

# Adsorption Equilibrium of Water on a Composite Adsorbent Employing Lithium Chloride in Silica Gel

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This paper presents a new method to develop a composite adsorbent hygroscopic salt inside silica gel which can eliminate corrosion effects. A salt solution would be formed when a conventional composite adsorbent adsorbed water and corroded the adsorbent bed quickly. The adsorption equilibrium of water on a composite adsorbent (lithium chloride confined to silica gel) was measured by the sorption isosteric method. The temperature  $T$  which ranged from (308 to 368) K was controlled by a thermostatic bath, and the absolute pressure was in the range (0 to 25 000) Pa using a membranometer. The results showed that though the isosteric enthalpy (the average isosteric enthalpy is  $42.82 \text{ kJ}\cdot\text{mol}^{-1}$ ) is lower than the conventional composite adsorbent, the new composite adsorbent still had a larger water sorption capacity ( $x = 0.702 \text{ g}\cdot\text{g}^{-1}$ ) in comparison with that of pure silica gel. The Freundlich equation, the modified Freundlich equation, and the Dubinin–Astakhov (D-A) equation are used to fit the experimental data. In our case, they are best-fitted by the D-A equation, even though it has fewer parameters than the modified Freundlich equation.

## 1. Introduction

Adsorption cooling systems have received significant attention since they use natural substances like water, methanol, and so forth as the refrigerant, which have no damage to the environment. Furthermore, adsorption chillers can be driven by solar energy and waste heat; also they have no moving parts and generate no noise or vibration. Silica gel is an amorphous, highly porous, partially hydrated form of silica. Water vapor is captured in a nonideal manner on its surface.<sup>1</sup> Silica gel–water adsorption chillers are the most promising now.<sup>2–8</sup>

A family of new working materials has been developed for refrigeration systems.<sup>9–17</sup> Composite adsorbents were synthesized by impregnating porous silica gel with hygroscopic salts (calcium chloride, lithium chloride, calcium nitrate,<sup>18</sup> lithium bromide, etc.). The results showed that the composite adsorbents had a higher sorption capacity and energy storage ability than silica gel. The widespread acceptance of the composite adsorbent is hindered by its corrosion to the adsorbent bed. In prior research, the synthesis procedure consisted of drying the pure silica at 473 K for 2 h, impregnation of the dried silica with a saturated aqueous solution at 298 K, and again drying the impregnating material at 473 K until its weight remained constant. The salt content amounted to mass fraction  $w = 0.45$ .<sup>18</sup> This method, however, was not suitable for the composite adsorbent used in industry. The salt on its surface would form a salt solution when it adsorbed water and quickly corroded the adsorbent bed. Also, after the adsorption, it was entirely possible to form a droplet which actually was salt solution, which would fall down and erode the components of the machine. So, a new method to prepare the composite adsorbent was proposed, and the adsorption equilibrium performance of the composite adsorbent–water working pair should be investigated.

It is difficult to get the water uptake cycle by silica gel since it is extremely small. Aristov et al. measured it by a thermal balance

method.<sup>9</sup> Ng et al. measured water uptake by the water liquid level change in an evaporator.<sup>19</sup> Freni et al. calculated the cooling power from the difference in the laboratory scale chiller water inlet and outlet temperatures.<sup>20</sup> However, the methods mentioned above are costly and not very convenient to test the equilibrium of water on silica gel. Xia once investigated the adsorption equilibrium of the working pair silica gel and water by the sorption isosteric method. The test rig used a thermostat-controlled tape heater to heat the connecting pipe to avoid condensation of water vapor inside the pipe.<sup>21</sup> Now a simple modified test rig has been developed, and it has a higher precision than the others. A comparison is made between pure silica gel and the composite adsorbents.

## 2. Experimental Section

**2.1. Preparation of Composite Adsorbent.** The commercial silica gel was purchased from Qingdao, China. The pore radius  $r$  of the silica gel was in the range of 7 nm and specific surface area  $S_{sp}$  in the range of  $481 \text{ m}^2\cdot\text{g}^{-1}$ , and the pore volume  $V_p$  was  $0.84 \text{ cm}^3\cdot\text{g}^{-1}$ . The composite adsorbent was prepared using the following steps. First, silica gel was put in a vacuum chamber and heated to 358 K under continuous evacuation to eliminate water and air in the silica gel. Second, lithium chloride solution ( $w = 0.30$ ) was prepared and then poured into the chamber which continued to be heated. In this process, the vacuum chamber should be kept enclosed by a liquid seal. Third, the chamber was stopped heating, and silica gel was kept in the chamber at atmospheric temperature for 24 h to be impregnated with the aqueous LiCl solution. Fourthly, the sample was taken out and dried at 393 K until reaching the dry weight. Finally, the sample was put on a strainer hanging in constant temperature/humidity equipment. The temperature and the humidity in the constant temperature/humidity equipment were set at 303 K and 90 %, respectively. The salt on the surface of the composite adsorbent would adsorb water and form a salt solution, which would then fall down through the strainer. The composite adsorbent was taken out until there was no more

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**Table 1. BET Surface Area  $S_{sp}$ , Pore Volume  $V_p$ , and Average Pore Diameter  $d_{av}$  of the Pure Silica Gel and the Composite Adsorbent**

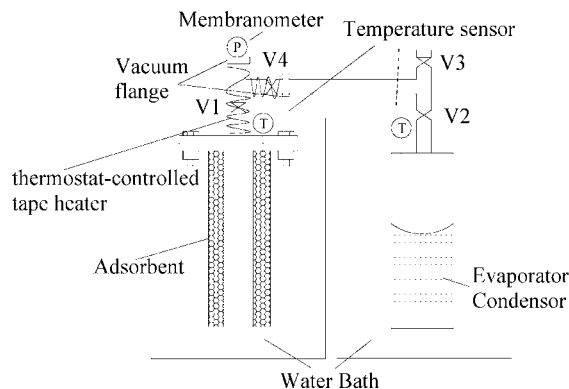
adsorbent	$V_p$ $\text{cm}^3 \cdot \text{g}^{-1}$	$S_{sp}$ $\text{m}^2 \cdot \text{g}^{-1}$	$d_{av}$ nm
pure silica gel	0.844	480.765	7.02
composite adsorbent	0.613	350.424	7.00

formation of salt solution and then dried at 393 K to reach a dry weight. The salt content amounted to 0.13 w.

When the composite adsorbent was prepared, a comparison was made between this material and a material prepared by the traditional method.<sup>18</sup> A portion of 50 g of these two samples was placed on the stainless steel screen which was hung in the constant temperature/humidity equipment for about 7 days. The temperature was set at 303 K, and the humidity was set at 90 %. The results showed that, between these two stainless steel screens, the one placed with composite adsorbent prepared by the conventional method was corroded seriously, while the other one was much better.

**2.2. Porous Structure.** The porous structures of the new composite adsorbent and pure silica gel were studied, and the results are presented in Figure 2 and Table 1. Figure 2 shows the pore volume distribution curves obtained from the  $\text{N}_2$ -desorption. The BET surface area  $S_{sp}$ , pore volume  $V_p$ , and average pore diameter  $d_{av}$  are listed in Table 1.  $V_p$  and  $S_{sp}$  decreased after employing lithium chloride in silica gel. The pore diameter  $d_{av}$  does not change much. It can be explained by the research of the pore structure of the composite adsorbent by Liu and Wang,<sup>22</sup> the pore structure is tapered with either one end open or both ends open. As a result, the pores fill with the salt particles, and some narrow ones are blocked by it. So, from Figure 2 and Table 1,  $V_p$  and  $S_{sp}$  of the new composite adsorbent are smaller than pure silica gel. According to Simonova,<sup>18</sup> the character of the salt assembling during its deposition on the silica surface can be very complex, so the average pore size can both increase and decrease. In this experiment, the average pore size remains unchanged.

**2.3. Experimental Procedure.** The test rig was similar to that of the early work by Xia et al.<sup>21</sup> It still consisted of one absorber, one evaporator/condenser, measuring cells, and a thermostat-controlled tape heater. The differences were that it had four valves (shown in Figure 1) and two vacuum flanges. The measuring cells were two four-wire PT1000 to measure the temperatures of the composite sample and the evaporating temperature. One membranometer measured the pressure from (0 to 25 000) Pa with a precision of 50 Pa. The equilibrium water uptake was measured by a Sartorius electronic balance BS2202S with a precision of 0.01 g. A thermostat-controlled heater was used to heat the pipe and valves to avoid the condensation of water vapor inside the pipe. Temperatures of the absorber and the evaporator were controlled by two constant-temperature water baths. The sample was placed into the absorber which was kept at 308 K by the water bath, and the mass  $m_0$  of the sample and absorber was measured to be 106.4 g. The test rig would be evacuated by a vacuum pump. Then valve V2 was kept open for 8 h, so that the water vapor could be adsorbed by the sample. After the sample was saturated with water, valve V4 was closed, and the absorber was disconnected from the evaporator. The sample in the vacuous absorber was heated to a fixed temperature. One hour later, the water equilibrium pressure was measured. Different temperatures of the absorber were set, and the corresponding equilibrium pressures were obtained. Finally the mass  $m_1$  of the sample and the absorber was measured. As the density of the water vapor

**Figure 1.** Schematic diagram of the modified test rig of Xia et al.**Table 2. Equilibrium Pressure ( $P(T_a)$ ) of Water on the Composite Adsorbent versus Water Uptake ( $x$ ) under Different Adsorption Temperatures ( $T_a$ )**

$x$ $\text{g} \cdot \text{g}^{-1}$	$T_a$ $^{\circ}\text{C}$	$P$ Pa
0.077	39.5	500
0.077	49.8	800
0.077	60.2	1250
0.077	69.7	1900
0.077	79.9	2800
0.077	89.3	4200
0.192	34.8	750
0.192	44.2	1300
0.192	55.2	2150
0.192	64.8	3450
0.192	75.1	5600
0.192	84.5	8850
0.192	94.2	13650
0.241	34.8	1100
0.241	50.2	2400
0.241	64.7	5000
0.241	79.8	9800
0.241	89.4	15100
0.289	34.6	1600
0.289	49.9	3550
0.289	64.5	7150
0.289	79.7	13950
0.289	89.3	21300
0.39	34.7	2621
0.39	49.9	5370
0.39	64.7	10600
0.39	79.9	19900

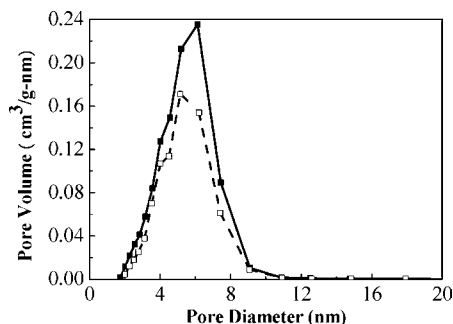
is very small, the effect of the small dead volume can be neglected. The water uptake at equilibrium conditions was the following:  $x = (m_1 - m_0)/m_0$ . The temperature of the evaporator was kept at (280, 288, 293, 298, and 303) K by the water bath for 8 h separately. The water uptake changed corresponding to the temperature of the evaporator.

**2.4. Adsorption Equations.** The Freundlich equation, Töth equation, Henry equation, Dubinin–Astakhov (D-A) equation, and Dubinin–Radushkevich (D-R) equation are mainly used to describe the equilibrium of physical adsorption. The Clausius–Clapeyron equation is often used to describe chemical adsorption. In this experiment, the Freundlich equation, D-A equation, and modified Freundlich equation were used to fit the data and compared to the results of Xia et al. The Clausius–Clapeyron equation was also used. The data obtained from the experiment are shown in Table 1.

**1. Freundlich Equation.** The Freundlich equation is the most widely used to describe physical and chemical adsorption.

$$x = x_0 \left( \frac{P(T_a)}{P(T_w)} \right)^{1/n} \quad (1)$$

where  $x$  is the adsorbed water uptake at equilibrium conditions,  $x_0$  is defined as the maximum water uptake by the adsorbent,  $n$



**Figure 2.** Pore volume distribution curves: ■, pure silica gel; □, composite adsorbent.

is a constant denoted by the distribution of the pore radius of the adsorbent, and  $T_w$  and  $T_a$  are the temperatures of the saturation water vapor and adsorbent, respectively.

**2. Dubinin–Astakhov (D-A) Equation.** The D-A equation is often used to describe physical adsorption.

$$x = x_0 \exp\left(-k \left(\ln \frac{P(T_w)}{P(T_a)}\right)^n\right) \quad (2)$$

where  $k$  and  $n$  are constants and were determined by a fitting process to the experimental data.

Table 2 shows that  $x_0$  of the composite adsorbent is larger, while  $n$  remains virtually unchanged. It indicates that the composite adsorbent prepared by the new method has a larger uptake of adsorbed water, and the average radius of the pores of the adsorbent remains the same. The reason is that the salt had been impregnated in the pores of the silica gel, and it enhances the water uptake capacity of the silica gel. The standard error of the estimates is defined as follows

$$\sigma = \sqrt{\frac{\sum (y - y')^2}{N}} \quad (3)$$

where  $\sigma$  is the standard error,  $y$  is the measured value,  $y'$  is the predicted value, and  $N$  is the number of pairs of values. The numerator is the sum of the squared difference between the measured value and the predicted value.

**3. Modified Freundlich Equation.** In Xia's research, the modified Freundlich equation agreed well with the experimental points. It was also used in this experiment.

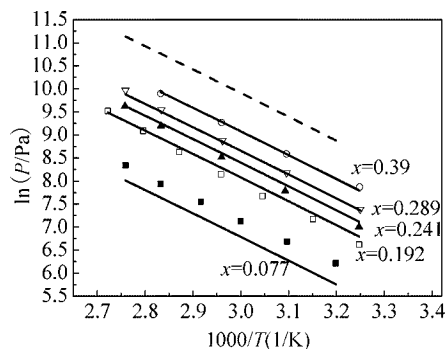
$$x = A(T_a) \left[ \frac{P(T_a)}{P(T_w)} \right]^{B(T_a)} \quad (4)$$

$$A(T_a) = A_0 + A_1 T_a + A_2 T_a^2 + A_3 T_a^3$$

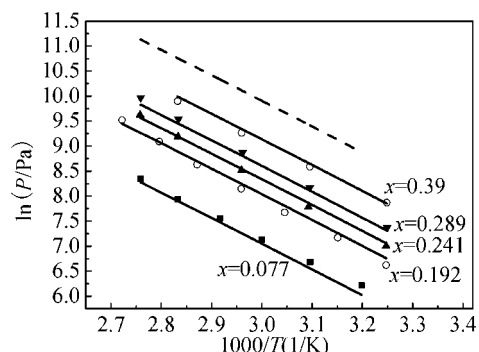
$$B(T_a) = B_0 + B_1 T_a + B_2 T_a^2 + B_3 T_a^3$$

where  $A_0$  to  $A_3$  and  $B_0$  to  $B_3$  are constants and determined by a least-squares fit to the experimental data. In his experiment, the fitted parameters were  $A_0 = 7.6032$ ,  $A_1 = -0.09554 \text{ K}^{-1}$ ,  $A_2 = 3.8305 \cdot 10^{-4} \text{ K}^{-2}$ ,  $A_3 = -4.717896 \cdot 10^{-7} \text{ K}^{-3}$ ,  $B_0 = -27.3789$ ,  $B_1 = 0.22503 \text{ K}^{-1}$ ,  $B_2 = 5.9629 \cdot 10^{-4} \text{ K}^{-2}$ , and  $B_3 = 5.2253 \cdot 10^{-7} \text{ K}^{-3}$ .

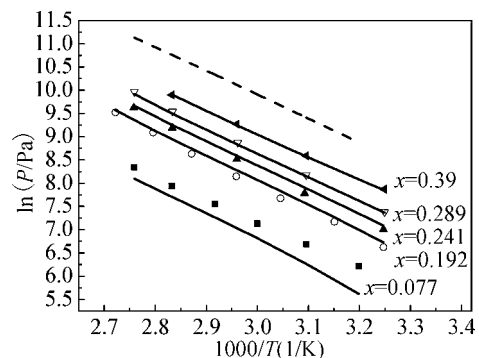
According to the research of Aristov et al. about silica gel modified by calcium chloride, when  $x$  is about  $0.11 \text{ g} \cdot \text{g}^{-1}$ , it does not depend on the temperature that indicates a monovariant type of sorption equilibrium while  $x$  is more than  $0.11 \text{ g} \cdot \text{g}^{-1}$ ; the equilibrium is always divariant.<sup>9</sup> In this experiment, Figures 2 to 5 show that when  $x = 0.077 \text{ g} \cdot \text{g}^{-1}$ , the data are not fit very well. This indicates that the adsorption of the composite adsorbent is not just simply physical adsorption. When the water uptake is relatively small, chemical adsorption will occur. Table 3 shows



**Figure 3.** Equilibrium pressure ( $P$ ) of water on the composite adsorbent as a function of temperature ( $T$ ) and water uptake ( $x$ ). Dashed line indicates the saturation line of water vapor. The solid lines denote the fittings with the Freundlich equation.



**Figure 4.** Equilibrium pressure ( $P$ ) of water on the composite adsorbent as a function of temperature ( $T$ ) and water uptake ( $x$ ). Dashed line indicates the saturation line of water vapor. The solid lines denote the fittings with the DA equation.



**Figure 5.** Equilibrium pressure ( $P$ ) of water on the composite adsorbent as a function of temperature ( $T$ ) and water uptake ( $x$ ). Dashed line indicates the saturation line of water vapor. The solid lines denote the fittings with the modified Freundlich equation.

**Table 3. Fitted Parameters of the Freundlich and D-A Equations and Standard Error of the Estimate**

equation	$x_0$	$n$	$k$	standard error $\sigma$
Freundlich (silica gel) <sup>21</sup>	0.444	1.342		0.023
Freundlich (composite adsorbent)	0.702	1.414		0.016
D-A (silica gel) <sup>21</sup>	0.348	1.609	0.449	0.019
D-A (composite adsorbent)	0.489	1.604	0.342	0.014

that the data of the experiment can be best fitted by the Dubinin–Astakhov equation, while in the case of pure silica gel, the modified Freundlich equation is the most appropriate equation (Table 4).

**4. Clausius–Clapeyron Equation.** The Clausius–Clapeyron equation is often used to calculate the isosteric enthalpy.

$$\ln P = \Delta H_{is}/RT - \Delta S/R \quad (5)$$

where  $\Delta H_{is}$  and  $\Delta S$  is the isosteric enthalpy and entropy of the adsorption.  $R$  is the gas constant. In physical adsorption, the



**Table 4. Comparisons of the Water Uptake Calculated by the Freundlich, D-A, and Modified Freundlich Equations ( $x_{\text{Freundlich}}$ ,  $x_{\text{DA}}$ , and  $x_{\text{Modified, Freundlich}}$ ) with the Experimental Water Uptake ( $x$ ) under the Equilibrium Temperatures ( $T$ ) and the Equilibrium Pressures ( $P$ )**

$T/K$	$P/\text{Pa}$	$(x_{\text{Freundlich}} - x)/x$	$(x_{\text{DA}} - x)/x$	$(x_{\text{modifiedFreundlich}} - x)/x$
39.5	500	38.2 %	21.4 %	48.2 %
49.8	800	32.4 %	14.3 %	34.5 %
60.2	1250	27.6 %	8.4 %	25.6 %
69.7	1900	27.0 %	7.7 %	23.3 %
79.9	2800	23.5 %	3.4 %	18.5 %
89.3	4200	26.7 %	7.3 %	19.5 %
34.8	750	-11.5 %	-10.6 %	-6.5 %
44.2	1300	-8.5 %	-7.1 %	-6.4 %
55.2	2150	-11.3 %	-10.4 %	-10.8 %
64.8	3450	-9.5 %	-8.3 %	-9.6 %
75.1	5600	-7.0 %	-5.3 %	-7.7 %
84.5	8850	-1.8 %	0.8 %	-4.1 %
94.2	13650	2.7 %	6.0 %	-3.5 %
34.8	1100	-7.5 %	-3.7 %	-5.2 %
50.2	2400	-9.3 %	-5.6 %	-8.3 %
64.7	5000	-6.0 %	-2.0 %	-5.1 %
79.8	9800	-4.0 %	0.1 %	-4.4 %
89.4	15100	-0.2 %	4.1 %	-3.4 %
34.6	1600	1.3 %	4.4 %	0.8 %
49.9	3550	0.9 %	4.1 %	1.8 %
64.5	7150	1.7 %	4.8 %	3.6 %
79.7	13950	3.1 %	6.1 %	3.8 %
89.3	21300	6.5 %	9.1 %	3.8 %
34.7	2621	6.0 %	1.0 %	1.6 %
49.9	5370	0.2 %	-2.7 %	1.0 %
64.7	10600	-1.1 %	-3.6 %	1.9 %
79.9	19900	-2.3 %	-4.5 %	-0.7 %

**Table 5. Isothermic Enthalpy and Standard Entropy of the Water Sorption on the New Composite Adsorbent**

$x$	$-\Delta H_{\text{is}}$	$-\Delta S$	
$\text{g} \cdot \text{g}^{-1}$	$\text{kJ} \cdot \text{mol}^{-1}$	$\text{J} \cdot \text{mol}^{-1} \cdot \text{K}$	standard error $\sigma$
LiCl ( $w = 0.13$ )/SiO <sub>2</sub> -water			
0.077	40.02	179.51	0.011
0.192	45.47	202.54	0.022
0.241	44.26	201.88	0.013
0.289	43.62	202.96	0.011
0.39	40.75	197.64	0.013
water	40.6		

isosteric enthalpy is actually made up of two main parts. One was the heat of condensation of the adsorbate, and the other was the surface energy caused by the attractive force between the adsorbate and the adsorbent. The average value  $\Delta H_{\text{is}} = -42.82 \text{ kJ} \cdot \text{mol}^{-1}$ , which is the excess latent heat of the adsorbate. The salt in the silica gel which enhances the water uptake capacity also improves the isosteric enthalpy, for the salt caused by a chemical reaction. As the salt content is lower than the conventional composite adsorbent, the isosteric enthalpy of the new composite adsorbent is also lower than the conventional composite adsorbent. It changes according to the different water uptake  $x$  (Table 5).

### 3. Conclusion

A new method to deal with composite adsorbents was proposed. This method could diminish the corrosion effect of the salts to the adsorbent bed and the components of the chiller. Four equations, the Freundlich equation, D-A equation, modified Freundlich equation, and Clausius-Clapeyron equation were used to fit the data obtained from this experiment. Comparisons of the water uptake between the data calculated by these equations and the data measured in the experiment were made. It was clear that the D-A equation agrees best with the experimental data. The maximum water uptake  $x_0$  of the composite adsorbent was larger than that of pure silica gel. If

the water uptake capacity was  $0.702 \text{ g} \cdot \text{g}^{-1}$ , the isosteric enthalpy of water sorption  $\Delta H_{\text{is}}$  was  $42.82 \text{ kJ} \cdot \text{mol}^{-1}$ . The energy storage capacity would be  $1.67 \text{ kJ} \cdot \text{g}^{-1}$ . It could be considered as a promising adsorbent for application in adsorption chillers.

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