Experimental investigation of the silica gel–water adsorption isotherm characteristics

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Abstract

In designing adsorption chillers that employs silica gel–water as adsorbent-adsorbate pair, the overriding objective is to exploit low temperature waste-heat sources from industry. This paper describes an experimental approach for the determination of thermodynamic characteristics of silica gel–water working pair that is essential for the sizing of adsorption chillers. The experiments incorporated the moisture balance technique, a control-volume-variable-pressure (CVVP) apparatus and three types of silica gel have been investigated, namely the Fuji Davison Type A, Type 3A and Type RD. As evidenced by the experimental results, the Henry-type equation is found to be suitable for describing the isotherm characteristics of silica gel–water working pair at the conditions of adsorption chiller. The regeneration of adsorbent depends on the correct allocation of temperature as well as the amount of regeneration time. From the experiments, the isotherm characteristics of silica gel–water in the low- to high-pressure regimes and hence, its isosteric heat of adsorption will be determined. Key parameters for optimizing the amount of heat recovery such as the cycle and switching time of chiller can also be implied from the measured results. © 2001 Elsevier Science Ltd. All rights reserved.

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Nomenclature

K	adsorption equilibrium constant	q^*	amount adsorbate adsorbed by the adsorbent at saturation condition (kg adsorbate/kg adsorbent)
k_a	adsorption rate constant		
k_d	desorption rate constant		
L_v	latent heat of vaporization (J/kg)	T	temperature
m_{sg}	total mass of silica gel packed into the system (kg)	T_{amb}	ambient temperature
		T_{cond}	condenser temperature
P	partial pressure in the gas phase (Pa)	T_{evap}	evaporator temperature
p_s	saturated vapour pressure (Pa)	T_{reg}	regeneration temperature
Q_{diff}	differential heat of adsorption	θ	fractional filing of the micropore
Q_{st}	isoteric heat of adsorption	Δq	differential of the amount adsorbed
Q_{total}	total heat adsorbed by the evaporator during the adsorption step		

1. Introduction

In recent years, the increase in awareness on non-CFCs refrigerant-based chillers has prompted relentless studies on sorption cooling systems. These systems have the advantages of, firstly, exploiting low-temperature heat sources of between 50°C and 90°C, and secondly, low electricity usage for circulation of coolant within the chiller [1–9]. Moreover, environmental-friendly water is one of the refrigerants used in sorption cycle and it has a large latent heat of vaporization. Some of the basic information required for design of sorption chiller is the isotherm characteristics of silica gel–water, in particular their behaviour under partial vacuum conditions during physical (as opposed to chemical) interactions. The adsorbate (refrigerant) uptake at the exchangers such as the adsorber/desorber of commercial sorption chillers will be investigated. Thus, the motivation for the present study is to unravel the sorption potentials of two commonly used adsorbents, namely, the Fuji Davison Type 3A and Type RD silica gel.

Silica gel is a incompletely dehydrated polymeric structure of colloidal silicic acid with the formula $SiO_2 \cdot nH_2O$. This amorphous material consists of spherical particles 2–20 nm in size, sticking together to form the adsorbent silica gel [10]. Silica gel exhibits an excellent capacity for adsorption of water up to 35–40% of its dry mass at conditions found in chillers and hence, chosen as the adsorbent for our experiment. It has regeneration capability at low heat source temperatures from 50°C to 90°C and making it suitable for use in sorption chillers. The surface of silica gel comprises mainly SiOH and SiOSi groups and being polar bonded, it absorbs similar adsorbates such as water and alcohol. Water is physically held onto the surface by dispersion forces and polar forces like in the case of hydrogen bonded water. For this paper, the thermodynamic characteristics of three types of silica gel namely Type 3A, Type RD and Type A will be studied and their thermophysical properties (data provided by Fuji Silysia Chemical Ltd., Japan) are presented in Table 1.

Table 1
Thermophysical properties of silica gel

	Type A	Type 3A	Type RD
Specific surface area (m^2/g)	650	606	650
Porous volume (ml/g)	0.36	0.45	0.35
Average pore diameter (A)	22	30	21
Apparent density (kg/m^3)	730	770	800
pH value	5.0	3.9	4.0
Water content (wt.%)	<2.0	0.87	–
Specific heat capacity (kJ/kg K)	0.921	0.921	0.921
Thermal conductivity (W/m K)	0.174	0.174	0.198
Mesh size	10–40	60–200	10–20

We can see from Table 1 that Type A and Type RD silica gel have many physical similarities, which give rise to their similar desorption characteristics at different regeneration temperatures. The surface characteristics and the pore structure are thus the determining factors affecting adsorption equilibrium.

The steady state or equilibrium behaviour of silica gel–water will be modeled using the Henry-type equation and their validity is fortified by measured data from the control-volume-variable-pressure (CVVP) apparatus. For this study, the silica gel within the CVVP apparatus is always in contact with mainly pure water vapour at isotherms ranging from 303 to 338 K, whilst the equilibrium sorption pressures are ranged from 500 to 6500 Pa. These are the conditions that would be found in real adsorption chillers during a normal operating cycle. To avoid unnecessary condensation of refrigerant, water vapour is introduced into the CVVP apparatus at a temperature slightly above the room temperature.

2. Experimental facility

Figs. 1(a) and (b) show a pictorial view and a schematic layout of the experimental CVVP apparatus used in this study, respectively. It consists of the vapour and water piping systems and an oil-bath heater. The main components are the charging and dosing tanks that are made from the corrosion-resistant stainless steel-304. The pressure in tanks is measured with two heat resistant Edwards Barocel-622 capacitance manometers. Their operational temperature ranges from 318 to 396 K. The temperatures in tanks are measured with two class-A Pt 100 Ω RTDs ± 0.15 K. The RTD for the charging tank, as indicated by T_c in Fig. 1(b), is designed to be in direct contact with the silica gel to give accurate temperatures of absorbent. A two-stage Edwards ''Bubbler'' rotary vane vacuum pump is used for evacuation of air and water vapour. Isothermal conditions in the dosing and charging tanks are effected by immersion of the apparatus in a water bath where the temperature of the latter is controlled by two external temperature baths: uniformity of bath temperature is enhanced by a cross-flow of water. To prevent any condensation, a thermostat-controlled tape heater covers the external surfaces of two pressure barocels whilst the dosing tank temperature is held above the dew point of charged vapour.
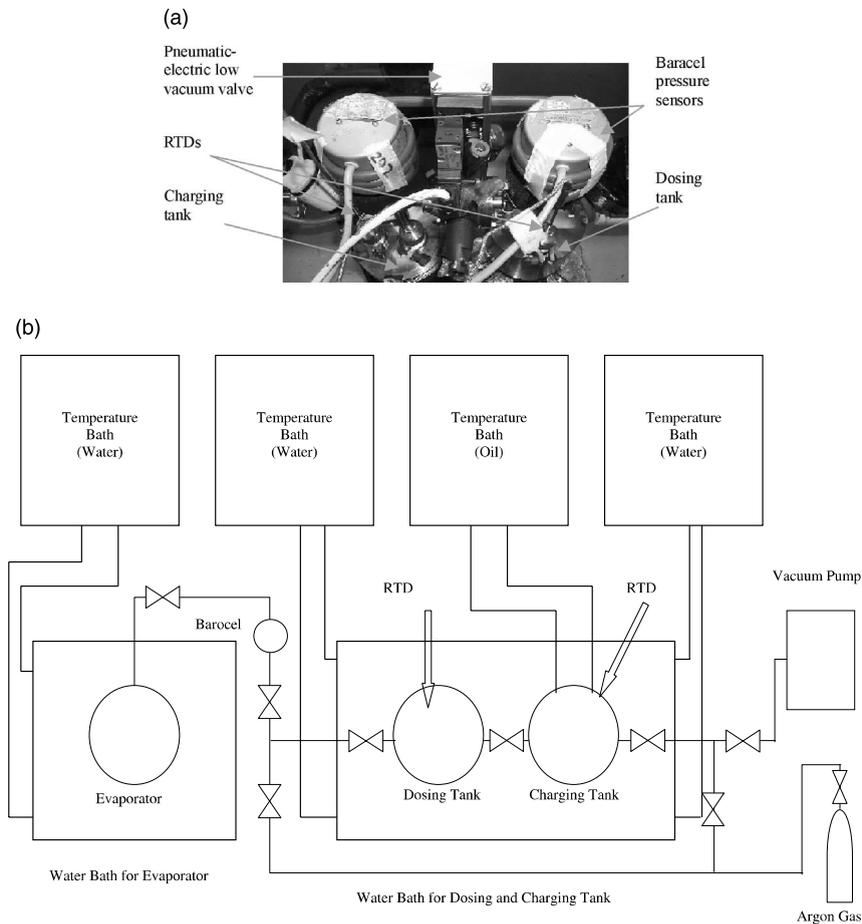
(a)

(b)

Fig. 1. (a) The experimental CVVP apparatus (top view). During experiments, the tanks are filled with circulating hot water till the brim of the Barocel. (b) Schematic diagram of experimental set-up showing the water baths, sensors and vacuum pump.

For the regeneration of adsorbent (silica gel), a hot oil circuit has been used, as also indicated in Fig. 1(b). The charge is fed from an evaporator that comprises a 2000 cm^3 round-bottom glass flask filled with distilled water. Isothermal condition of evaporator is maintained by another thermal bath. A calibrated data logger is used to monitor all pressure and temperature readings. The networked data acquisition units use an Ethernet connection that supports 100 measurements per second, with 5.5 digits of resolution of 0.02% accuracy.

2.1. Experimental procedures

Firstly, the test system is collectively made up of the dosing, charging tanks all other related piping components. Argon gas is used to purge the vacuum system of atmospheric gases. A

column of packed calcium sulphate is used to dehydrate the argon gas before it is used to purge the system. The purged system is evacuated and then isolated. The silica gel within the charging tank is regenerated 413 K for 24 h. At the end of the regeneration process, the test system is evacuated again and purged using dehydrated argon gas. The silica gel further regenerated for another 8 h. A residual argon pressure of approximately 1 mbar is obtained in the vacuum system after evacuation by the vacuum pump. Though the effect of the partial pressure of argon gas on the results is small, the calculations consider the presence of argon in order to avoid systematic error.

Before introducing water vapour from the evaporator into the dosing tank, the vapour pipe from the evaporator towards the dosing tank is evacuated to remove any traces condensed water vapour, which cause experimental errors. The dosing tank temperature is kept at 5°C over the evaporator temperature to eliminate condensation of water vapour within. Condensation will lead to errors in the pressure reading. The effect is that the initial calculated mass of water vapour charged will be lower than the actual mass charged. To eliminate error due to condensation, the evaporator is closed off from the dosing tank quickly once enough water vapour has been charged. The charging stops once equilibrium is achieved between the pressures of the evaporator and the dosing tank. The mass of initial water vapour charged into the system is calculated by using the ideal gas law, taking the effect of residual argon pressure into account.

Once the test system achieves thermodynamic equilibrium at the desired temperature, the interconnecting pneumatic-electric valve between the two tanks is opened and the pressures approach equilibrium. The water temperature in the water bath is decreased to 303.15 K, to facilitate the recording of the first temperature and pressure reading. The temperature of the charging tank (silica gel tank) is slightly higher than that of the dosing tank due to the release of heat of adsorption when the adsorbate is adsorbed by the adsorbent. On reaching equilibrium and the temperature of the charging tank stabilizes, thermodynamic equilibrium is achieved and the readings are taken.

The mass of water vapour absorbed by the silica gel is computed as follows:

$$M_{ads} = M_{di}(\Delta P_i, T_i) - M_{cd}(\Delta P_f, T_f) = \frac{1}{R_w} \left[\left(\frac{(P_{di} - P_{rdi})V_d}{T_i} \right) - \left(\frac{(P_{cd} - P_{rf})V_{cd}}{T_f} \right) \right] \tag{1}$$

where M_{ads} is the mass of water vapour absorbed by silica gel, M_{di} is the initial mass of water vapour in the dosing tank, M_{cd} is the final mass of water vapour left in charging and dosing tank, P_{di} is the initial pressure of the dosing tank, P_{rdi} is the initial residual pressure of argon gas, R_w is the characteristic gas constant of water, P_{cd} is the final pressure of charging and dosing tank, P_{rf} is the final residual pressure of argon, V_{cd} is the volume of dosing tank, R_w is the characteristic gas constant of water, T_f is the final temperature of dosing and charging tank.

The temperatures of both the charging and dosing tank are then adjusted to desired isotherms' temperature. In this experiment, the isotherms to be determined are at 303.15, 308.15, 313.15, 323.15 and 338.15 K. The test system is then prepared for later runs. After the test system is evacuated and the silica gel regenerated, the system is purged with dry argon gas and the process of charging water vapour at a different initial pressure into the dosing tank begins for further measurement along the isotherms.

2.2. Moisture balance

A moisture balance technique is used to measure the difference in weight of the silica gel between its saturated state and the final desorbed state with respect to temperature and time. An analysis of the desorption characteristics of the Type A, Type 3A and Type RD silica gel with respect to temperature and time has been done. The study of the desorption characteristics of the Type RD, Type 3A and Type A silica gel over a range of temperatures was done using a Sartorius moisture balance. The MA40 device which uses the thermogravimetry technique and moisture analysis is done based on infrared drying of the silica gel. Data such as temperature, time and weight of silica gel are simultaneously captured and recorded using the MA40 Wedge software. The principal adsorbate from desorption is water. The masses of the three types of silica gel in a saturated condition are obtained at atmospheric pressure and at a room temperature of 24°C. Using the control software for the MA40 moisture balance, the heating temperature is raised from room temperature to the various regeneration temperatures between 40°C and 140°C in 10°C intervals.

2.3. Adsorption model

Adsorption depends on the nature of the solid surface and of the solvent medium. Solids with a highly developed surface (silica gel in the present case) generally adsorb gases and vapours. The heat evolved is known as the heat of adsorption. The heat of adsorption is usually 30–100% higher than heat of vaporisation of the adsorbate. Thus, if an adsorbent and adsorbate in liquid form were put separately in a closed container, the adsorbate would be transported from the liquid phase to the adsorbent in vapour form, since adsorption is stronger than condensation to the liquid phase. As such, the temperature of the liquid phase decreases while the adsorbent temperature is increased. Air conditioning and refrigeration work based on this phenomenon.

One of the simplest cases when discussing adsorption equilibrium is that involving one adsorbate like in the case of single-component adsorption. The amount of adsorbate adsorbed by an adsorbent when equilibrium is reached under a given temperature and pressure (gas-phase adsorption) is a function of the nature of the adsorbate and adsorbent.

For a given adsorbate–adsorbent system, the adsorption equilibrium data can be expressed as

$$q^* = q^*(P, T) \tag{2}$$

where q^* the amount adsorbed per unit mass adsorbent, T is temperature and P is the partial pressure of the adsorbate in the gas phase. At equilibrium after a sufficiently long period of time, the adsorption rate ($k_a P(1 - \theta)$) and desorption rate ($k_d \theta$) are assumed equal thus the commonly quoted form of the Langmuir [11] equation is as follows:

$$\theta = KP/(1 + KP) \text{ or}$$
$$KP = \theta/(1 - \theta) \tag{3}$$

where θ ($= q^*/q_0^*$) is the surface coverage or fractional filing of the micropore, k_a is the adsorption constant, k_d is the desorption constant, q^* is the adsorbed phase concentration at equilibrium, q_0^* is

the adsorption capacity of the adsorbent, P is the partial pressure in the gas phase and $K \ (= k_a/k_d)$ is the adsorption equilibrium constant.

At low adsorbate concentrations ($q^* \ll q_0^*$), Henry's law is approached and Eq. (3) can be reduced to the Henry type equation.

$$\theta \Rightarrow KP \tag{4}$$

The isosteric heat of adsorption, Q_{st}, is related to adsorption isotherms at different temperatures by a vant Hoff equation [12,13].

$$Q_{st}/RT^2 = (d\ln P/dT) \tag{5}$$

As there is a linear relationship between q^* and P and by differentiation of Eq. (4), Q_{st} is related to the adsorption equilibrium constant, K (temperature dependent), and rearranged as

$$-Q_{st}/RT^2 = (d\ln K/dT) \tag{6}$$

and we can integrate Eq. (6), which gives

$$K = K_0 e^{Q_{st}/RT} \tag{7}$$

where K_0 is a constant. Substituting Eq. (7) into Eq. (4), we obtain

$$\theta = q^*/q_0^* = K_0 \exp^{(Q_{st}/RT)} P \tag{8}$$

This is the working equation for the adsorption experiments. Should one were to recast the isotherms data in terms of $\ln(q^*/P)$ versus $1/T$, the gradient and intercept of Eq. (8) allows the determination of Q_{st} and $K_0^I = K_0 q_0^*$, respectively.

3. Results and discussion

The weight of silica gel versus heating temperatures is obtained from the moisture balance experiments and typical plots for the three types of silica gel, namely the Type "3", "3A" and "RD", as shown in Fig. 2a. It is observed that a heat source temperature of 90°C is sufficient to achieve 95% regeneration for all three types of gel investigated. In terms of the rates of desorption of water vapour, their behaviour is found to be both time and temperature dependent. For a regeneration temperature of 140°C, Type "3A" would take 2.5 min to desorb 95% of its original mass of absorbate; Type "RD" would take 4 min whilst Type "A" is about 5 min, as observed in Fig. 2b. Type "3A" silica gel has the fastest response time, due probably by the larger porous volume.

The isotherm characteristics of water vapour, at temperatures and pressures ranging from 303 to 338 K and from 500 to 6500 Pa, respectively, have been obtained using the CVVP apparatus (as shown in Fig. 1). The experiments focus on the conditions that are found in a typical adsorption chiller where the beds are subjected to periodic switching from a cooling to heating mode.

Figs. 3a and b show the experimental data obtained for the Type 3A and Type RD silica gel–water system respectively. Our experiments are also compared with the equilibrium data of the 1992 batch of the Type RD silica gel–water system, as employed by the adsorption chiller manufacturer [14]. It is noted that the data from the manufacturer were obtained for temperatures between 328 and 358 K and for pressures between 1000 and 3500 Pa but the measurement uncertainty was not reported. It is noted that the isotherms of the current batch of the Type RD and
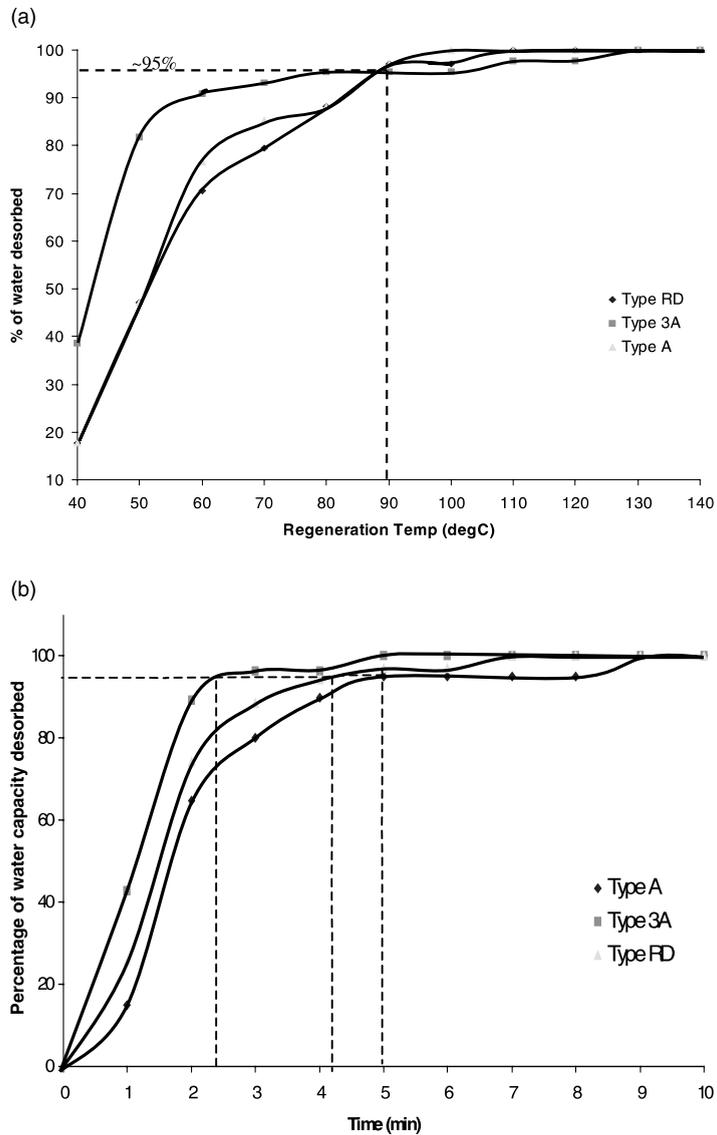
Fig. 2. (a) Graph of the percentage desorption achieved at various regeneration temperatures. (b) The graph of the percentage desorption achieved with respect to time.

Type 3A silica gel–water system is also consistent with the 1992 batch. Analysis of the results show that water vapour uptake of the current batches of Type 3A and Type RD silica gels is proportional to the vapour pressure to within experimental error of 7% and thus their performances can described by Henry's law.

When plotted in terms of the $\ln(q^*/P)$ versus $1/T$, the performance of the Type 3A and Type RD silica gel–water systems, including that for the 1992 NACC batch of the type RD silica gel–water system, is shown in Fig. 4. As observed, it is a linear plot with good agreement with the manufacturer's data over a small overlapping temperature range. The results reinforce the present
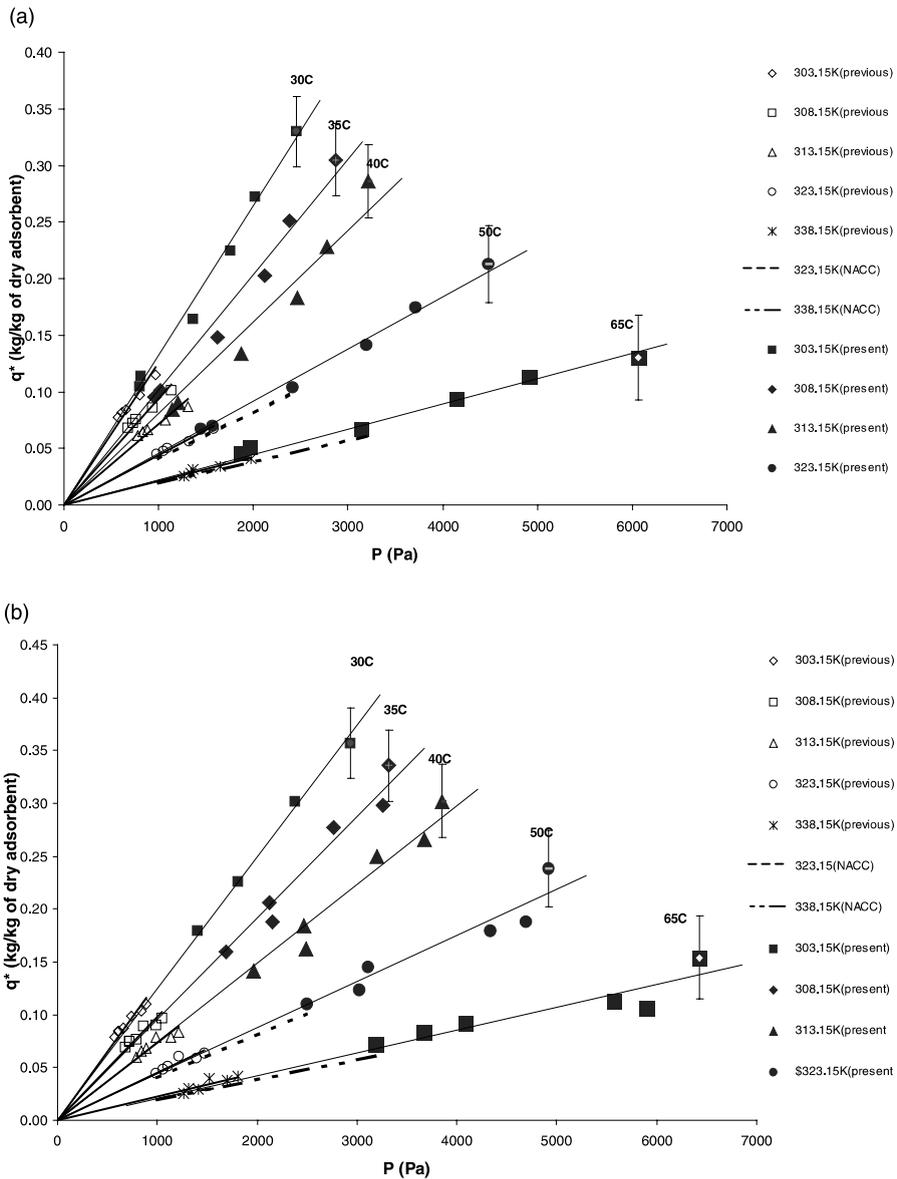
Fig. 3. (a) Isotherm data for the Type 3A silica gel–water system obtained from the present experiment. NACC Type RD data (NACC, 1992) plus previous work [9] is included for comparison. (b) Isotherm data for the Type RD silica gel–water system obtained from the present experiment. NACC Type RD data (NACC, 1992) plus previous Masters work is included comparison.

belief that Henry's law can characterize the silica gel–water system and a detailed Henry's law's correlation for the two types of silica gel–water binary system is summarized in Table 2. The two empirical coefficients used to correlate the data are presented.

To demonstrate the practical cycles of adsorption chillers using the silica gel–water system, Fig. 5a and b presents the experimental data at two useful vapour isobars ranges, namely, the vapour
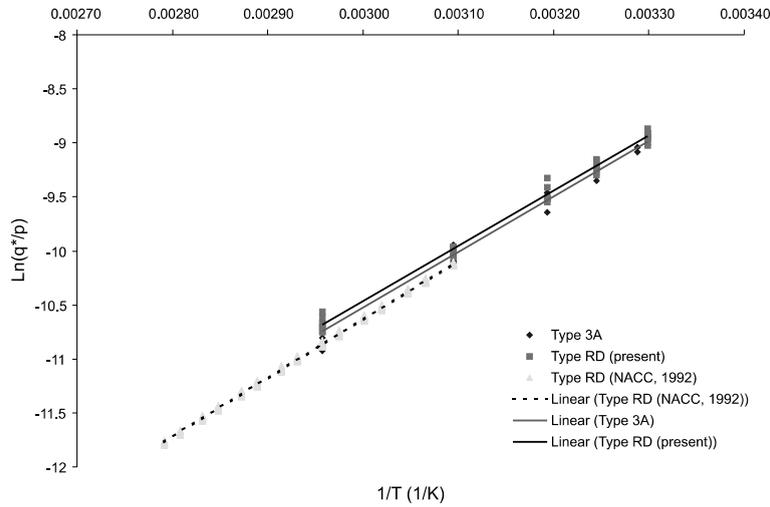
Fig. 4. Henry's law plots of the current batch of Type 3A and Type RD data. NACC Type RD (1992) data is also included for comparison.

Table 2
Correlation coefficients for the Type 3A and Type RD Fuji Davison silica gel–water systems

$q^* = K_0^1 \exp[Q_{st}/(RT)]P$		
Silica gel type	K_0^1 (Pa^{-1})	Q_{st} (kJ/kg)
Type 3A	5.2×10^{-12}	2.38×10^3
Type RD (1992 batch)	2×10^{-12}	2.51×10^3
Type RD (current batch)	5.5×10^{-12}	2.37×10^3

pressure of the evaporator (temperature = 285 K; pressure = 1401 Pa) and the condenser (temperature = 303 K, pressure = 4498 Pa), respectively. For simplicity, the regeneration temperature is assumed to be 363 K (point B) and the ambient temperature is taken as 303 K (point A). The vertical ordinates at these mentioned temperatures affect the effective cooling achievable by the silica gel–water system from each ideal cycle. As discussed in the earlier section, a regeneration temperature of 363 K would achieve 95% regeneration of the silica gel at equilibrium condition. The total heat absorbed by the evaporator during the adsorption cycle, Q_{total}, would be computed by taking the product $\Delta q L_v m_{sg}$, where $\Delta q = q_A - q_B$ (as can be seen in Fig. 5a and b).

Fig. 5c shows the differential amount of absorbate absorbed, Δq, by the silica gel for two types of silica gel, namely the Type 3A and Type RD. We see that Type 3A has a slightly lower cooling capacity compared with Type RD due perhaps to lower specific surface area and lower apparent density. The available regeneration temperature is one factor in the determination of the choice of silica gel used in the chiller system.

4. Conclusions

Using the moisture balance method, the regeneration and isotherm characteristics of Fuji Davison Type A, Type 3A and Type RD silica gel have been studied for temperatures up to
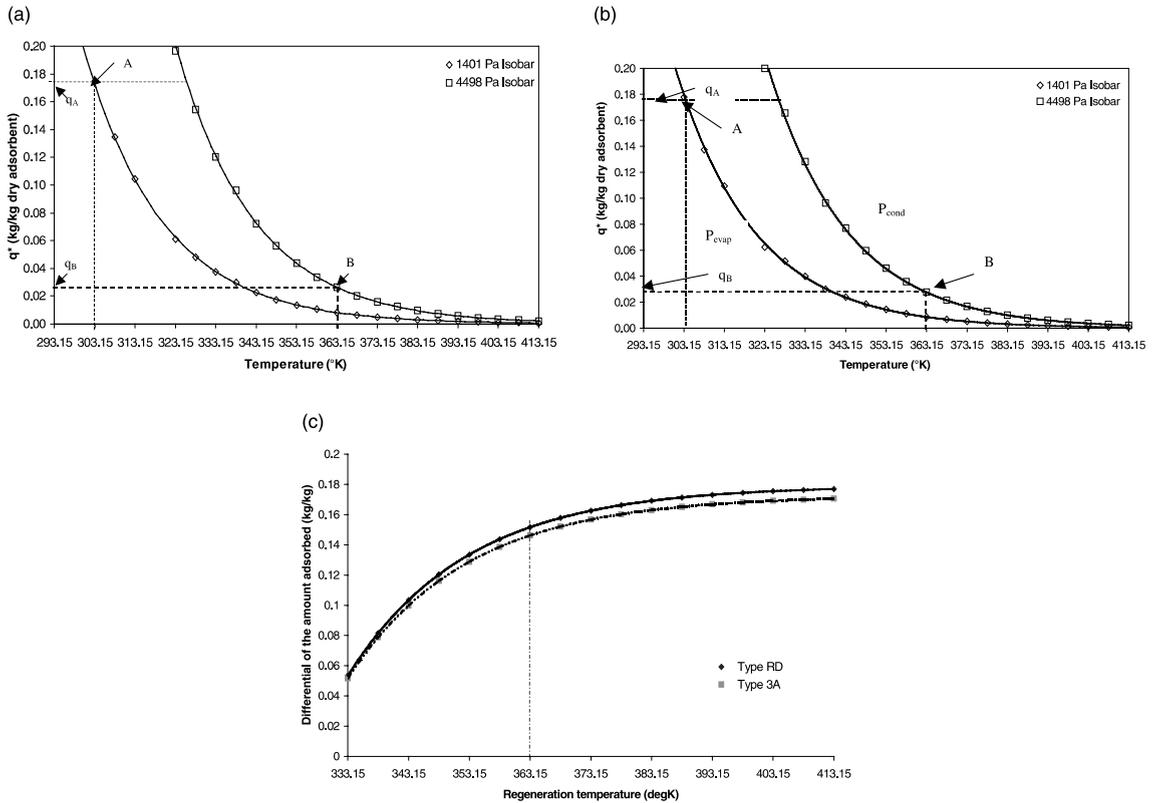
(a)

(b)

(c)

Fig. 5. (a) Practical cycles of adsorption and regeneration on the assumption of $T_{reg} = 363.15$ K, $T_{amb} = 303.15$ K using Type 3A silica gel. (b) Practical cycles of adsorption and regeneration on the assumption of $T_{reg} = 363.15$ K, $T_{amb} = 303.15$ K using Type RD silica gel. (c) Plot of differential of the amount adsorbed versus the regeneration temperature.

413.15 K. The Type 3A and Type RD silica gel batches can achieve around 95% regeneration within 250 s. The tests were conducted under ideal conditions with mono-layer adsorption and isothermal chamber. Thus, the measured regeneration time is expected to be shorter as compared with the cycle time of real adsorption chillers where the cycle time is typically 450 s. The rate of heat released was not measured during the regeneration process due to the limitation of the present experimental apparatus. Our objective was to determine only the isosteric heat of adsorption at equilibrium condition, which could be deduced from the gradient of Fig. 4. The value of Q_{st} would be employed as a key parameter in the quantification of heat released by absorbent caused by vapour uptake.

The Type 3A and Type RD silica gel are thus better choices as adsorbents in cases where cycle time is an important factor during chiller design. Between the temperatures of 70°C and 90°C, at least 75% regeneration of silica gel can be achieved. In the event only low temperature waste heat sources are available, Type 3A silica gel should be used as the adsorbent.

Through the use of the control-volume-variable-pressure apparatus, the isotherms at temperatures between 303 and 338 K and pressures ranging from 500 to 6500 Pa have been obtained. The

empirically determined parameters for the isotherm equations have also been calculated successfully. The isotherm plots reveal that this experiment's silica gel–water system can be satisfactorily described by Henry's law. This fact is further reinforced by Henry's plot, which allows the coefficients for the isotherm equation to be calculated. The isosteric heat of adsorption for the Type RD system is calculated to be slightly higher than the Type 3A system. Adsorption chiller manufacturer's data of a 1992 batch of the Type RD silica gel–water system (NACC, 1992) has also been included for comparison. It is noted that Henry's law can also describe the behaviour of this 1992 batch of the Type RD silica gel–water binary system. The performance of the 1992 batch of the Type RD silica gel–water system is found to be similar to that of the current batch of the Type 3A and Type RD silica gel–water system. The experiments also reveal that the current batch of the Type RD system has a slightly higher uptake at equilibrium condition than that of the chiller manufacturer's.

The isobar analysis is also performed to determine the adsorption capacity of the Type 3A and Type RD silica gel–water system. This allows the determination of cooling capacity of a silica gel–water system from the differential of the amount adsorbed. For temperatures between 333 and 343 K, Type A silica gel has better potential in terms of cooling capacity. For temperatures above 353 K, Type 3A and Type RD silica gels are the better choices as adsorbents.

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