnating Salt on the Hygroscopic Properties of Silica Gel

Figure 13 showed the kinetics of water vapor sorption on the initial and modified silica gels with different surface areas, pore size distributions and pore volume at 303 K and RH = 30%. It was found that the sorption capacity of Ca/B_{sg} and Ca/C_{sg} was improved significantly. A comparison of saturated water sorption between initial and modified silica gels is shown in Fig. 14. The content of the saturated hygroscopic moisture in Ca/A_{sg}, Ca/B_{sg}, and Ca/C_{sg} was 0.989, 146.0, and 291.4% in comparison with those in initial silica gels, respectively. It is seen that saturated hygroscopic moisture contents after modification become much larger with increasing in the average pore diameter and pore volume of initial silica gels. So the silica gels with larger average pore diameter and pore volume were mostly suitable for the modification in order to improve their hygroscopic abilities.

Thus, the effect of the modification of silica gels by impregnating with different salts on sorption of water vapor was studied through the isotherms and dynamics of water sorption and the activation energy of water desorption. It was found that the surface modification with hygroscopic salts increased the average pore diameter and decreased significantly the pore volume. The hygroscopic abilities of the silica gels were improved through the modification by $CaCl_2$, LiCl, $MgCl_2$, and $ZnCl_2$. $CaCl_2$ and LiCl were better to modify the mesoporous silica gels. The sorption capacity and the sorption rate of C_{sg} were both improved greatly through modification by $CaCl_2$ and LiCl, which were larger than those of A_{sg} or B_{sg} with smaller pore diameter, a tendency can be seen that the sorption capacity decreased with the increase of the ionic radius except of calcium ion. The mesoporous silica gels should be applied for the medium and higher humidity control because their latent sorption capacity was largest but the rate of water uptake need to be

Table 2. Water desorption peak temperatures at different heating rates and activation energies of water desorption from modified silica gels

Silica gel	Desorption peak temperature (T_p, K) at heating rates, K/min					Activation energy of desorption E_d, kJ/mol
	4	5	6	7	8	
Ca/C_{sg}	360.8	366.0	370.2	373.9	376.6	43.1
Li/C_{sg}	358.2	362.3	365.8	369.0	371.8	36.4
C_{sg}	337.8	343.7	349.0	355.2	359.3	26.2

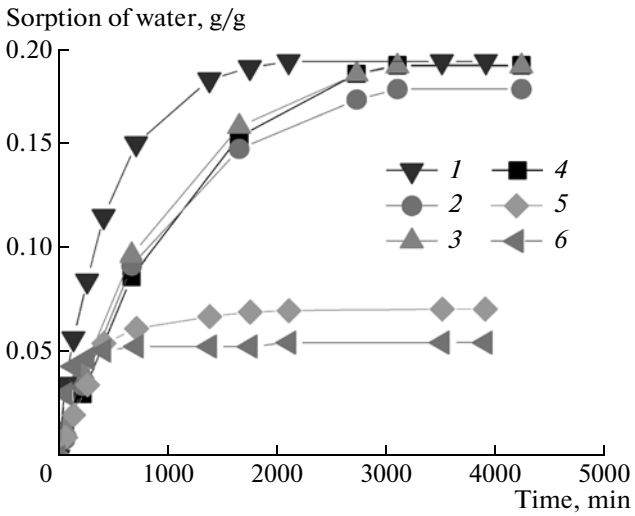

Fig. 13. The kinetics of water vapor sorption on the initial and modified silica gels at 303 K and RH = 30%: *1*—Ca/C_{sg}, *2*—Ca/B_{sg}, *3*—Ca/A_{sg}, *4*—A_{sg}, *5*—B_{sg}, *6*—C_{sg}.

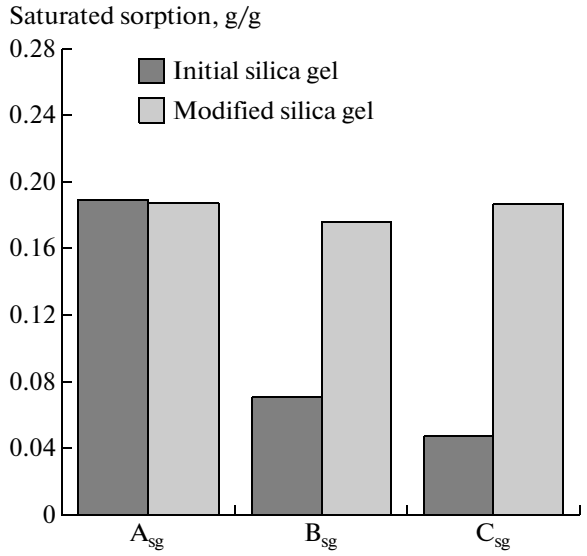

Fig. 14. A comparison of the saturated sorption water between initial and modified by 1 M $CaCl_2$ silica gels.

improved. The larger the average pore diameter and pore volume of the initial silica gels, the higher the accrual water vapor sorption rate and capacity after modification. And it was also found that the activation energy of the water desorption from the mesoporous silica gel modified by $CaCl_2$ were much higher than that from the silica gel modified by LiCl, because the polarizability of the Ca^{2+} was higher than that of Li^+.

ACKNOWLEDGMENTS

The authors would like to thank the National Natural Science Foundation of China (no. 20936001 and no. 20906034) and the key discipline construct project (211 projects, no. 2009B010100001) for financial supports.

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