Formulation of Water Equilibrium Uptakes on Silica Gel and Ferroaluminophosphate Zeolite for Adsorption Cooling and Desalination Applications

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This paper studies the water equilibrium uptake on two type of adsorbents, namely silica gel RD powder and Zeolite FAM Z01. A novel adsorption isotherm model is proposed to regress the experimental data of the both adsorbents. For the silica gel-water pair, the proposed model generates more precise data regression than the classic DA and Tóth equations. In the case of the zeolite-water pair, the new model well fits its unique S-shape isotherms at various adsorption temperatures. The linear behavior at both low and high relative pressure regions, and the sharp rise in between the two regions are accurately captured. The model produces R^2 value 0.997 and NRMSE error 3.15% for the former pair, and 0.997 R^2 and 6.05% error for the zeolite-water pair in the experiment range.

1. Introduction

In recent decades, the boom of economy from the third world countries has sparked an increasing demand of energy worldwide. One of the major sectors aggravate this is cooling. Isaac and van Vuuren[1] estimated that the air conditioning demand from the residential sectors will grow 40 folds this century. To meet the tremendous rise of cooling needs and meanwhile minimize consequent energetic and environmental issues, green solutions are surged, amongst which the thermally-driven adsorption cycle has been regarded as one of the most attractive technologies[2,3].

An adsorption chiller utilizes the characteristics of adsorbent-adsorbate pairs at different thermodynamic conditions to achieve vapor compression. It requires only low-grade heat as energy input. As such, waste heat from industrial power plants or renewable energy like solar and geothermal are desired without extra CO_2 emission. Moreover, there are no major moving parts constructed in the system. Only minimum maintenance is needed. Furthermore, the chiller uses environmentally benign adsorbates as working fluids. It eliminates the corresponding deterioration of environment caused by ozone- depleting refrigerants[4-8].

Extensive work has been performed in the literature to study the feasibility of various adsorbent-adsorbate pairs to perform adsorption cooling cycle. Silica gel-water is one of the most common pairs that have received considerable attention. Adsorption chillers using this pair was investigated in the early years by Chua et al.[9-11], Wang. et al.[12], Tahat[13], Liu et al.[14], etc. Recently, 25% improvement on the COP of the chiller was achieved by Daou et al.[15]. and Saha et al.[16] to impregnate $CaCl_2$ into silica gel. Similar developments by adding other inorganic salt were studied by Aristov and co-workers[17-18]. Silica gel-water adsorption chillers usually operate at heat source temperature 55 °C – 90 °C with the COP ranged from 0.2 to 0.65. Due to the excellent affinity of silica gel to water, Ng and his research group[19-22] innovatively convert the adsorption cooling cycle to desalination. Recent achievement from the group is a creative hybridization of adsorption and multi-effect desalination (MED), which breakthrough lower temperature limit of the conventional MED to below ambient condition[23-24]. Another frequently used adsorbent is zeolite. Due to variety of molecular structures, many synthetic types were developed, such as zeolite 4A, 5A,10X and 13X, etc.[25]. The zeolite-water[26-28] chillers generally require higher heat source temperatures than the silica gel-water type.

To design an adsorption chiller, it is indispensible to understand the adsorption characteristics of the adsorbent-adsorbate pairs. The equilibrium uptake of the

adsorbate on the adsorbent must be accurately measured and mathematically formulated. Silica gel-water adsorption isotherms belong to Type I category according the classification of Brunauer et al.[29]. They are frequently regressed by Tóth[30] and Dubinin-Astakhov (DA)[31-33] models. In this work, a novel water adsorbent zeolite, namely Functional Adsorbent Material (FAM) Z01, is studied[34,35]. It owns a unique S-shape water adsorption isotherm pattern that is classified into Type V. The water uptake increases linearly both when relative pressure is small (Henry's region) and approaching unity, and rise sharply in between the two linear regions. Neither Tóth nor DA is suitable for this pattern. In the recent literature, attempts have been made to fit such isotherms. Sun and Chakraborty[36,37] developed two new models to generate the S-shape with four, and three regression parameters, respectively. However, the two models yield null uptake in the Henry's region, and saturated uptake in the high relative pressure region. In addition, only the data of one temperature is demonstrated for regression. The ability of the models to fit the isotherm of different temperatures remains unknown. A hybrid Henry and Sips isotherm model developed by Kim et al. [38], in contrast, well captures the trend in the lower end. Yet the formulation requires nine parameters to regress, and still does not effectively avoid the saturation problem at high relative pressures.

In the present work, a novel model is proposed to fit the adsorption isotherms of the zeolite FAM Z01-water pair. As demonstrated below, the model fits accurately the three regions of the isotherms at all experimental conditions. Moreover, the model is not restricted to Type V category. It is able to generate more precise data regression for Type I silica gel-water pairs than the classic DA and Tóth equations. Furthermore, only four parameters are needed to regress for the different types of isotherms.

2. Adsorbents

This work studies the water equilibrium uptake characteristics of two adsorbents, silica gel type RD powder and zeolite FAM Z01. The silica gel (Composition: $SiO_2 \cdot nH_2O$) accommodates the adsorbed water molecules mainly by the microporous structures on the solid surface. The zeolite FAM Z01 (Composition: $Fe_xAl_yP_zO_2 \cdot nH_2O$, x = 0.02 – 0.10, y = 0.35 – 0.5, z = 0.4-0.6, n = 0 – 1), however, uses its inherent nano-window structures on the molecular framework. The framework is one-dimensional AFI type. Its nano-windows have diameter 0.73nm which permits free migration of the smaller size water molecules. The zeolite FAM Z01 sample employed in the present work is supplied by Mitsubishi Plastics, Inc. under the brand AQSOA™. The thermophysical properties of the two adsorbents are summarized in Table 1[25,34,39].

Table 1. Thermophysical properties of the silica gel RD powder and Zeolite FAM Z01.

Properties	Silica Gel RD Powder	Zeolite FAM Z01
BET surface area [m²/g]	573	147.3
Average particle diameter [μm]	70	100
Porous volume [ml/g]	0.39	0.071
Apparent density [kg/m³]	800	600-700
Thermal conductivity [W/m.K]	0.198	0.113 (30 °C)
Heat of adsorption (H_2O) [kJ/kg of H_2O]	2800	3110 (25 °C)
Specific heat capacity [kJ/kg.K]	0.921	0.805 (30 °C)

3. Experimental setup and procedures

The water adsorption isotherms of the silica gel RD powder and zeolite FAM Z01 are measured using HYDROSORB analyzer manufactured by Quantachrome Instruments. Its working principle is based on constant volume variable pressure (CVVP) method. The water equilibrium uptake on the adsorbent during adsorption / desorption process is determined by calculating the amount of water vapor entering / leaving the sample cell and filling the void space around the adsorbent in the cell through pressure change. Only initial and equilibrium pressure readings are used in the calculation. Data acquisition and reduction are performed by Quantachrome HydroWin software.

A schematic representation of HYDROSORB analyzer is shown in Figure 1. In the analyzer, two major components, a water reservoir and a manifold, are located inside a heater oven that maintains internal temperature at 100 °C. In other words, the water vapor generated in the machine prior entering the sample cell is at the same temperature. Before any sample is tested, an empty sample cell to be utilized in the experiment is calibrated to determine the volume of the cell and the manifold at every experimental adsorption temperature. For a particular adsorbent sample, the experimental procedures of its water isotherm measurement are described as follows.

First of all, the weight of the empty sample cell and the adsorbent sample are measured by Ohaus E12140 mass balance with accuracy ±0.1 mg. Before adsorption analysis, the sample cell is degassed by wrapping the degasser heater and vacuumed by the Edwards type 18 vacuum pump. The degassing is 6 – 8 hours at 120 °C. The dry weight of the sample is again measured after the degassing as input for the data reduction. During the adsorption analysis, the sample cell is partially immersed into a temperature dewar which sets the adsorption temperature and is maintained by Julabo F12 water

circular with $\pm 0.1^{\circ}$C temperature control accuracy. The sample cell is vacuumed again and then dosed by water vapor stored in the manifold till equilibrium is achieved between the adsorbent and water vapor at preset pressure range. The pressure of the manifold and the sample cell is measured by a 100 torr transducer with resolution 0.01% of full scale. High purity nitrogen (99.99%) is used for backfill purpose during the measurement.
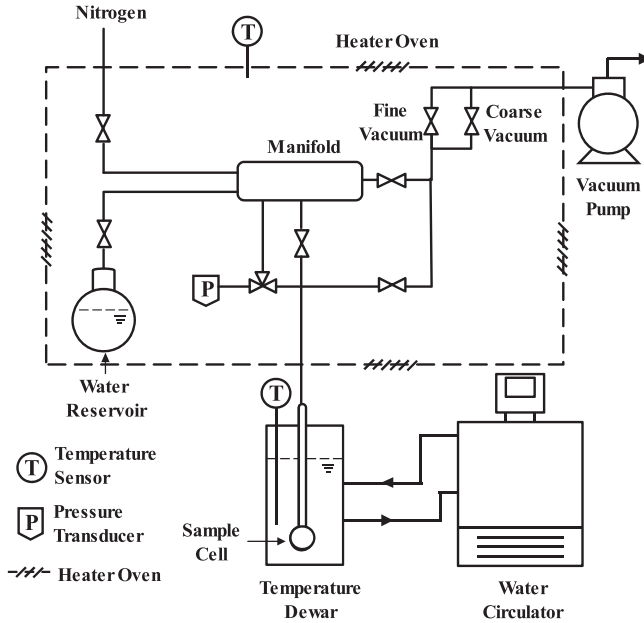

Fig. 1. Schematic representation of HYDROSORB analyzer.

4. Data reduction

Data reduction in the HYDROSORB analyzer is as follows. Firstly, the mass of water vapor dosed from the manifold into the sample cell (denoted as m_1) is calculated. The cell is partially immersed into a temperature dewar, named as cold zone. The other heated part is named as warm zone. When the cell is empty and the pressure equalization is achieved between the manifold and the sample cell, according to the mass conservation,

$$m_1 = m_H + m_C \tag{1}$$

where m_H and m_C are the mass of water vapor in the warm and cold zone, respectively. Assuming both temperature zones are apportioned without temperature gradient in between, invoking ideal behavior in the warm zone, and non-ideality correction for the cold zone, the above equation becomes,

$$m_1 = \frac{\Delta P_H}{R}\frac{V_H}{T_H} + \frac{\Delta P_C}{R}\frac{V_C\left(1+\alpha P\right)}{T_C} \tag{2}$$

in which the subscript H and C denote warm and cold zone, correspondingly; P, T and V represent pressure, temperature and volume; R is the universal gas constant. The Greek letter α is a non-ideality correction factor, and has value 3 x 10^{-5} /torr for water vapor[40]. Since the pressure in the sample cell is equal anywhere, that is,

$\Delta P_H = \Delta P_C = \Delta P$, rewrite Equation 2,

$$m_1 = \frac{\Delta P}{R}\left[\frac{V_H}{T_H} + \frac{V_C\left(1+\alpha P\right)}{T_C}\right] \tag{3}$$

When the adsorbent with mass M and skeletal density ρ is present in the sample cell, the mass of water vapor leaving from the manifold to the sample cell after pressure equalization but before adsorption occurring is,

$$m_1 = \frac{\Delta P}{R}\left[\frac{V_H}{T_H} + \frac{\left(V_C - M/\rho\right)\left(1+\alpha P\right)}{T_C}\right] \tag{4}$$

At the moment adsorption starts to take place, the sample cell is isolated from the manifold. If equilibrium between the water vapor and the adsorbent sample is not achieved within the preset pressure range, m_1 is recorded as the mass of water vapor adsorbed by the adsorbent at that particular dose action. If the equilibrium is established within the preset pressure range, Equation 4 can also be used to determine the mass of water vapor adsorbed, by replacing ΔP with the pressure difference between the start of the adsorption and the end equilibrium ΔP_e, and P with the final equilibrium pressure P_e. The total mass of water vapor adsorbed by the adsorbent at preset pressure after n times dose actions is given by,

$$m = \sum_{i=1}^{n-1} m_i + m_n$$

$$= \sum_{i=1}^{n-1}\frac{\Delta P_i}{R}\left[\frac{V_H}{T_H} + \frac{\left(V_C - M/\rho\right)\left(1+\alpha P_i\right)}{T_C}\right]$$

$$+ \frac{\Delta P_e}{R}\left[\frac{V_H}{T_H} + \frac{\left(V_C - M/\rho\right)\left(1+\alpha P_e\right)}{T_C}\right] \tag{5}$$

5. Isotherm regression

The water adsorption isotherms of the adsorbent presented in this work are from two categories. The silica gel-water pair belongs to Type I category, and the zeolite FAM Z01-water is Type V. The Type I isotherms can be fitted by classical Dubinin-Astakhov (DA) and Tóth models as expressed below.

DA model:

$$\frac{q^*}{q_0} = \exp\left[-\left(\frac{RT}{E_c}\ln\frac{P_s}{P}\right)^{\gamma}\right] \tag{6}$$

where, q^* is the equilibrium adsorption uptake at temperature T and pressure P; q_0 denotes the limiting uptake of the adsorbate at the saturation pressure P_s of the temperature T; R, E_c and γ represent the universal gas

constant, characteristic energy; and exponential constant, respectively.

Tóth Model:

$$\frac{q^*}{q_0} = \frac{K_T P \exp\left(\dfrac{Q_{st}}{RT}\right)}{\left\{1+\left[K_T P \exp\left(\dfrac{Q_{st}}{RT}\right)\right]^t\right\}^{1/t}} \quad (7)$$

in which, Q_{st} is the isosteric heat of adsorption; t is surface heterogeneity factor, and K_T is the Tóth pre-exponential constant.

In the present work, a novel model is proposed to fit the adsorption isotherms of the silica gel and zeolite FAM Z01-water pairs. The mathematical form of the novel model is expressed below as,

$$\frac{q}{q_0} = \frac{A\phi \exp(\beta \dfrac{P}{P_s})\dfrac{P}{P_s}+C\dfrac{P}{P_s}}{\left\{1+\phi \exp(\beta \dfrac{P}{P_s})\dfrac{P}{P_s}\right\}^t} \quad (8)$$

and,

$$\beta = \exp\left(\frac{E_c}{RT}\right) \quad (9)$$

$$A = \frac{\left[1+\phi \exp(\beta)\right]^t - C}{\phi \exp(\beta)} \quad (10)$$

where, the alphabet ϕ, C, are constants; Other alphabets have their usual meanings. The two constants, together with E_c and t are the parameters required to calculate in the regression process. The equation satisfies the conditions of the adsorption thermodynamics as follows,

1) When pressure is far below saturation pressure, the equation reduces to Henry's linear relation, i.e.,

$$\text{at } \frac{P}{P_s} \Box \ 1, \ \exp(\beta \frac{P}{P_s}) \approx 1 \ \text{ and, } \ \frac{q}{q_0} = \left(A\phi + C\right)\frac{P}{P_s}$$

2) When pressure is approaching to zero, the adsorbent has null uptake,

$$\text{at } \frac{P}{P_s} \to 0, \text{ then, } \lim_{P/P_s \to 0} \frac{q}{q_0} = 0$$

3) When pressure is close to saturation, the adsorbate equilibrium uptake reaches its limiting amount at saturation conditions,

$$\text{at } \frac{P}{P_s} \to 1, \text{ then, } \lim_{P/P_s \to 0} \frac{q}{q_0} = 1$$

6. Results and discussion

6.1. Comparison with literature data on silica gel isotherms

The experimental results of the silica gel RD-water adsorption from the current CVVP approach based Hydrosorb analyzer is benchmarked with the Tóth regression in literature data provided by Ng *et al.*[39], as shown in Figure 2. It is noted that the current type RD silica gel is in powder form grinded originally from the granule of the same type used in the literature. The surface feature and the pore distribution of the powder may differ from the particle type, though the adsorption mechanism remains unchanged. As expected, the isotherm of the current type RD powder deviates slightly with the previous work. A reasonable coefficient of determination (R^2) value of 0.97 and normalized root mean square error (NRMSE) of 13.87 % are noticed.
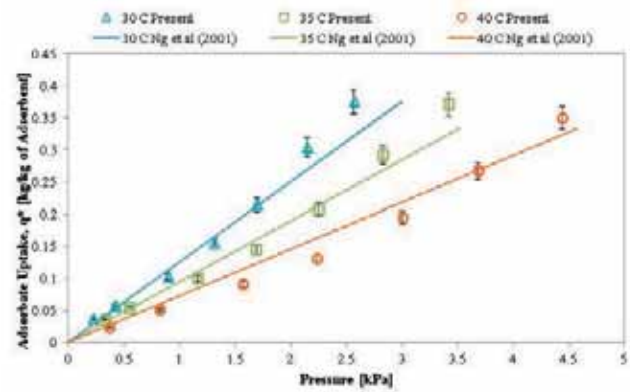

Fig. 2. Comparison of the experimental water adsorption isotherms on the present silica gel type RD powder with the Tóth regression relation on the type RD granules provided by Ng *et al.*[39]. The present data are displayed with 5% error bar.
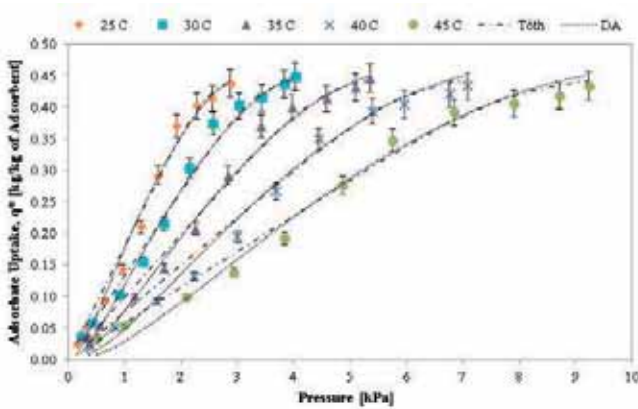
6.2. Silica gel RD powder-water

The silica gel type RD powder-water adsorption isotherms is described in this section. Particularly in Figure 3(a), the data are fitted by classic Tóth and DA isotherm models. The former yields R^2 value 0.989 and NRMSE 6.6 % in contrast with the latter's 0.988 and 6.85 % respectively. Figure 3(b) illustrates the data regression by the proposed model of this work expressed in Equation 8. The model very well captures the linear characteristics of the adsorption isotherm at low pressure Henry's region, and near saturation at relatively high pressure. The R^2 and NRMSE of the new model are 0.997 and 3.15 %, which improves the accuracy as compared to the classic models. Table 2 summarizes the regression parameters of respective models.

Table 2. Value of regression parameters of Tóth, DA, and proposed isotherm models for silica gel RD powder, Zeolite FAM Z01-water pairs.

Silica gel type RD powder-water						Zeolite FAM Z01-water	
Tóth Model		**DA Model**		**Proposed Model**		**Proposed Model**	
q_0 (kg/kg)	0.455	q_0 (kg/kg)	0.455	q_0 (kg/kg)	0.455	q_0 (kg/kg)	0.226
Q_{st} (kJ/kg)	2519.6	Ec (kJ/kg)	164.9	Ec (kJ/kg)	401.9	Ec (kJ/kg)	632.5
K_T (kPa^{-1})	4.5 x 10^{-12}	γ (-)	1.443	ϕ (-)	2.54 x 10^{-4}	ϕ (-)	6.59 x 10^{-8}
t (-)	10.3			C (-)	1.029	C (-)	0.26
				t (-)	0.974	t (-)	0.996
*R^2	0.989	R^2	0.988	**R^2**	**0.997**	**R^2**	**0.993**
**Error	6.6 %	Error	6.85 %	**Error**	**3.15 %**	**Error**	**6.05 %**

*Coefficient of determination
** Normalized root mean square error to the mean experimental data
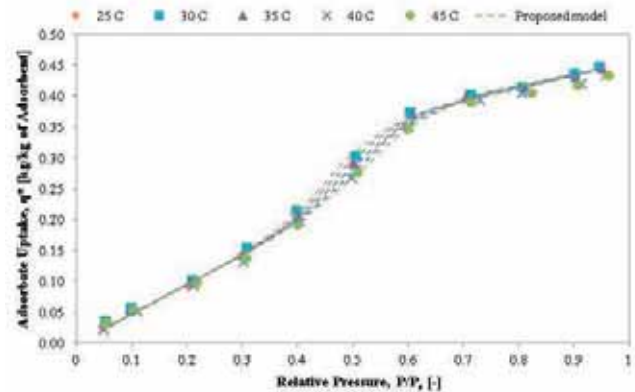

(a) Regression by DA and Tóth models
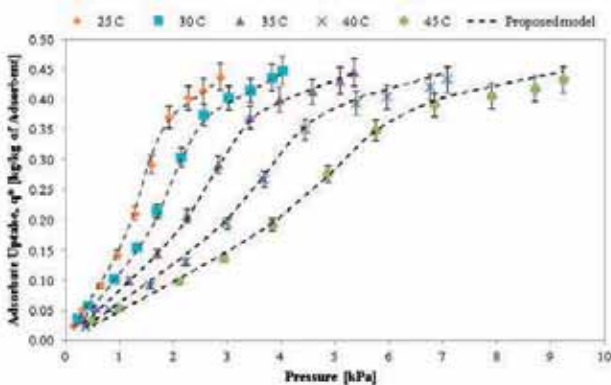
(b) Regression by the proposed model

Fig. 3. Experimental water adsorption equilibrium uptake on silica gel type RD powder at temperatures from 25~45 °C. The data are regressed with (a) the Tóth model and DA model, and (b) the proposed model of this work. The present data are displayed with 5% error bar.

The water equilibrium uptake on the silica gel type RD powder with respect to relative pressure at the fire temperatures, and regression by the proposed model are plotted in Figure 4. It is observed that the isotherms of various temperatures fall onto the same trend line, which implies that the isotherm of other temperatures can be interpolated or extrapolated from the trend line. This is especially useful in the design and performance prediction of adsorption based instrument when precise isotherm data are not available. Moreover, the equilibrium uptake at saturation vapor pressure, q_0, which are formulated in the various isotherm models, can be determined from the graph. For silica gel type RD-water adsorption in the experimental conditions, q_0 equals to 0.455 kg/kg of adsorbent.

Fig. 4. Experimental water adsorption equilibrium uptake on silica gel type RD powder against relative pressure at temperatures from 25~45 °C.

6.3. Zeolite FAM Z01-water

Figure 5 shows the zeolite FAM Z01-water adsorption isotherms with respect the absolute pressure at various temperatures. It is clearly observed that the water uptake increases sharply in a very narrow pressure range for all adsorption temperatures. Outside this pressure range, the uptake increments much less with respect to the elevating pressure. Moreover, there is no tendency of

uptake saturation when the pressure is approaching the saturation pressure of the water. The water uptake data plotted against the relative pressures are shown in Figure 6. The figure shows that the kick-off pressure range at which the sharp uptake increase takes place shifts towards right hand side as the adsorption temperature rises. The range falls typically between the relative pressures 0.15 to 0.25. This is especially beneficial to the adsorption machines which operate in the similar relative pressure range. The dramatic increase of the adsorbent uptake capacity permits large mass transport.

From Figure 6, the limiting water uptake on the zeolite at the saturation pressure of respective adsorption temperatures is approximately 0.226 kg/kg of adsorbent. Its S-shape Type V characteristics are accurately fitted by the proposed model given in Equation 8. The model not only predicts well the kick-off pressure range, the upward trend of the isotherm at lower and higher end of the pressure is also well captured. The equation provides high regression accuracy with R^2 of 0.993, and NRMSE of 6.05% at the experimental conditions. A summary of the regression parameters is provided in Table 2.
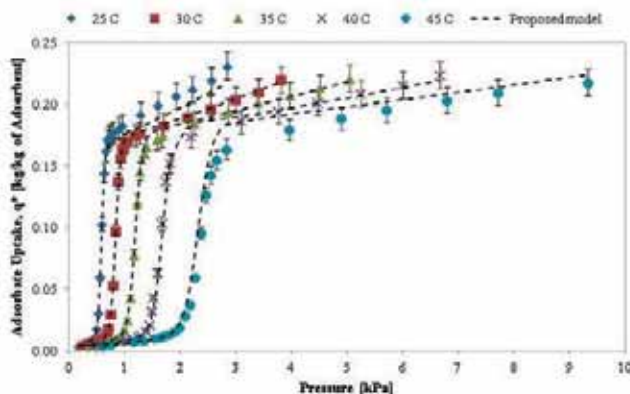

Fig. 5. Experimental water adsorption equilibrium uptake on zeolite FAM Z01 at temperatures from 25~45 °C with regresstion by the proposed model of this work. The present data are displayed with 5% error bar.
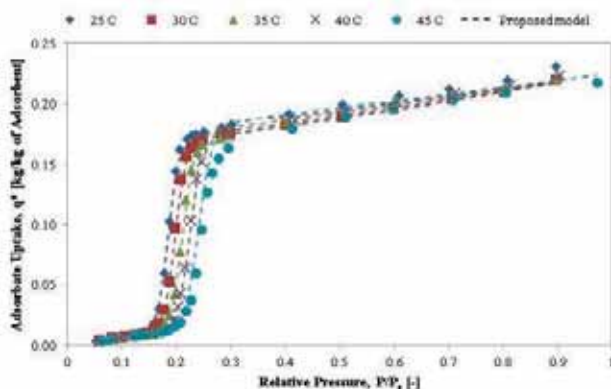

Fig. 6. Experimental water adsorption equilibrium uptake on zeolite FAM Z01 against relative pressure at temperatures from 25~45 °C.

6.4. Comparison on the adsorbents

To understand the performance of the adsorbents for adsorption cooling and desalination cycle, a comparison on the water adsorption capacity is provided in Figure 7. The isotherm of each adsorbent at 35 °C is plotted. As shown, one can observe that the zeolite is more advantageous than silica gel to cooperate with water as working pair for adsorption chiller. First of all, this is due to the uptake difference at typical chiller operation range. For instance, for an adsorption chiller that is operated in a typical condition of evaporator temperature 12 °C, and adsorption bed temperature 35 °C, the relative pressure of adsorption equals to the water saturation pressure at 12 °C divided by that at 35 °C, that is, 0.25. Similarly, if the condenser temperature and the desorption bed temperature are 35 °C and 75 °C respectively, the relative pressure of desorption is 0.15. In this relative pressure range, the zeolite FAM Z01 is able to migrate amount of water 3.4 times that of the silica gels. This means for the same design capacity, the zeolite FAM Z01 equipped chiller has much smaller size than the conventional silica gel-water chiller. Moreover, if the same amount of the adsorbent is used, the zeolite FAM Z01 can produce much more cooling capacity. The advantage well addresses the drawback of the large footprint area faced by the conventional silica gel-water adsorption chiller. Secondly, the cooling capacity produced by the zeolite FAM Z01 chiller is close to a constant quantity for various operation conditions within the designed range, given that the uptake difference experiences little changes when the relative pressures shift. However, due to the relatively large change in the water uptake difference in the designed operation range, the cooling capacity of the conventional silica gel-water adsorption chiller deviates significantly at different operation conditions. As such, it is easier to design the accessories and conjugate cogeneration plants for the zeolite FAM Z01-water adsorption chiller.
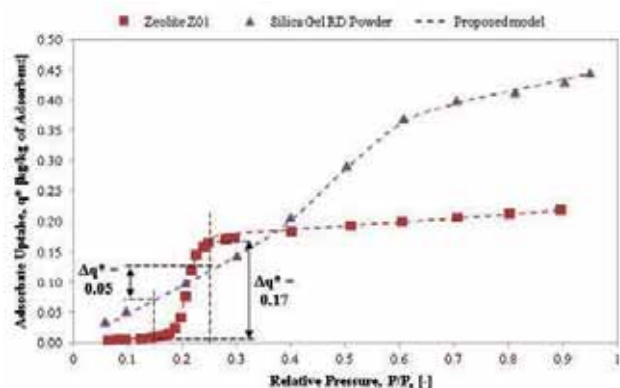

Fig. 7. Comparison of water uptake capacity against relative pressure for a typical adsorption temperature 35 °C on zeolite FAM Z01 and silica gel RD powder.

7. Conclusion

In this work, a novel adsorption isotherm model is proposed for both Type I and V categories with four regression parameters. The data fitting with the proposed model for Type I silica gel RD powder-water pair has R^2 0.997 and NRMSE 3.15 %, which is considerably more accurate than classic DA and Tóth equations. The new model also well avoids the problem of null uptake at low relative pressure region and saturated uptake at high relative pressure region for Type V zeolite FAM Z01-water pair. The regression accuracy for the pair is 0.993 R^2 value and 6.05% error, respectively. In addition, the zeolite FAM Z01 is demonstrated a 3.4 times higher water adsorption uptake than silica gel type RD at typical adsorption chiller operation range, which will significantly benefit the existing adsorption chiller design.

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