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Adsorption and Desorption Isotherms of Desiccants for Dehumidification Applications: Silica Aerogels and Silica Aerogel Coatings on Metal Foams

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ABSTRACT

Silica aerogels are frequently employed as solid desiccants in enthalpy wheels for dehumidifying the supply stream in air-conditioning systems. These desiccant materials possess good moisture adsorption and desorption characteristics due to their porous structure. Analysis of adsorption and desorption isotherms is critical for performance characterization and is often performed to evaluate the capacity and transient performance of desiccant-based dehumidification systems. The current study is focused on the adsorption and desorption isotherms of solid silica aerogels and silica aerogel coatings on open-cell metal-foam substrates. The sol-gel process is adopted to synthesize silica aerogels using different basic (ammonium hydroxide, potassium hydroxide) and acidic (hydrofluoric acid, steric acid, hydrogen peroxide) catalysts, with the same precipitator (tetra methyl orthosilicate-TMOS) and solvent (methanol). Scanning electron microscopy is used to characterize the microstructure of supercritically dried aerogels and adsorption/desorption isotherms for the different samples are obtained by the dynamic vapor sorption method. The steady-state moisture adsorption and desorption capacity of silica aerogels is affected by their porous structure, which depends on the synthesis technique used to prepare the silica aerogels. For the silica aerogel coatings on metal foams, the substrate structure and surface area also play an important role. The effect of the substrate surface area on adsorption/desorption capacity is analyzed by comparing the isotherms for solid silica aerogel samples, and silica aerogels coatings on flat plates and on metal foams with different pore sizes.

1. INTRODUCTION

Because of the importance of energy efficiency in the built environment, there is significant interest in separating the sensible and latent loads when conditioning air for human comfort. Separating the sensible and latent loads offers significant potential in energy savings and provides opportunities for improved control of temperature and humidity. The psychrometric process of a SSLC system is presented in Figure 1. This system consists of one vapor compression system and one solid desiccant wheel (enthalpy wheel). The vapor compression system provides only sensible cooling (point A to point B) required by the conditioned space at both elevated air temperature leaving the

evaporator and a higher air mass flow rate. The reason for a higher air mass flow rate requirement is to compensate for the reduced enthalpy difference of air across the evaporator, and to maintain the capacity of sensible cooling. Since the vapor compression system operates above the dew point temperature of supply air and is not required to provide the latent cooling, the desiccant wheel is used to reduce the water vapor content in the part of the air leaving from the sensible evaporator. The part of the dry air from the desiccant wheel mixes with the rest of the air from the evaporator and is delivered to the conditioned space (point D).

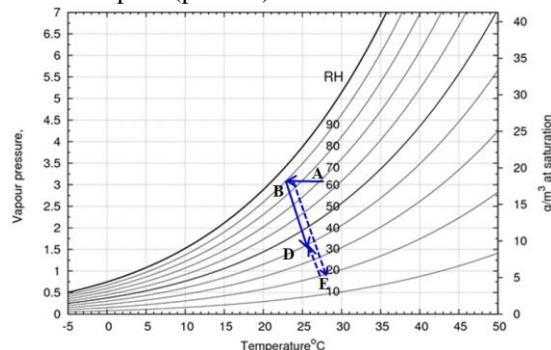


Figure 1: Operation of SSLC system (with enthalpy wheel)

Many recent studies of SSLC system have focused on the development of solid adsorbent deployed for dehumidification that can provide improved sorption capacity and higher mass and heat transfer rates, as well as favorable equilibrium isotherms (Aristov *et al.*, 2002, Dawoud *et al.*, 2003, Zhang *et al.*, 2005). Adsorption systems with improved performance result in substantial decreases in the initial and operating costs and in some cases make such dehumidification systems attractive alternatives to existing vapor compression systems for cooling and dehumidification. There are numbers of commercially available water sorbents used for specific sorption processes. The development of environmentally friendly technologies for sorption of water at different conditions coupled with the preparation of advanced materials with improved sorption properties, recycling possibilities and long-term use are enduring tasks. In general, the efficiency of hygroscopic materials in adsorbing water depends on two factors, i.e. the amount and the type of sorbents in use. Up to date, several water sorbents have been discovered, and they are classified in three main categories: first: inorganic materials (zeolites, clays and silica), second: carbon based adsorbents (activated carbons, graphite, carbon molecular sieves, and pre-shaped carbon fibres and nanotubes), and third: organic polymers. The most studied solid adsorbents for moisture removal are microporous materials (zeolites), activated carbon, and silica gels. The water sorption behavior of a sorbent depends on many factors such as the structure and the chemical composition of the nanoporous material, the presence of charged species, type of framework structure, and hydration level. In many cases, humidity control by adsorption meets much lower dew points and is less energy demanding compare to compression and condensation methods. Traditionally, highly hygroscopic salts such as LiBr, LiCl, KBr, CaCl₂ and MgCl₂ are used for humidity control via adsorption of water. However, a crystallization process tends to happen when the salts are used at high water concentrations. In addition, the high solubility of these salts in water at high humidity limits their application at certain conditions. The search for alternative adsorbent other than salts in water sorption to avoid the risk of salt crystallization, led to the evaluation of silica aerogels as one possible desiccant material (Besant and Simonson, 2003).

Silica aerogels are highly porous materials with low density, low thermal conductivity, as well as large surface area. They have received significant attention in heat insulation (Omar *et al.*, 2007), waste treatment (Ahmed and Attia, 1995), drug delivery and targeting systems (Smirnova *et al.*, 2004 a) as well as many others. Silica aerogel has relatively high moisture adsorption capacity because of its microporous structure of internal interlocking cavities, which gives a high internal surface area (up to 800 m²/g, or 10⁸ to 10⁹ m²/m³) (Smirnova *et al.*, 2004 b). When the water vapor pressure at or near any pore region of a silica gel particle is lower than the adjacent air water vapor pressure, water molecules diffuse through the air to the surface and adhere to the surfaces, especially the internal surface of the silica gel particles. The higher the humidity of the air, then the greater the mass of the water adsorbed by the silica gel. Another advantage of using silica aerogel is fact that there is no chemical reaction during adsorption, unlike many salt adsorbents which change the chemical composition and physical appearance with addition of moisture. Even when saturated with water vapor, silica gel still has a dry appearance with its geometry unchanged.

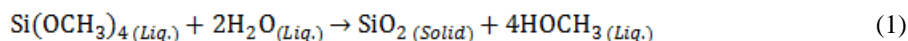
The adsorption and desorption characteristics of different silica gel samples may vary because of different manufacturing procedures (Zhang *et al.*, 2005). In general, the salt adsorbents, such as calcium chloride, have better moisture absorbing capacity than the organic adsorbents, such as silica gel, but deliquescence occurs on the surface of calcium chloride granules beyond a certain adsorption level and a hydrate solution is formed). This behavior limits the effectiveness of salt desiccants (Zhang *et al.*, 2005). In order to overcome this problem, desiccant materials based on silica aerogel have become an attractive alternative to the existing salt-based adsorbents (Pesaran and Mills, 1987). They have been used as a high-performance desiccant to remove water vapor from humid ventilation air for buildings (Besant and Simonson, 2003). There is no chemical reaction involved during adsorption and desorption. Even when saturated with water vapor, silica gel still has a dry appearance with its geometry unchanged, which is an advantage over liquid desiccant systems.

The solid desiccant can be deployed by coating a solid substrate. The characteristics of the substrate, such as surface area and thermal conductivity, affect the moisture removal performance considerably. Therefore, an appropriate dehumidification performance evaluation of the desiccant coated on the substrate is important (Nawaz *et al.*, 2014a,b). One potential candidate for a substrate material is metal foam. There has been considerable interest in establishing the thermal-hydraulic performance of metal foams when used as a heat exchanger. Despite manufacturing and implementation issues, these materials hold promise as both heat exchangers and heat sinks (Nawaz *et al.*, 2010 and 2012, Dai *et al.*, 2012). The open porosity, low relative density, high thermal conductivity, large surface area per unit volume, and the ability to enhance fluid mixing can make metal foam thermal management devices efficient, compact, and light-weight. Two major advantages of using metal foams as substrates are the large surface area per unit volume and a thermal conductivity higher than that of the desiccant. A relatively large quantity of silica aerogel can be deployed as thin coating on the foam, and the higher thermal conductivity of the foam assists in removing the heat of adsorption and can provide heat for desorption (Nawaz *et al.*, 2014 a,b). The adsorption and desorption characteristics of different silica gel coatings may vary because of different manufacturing procedures (Zhang *et al.*, 2005). Although silica gel is frequently used as a desiccant, the transport of heat and moisture within the pores of silica gel particles is complex and research is ongoing. Comprehensive experimental studies of the physicochemical properties and some research applications of the organic and salt-based adsorbents have been reported by Aristov *et al.* (2002) and Zhang *et al.* (2005). These studies show that silica-aerogel-based adsorbents have a higher adsorption capacity and can be regenerated with a lower temperature than the other commercially available desiccants, such as activated carbon.

Despite such promising properties, conclusions as to the feasibility of these materials for dehumidification systems can only be drawn after detailed analysis of the adsorption and desorption performance of the silica aerogel coated on the substrate under realistic operating conditions. The capacity of a porous adsorbent solid in adsorption of an adsorbate gas is determined by the adsorption isotherm, and the mass diffusivity affects the adsorption rate. The steady state adsorption and desorption properties of adsorbents with different microstructures have not been widely reported, and this is especially true for the aerogels coated on a metal foam surfaces. The primary objective of the present work is to investigate the steady state adsorption/desorption capacity of different silica aerogels coated on metal foams by analyzing the equilibrium isotherms.

2. PREPARATION OF SAMPLES

Silica aerogel coated metal foam samples were prepared using a dip coating method. Metal foams with pore size of 4.02 mm (5 PPI), 3.28 mm (10 PPI) and 2.58 mm (20 PPI) were used for this study. Small metal foam blocks with dimensions of about 8 mm were machined for coating. Wet silica gels were prepared using silicon alkoxide precursor, tetramethyl orthosilicate (TMOS, Si (OCH₃)₄). The formation of a wet gel by TMOS is presented by Equation (1)



The kinetics of the above reaction is impracticably slow at room temperature, and often requires many days to complete. For this reason, acid or base catalysts are added to increase the reaction rate. The amount and type of catalyst used in synthesis played key roles in the microstructural and physical properties of the silica aerogel product as will be explained later in the paper. Acid catalysts included hydrofluoric acid and hydrogen peroxide. Basic

catalysts used were ammonium hydroxide and potassium hydroxide. During the gelation process, metal foam samples were dipped in the viscous solution and were removed at moderate speed (about 0.5 cm/s). It was important to perform the process at an appropriate speed to try to obtain a uniform coating. Leaving the sample in the gel solution for long time would cause the resulting sample to have solid gel particles in the pores.

The final and most important step in making silica aerogel-coated samples is drying, where the liquid within the gel is removed, leaving only the linked silica network. The wet gel can be dried either by evaporation or by supercritical drying with CO₂ or alcohols. It was observed in a previous study (Nawaz *et al.*, 2013 and 2014 a) that when the gels were dried by evaporation, the resulting glass-like material (xerogel) cracked and the coating was not durable (Figure 2a). Instead of evaporation, if a sample was dried under supercritical conditions, the effects of surface tension resulting in cracking of the coating were eliminated and the final sample was more stable (Figure 2b). The process for the preparation of silica-aerogel-coated metal foams is summarized in the flow chart given in Figure 3.

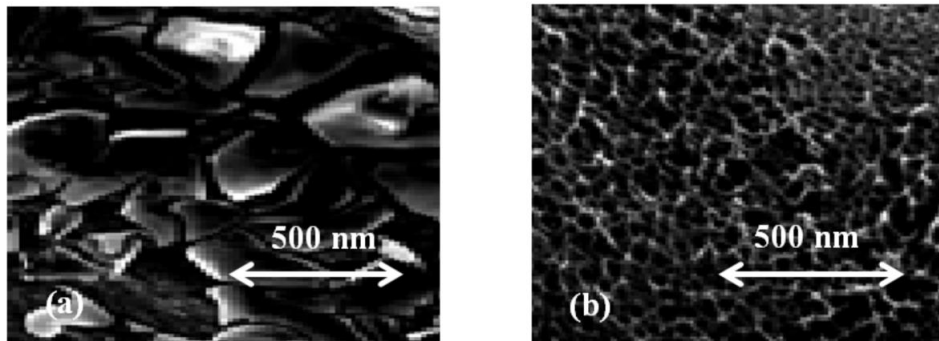


Figure 2: SEM images of silica aerogel (prepared using ammonium hydroxide catalyst) dried by (a) evaporation (b) supercritical drying (CO₂)

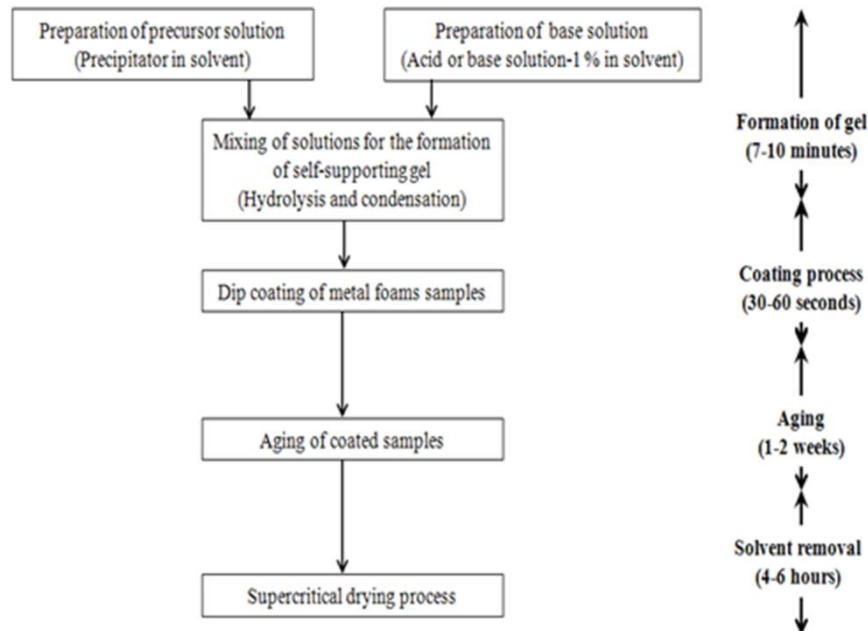


Figure 3: Manufacturing of silica aerogel coated metal foams (Adopted from Brinker and Scherer, 1990 and modified)

It is important to evaluate that the geometric characteristics of metal foam after coating change (Figure 4) to evaluate the uniformity and thickness of coating. The geometric characteristics of metal foams samples before and after coating are presented in Table 1.

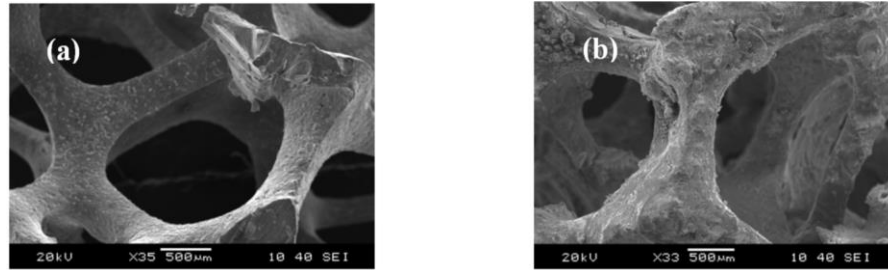


Figure 4: SEM images of 5 PPI metal foams (a) Uncoated and (b) Coated with silica aerogel (catalyst: ammonium hydroxide)

Table 1. Geometric properties of coated and uncoated metal foams

| Type of Foam (PPI) | Ligament Diameter (mm) | | Pore Diameter (mm) | |
|--------------------|------------------------|-----------------|--------------------|-----------------|
| | Uncoated | Coated | Uncoated | Coated |
| 5 | 0.50 ± 0.05 | 0.58 ± 0.06 | 4.02 ± 0.04 | 3.94 ± 0.05 |
| 10 | 0.45 ± 0.04 | 0.52 ± 0.03 | 3.28 ± 0.04 | 3.21 ± 0.05 |
| 20 | 0.35 ± 0.04 | 0.39 ± 0.04 | 2.58 ± 0.05 | 2.54 ± 0.04 |

3. MICROSTRUCTURE ANALYSIS OF SAMPLES

The catalyst used in the chemical reaction affects the microstructure of the resulting silica aerogel. In order to evaluate the effect of the catalyst used in the Sol-Gel process on microstructure, samples were prepared using different catalysts. The specimens were prepared for scanning electron microscopy. The images were taken with same magnification (40000x) for all specimens using a *Hitachi 4800* (Hitachi High-Technologies Corporation, Japan) environmental scanning electron microscope at Materials Research Laboratory (MRL) at University of Illinois at Urbana- Champaign, IL. The microstructures of some supercritically dried silica aerogel samples are presented in Figure 5.

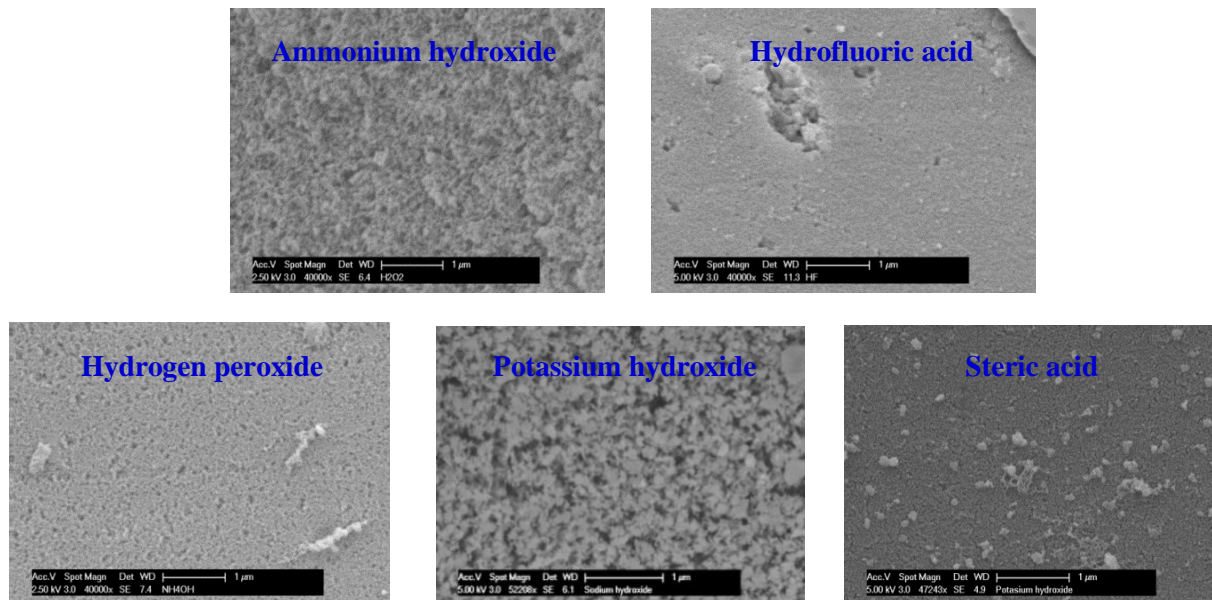


Figure 5: Microstructures of five different aerogels prepared by different catalysts

It can be observed from the images of supercritically dried silica aerogel samples that the microstructure (pore size) depends upon the catalyst used in preparation (Sol-Gel process). Samples prepared by hydrofluoric acid as the

catalyst resulted in a more dense structure with small pores, while for sodium hydroxide as the catalyst the structure was highly porous and relatively less dense. It is important to note that the titanium gold sputtering process was avoided to preserve the original microstructure. The sputtering process is often employed in SEM to increase the conductivity of electrically non-conducting material (ceramics and powders) for better magnification.

4. MOISTURE ADSORPTION/DESORPTION CAPACITY OF AEROGEL-COATED METAL FOAMS

As both equilibrium and transient adsorption/desorption behavior of the desiccant are affected by the microstructure (pore size), it is important to determine which catalyst will result in an appropriate pore size range and hence will adsorb/desorb more moisture at relatively faster rates compared to the others. Furthermore, as the substrate can also affect the adsorption/desorption rate, the effect of different types of metal foams used as substrates should be evaluated as well.

4.1 EXPERIMENTAL APPARATUS AND METHOD

For the transient response of silica aerogels as desiccants, and to determine the diffusion coefficients, a Dynamic Vapor Sorption (DVS Advantage, *Surface Measurement System, UK*) apparatus was used (Figure 6). Experiments were performed in which samples were suddenly exposed to a humid or dry environment (compared to the sample water activity) and the weight change was measured (which was converted to moisture content) as a function of time and ultimately used to calculate the diffusion coefficients.

The DVS apparatus is equipped with mass flow controllers for mixing wet and dry nitrogen gas and controlling the relative humidity, and a vapor measurement sensor to monitor the resultant relative humidity and a microbalance to determine the response of the samples. The apparatus has a dry carrier gas (nitrogen), and precise control of the ratio of saturated and dry carrier gas flows was enabled with mass flow control combined with the use of real-time vapor concentration monitoring for water. A known concentration of water vapor then flowed over a sample suspended from a recording ultra-microbalance, which was used to measure the weight change of the sample caused by adsorption or desorption of the vapor molecules.

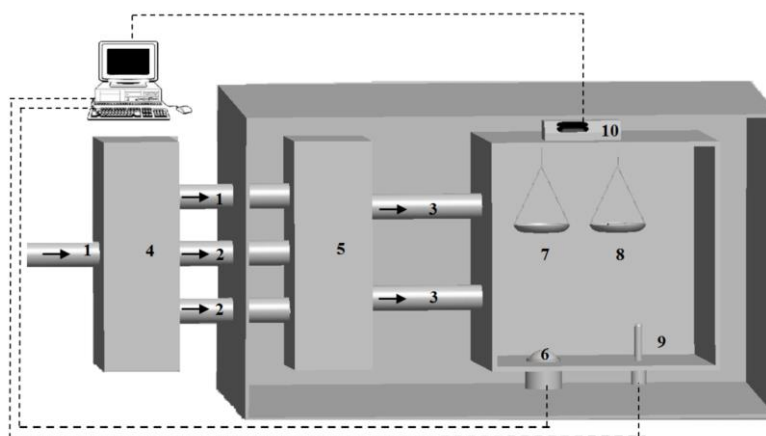


Figure 6: Apparatus for dynamic vapor sorption experiments (*Surface Measurement System, UK*) 1-Dry gas, 2-gas for mixture, 3-vapor mixture, 4-mass flow controllers, 5-vapor generator module, 6-camera, 7-sample, 8-reference, 9-humidity sensor, 10-microbalance

The temperature of the entire system was selected and precisely controlled under closed loop conditions to ensure that the solute vapor pressure at the sample is constant. The good sensitivity and precision of the DVS enables the use of very small sample sizes (typically 1 to 30 mg), thus allowing equilibrium to be reached quickly, and sample sizes smaller than 2 g were used to ensure accuracy. The instrument was fully programmable and controlled by software through a smart operator interface. All experiments were conducted at a temperature equal to 25°C.

3.2 DETERMINATION OF SORPTION/DESORPTION ISOTHERMS

The DVS sorption automatic operation (SAO) method was used to set both the desired percent relative humidity steps and the equilibrium criterion. First, each sample was equilibrated to 0 %RH using an equilibrium criterion of a change in mass over time (dm/dt) of no greater than 0.0005% for 5 consecutive minutes. After this equilibrium criterion was reached at 30%RH, the relative humidity was increased automatically to the target value (40%) and equilibrated using the same dm/dt criterion (0.0005% for 5 consecutive minutes). The relative humidity was increased with an increment of 10%. After reaching the equilibrium at 90%, the relative humidity was decreased to 20 %RH using a dm/dt criterion (0.0005% for 5 consecutive minutes) with a decrement of 10 %. Sample mass data and sample and reference chamber relative humidity and temperature data were automatically collected every 60 s. A new sample was used for each relative humidity and temperature experimental run. A silica aerogel sample in the shape of small cylinder (Figure 7) was placed on a DVS quartz round bottom sample pan (13 mm in diameter). Total gas flow was parallel to the sample surface and was set at 500 cm³/min for all experiments. Duplicate runs were done for each sample.

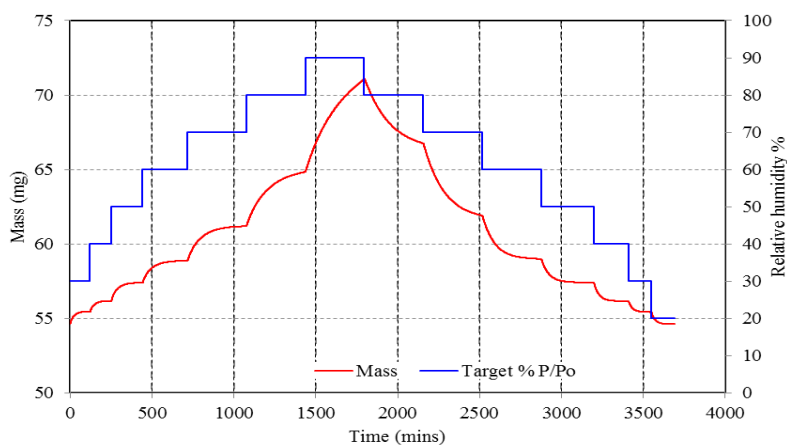


Figure 7: Mass variation of the sample due to step change in relative humidity

3.3 EFFECT OF CATALYST USED IN SOL-GEL PROCESS ON SORPTION/DESORPTION ISOTHERMS

The adsorption capacity for silica aerogels depends on their microstructure, which is affected by the catalyst used in the Sol-Gel process. A representative adsorption/desorption isotherm is presented in Figure 8. It is obvious that the equilibrium isotherm is of type IV, as classified by IUPAC (Rouquerol *et al.*, 1999). The adsorption and desorption isotherm don't follow the same path and there is a hysteresis, which exists due to the capillary condensation.

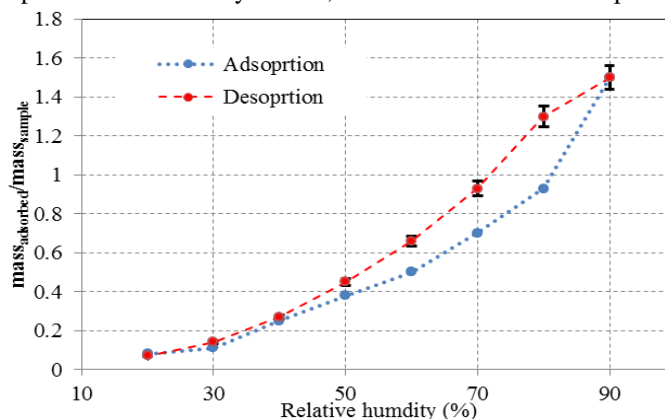


Figure 8: Hysteresis in adsorption and desorption isotherms

Adsorption and desorption isotherms for five different type of silica aerogel are presented in figure 9a and 9b. All isotherms are of type IV and the hysteresis exists for all of them. Aerogel prepared by hydrofluoric acid and by ammonium hydroxide has the maximum moisture retention capacity which can be upto 150 % of the mass of the dry

sample at 90 % relative humidity when the equilibrium is reached. While the silica aerogel prepared by hydrogen peroxide and potassium hydroxide can retain moisture only about 80 % of the mass of the dry sample under same conditions. The sorption desorption isotherms are also an indicator of the surface area of the microstructure. Hence based on the equilibrium isotherms data it can be concluded that silica aerogel prepared by hydrofluoric acid and ammonium hydroxide as catalysts have the largest surface area among the five samples prepared by different catalysts.

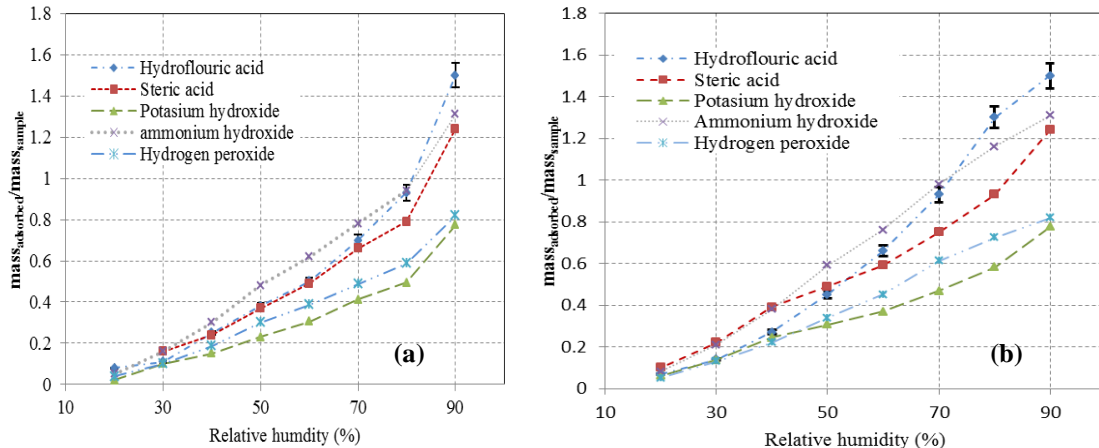


Figure 9: (a) Adsorption and (b) desorption isotherms of different silica aerogels

3.4 EFFECT OF TEMPERATURE ON SORPTION/DESORPTION ISOTHERMS

Moisture adsorption and desorption capacity of the desiccant materials is affected by pressure and temperature. The equilibrium isotherms are always presented with appropriate temperature. Desiccant used for dehumidifying application in HVAC applications typically don't go through huge temperature difference. However still it is important to account for the change based on the temperature. Adsorption and desorption isotherms for silica aerogel prepared by hydrofluoric acid at three different temperatures are presented in Figure 10. DVS equilibrium tests for conducted at 15 °C, 25 °C and 35 °C using new samples for each test. As indicated in Figure 11, the adsorption capacity of the desiccant increases about 15 %, when the experiment temperature is increased from 15 °C to 35 °C. It is expected that all other silica aerogel sample follow the same trend, that's the capacity is increased as the temperature of the environment is increased.

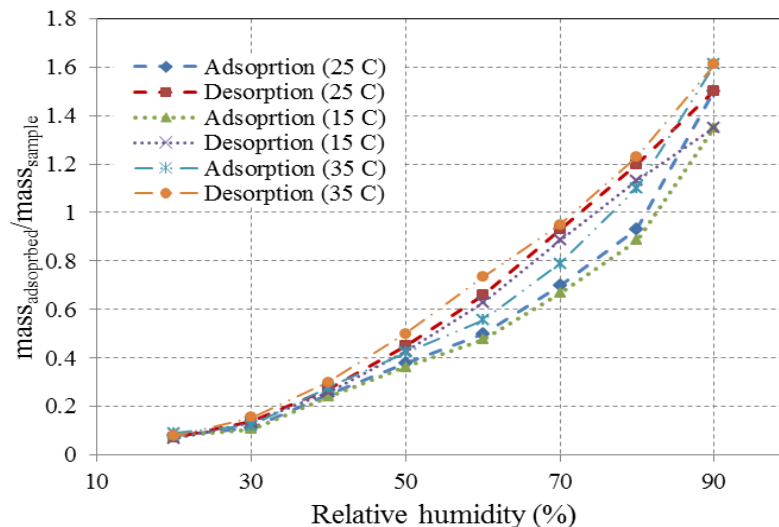


Figure 10: Effect of temperature on adsorption/desorption isotherms

3.5 EFFECT OF SUBSTRATE ON ADSORPTION/DESORPTION ISOTHERMS

Adsorption and desorption isotherms for four different cases (silica aerogel prepared by hydrofluoric acid) are presented in Figures 11a and 11b respectively. Three different of substrates have been used for comparison and to evaluate the effect of the substrate surface area for coating. As can be observed that unlike the diffusivity (mass diffusion coefficient), the adsorption capacity of the desiccant depends on the type of substrate. As the surface area increases ($1200 \text{ m}^2/\text{m}^3$ for 10 PPI metal foam, $700 \text{ m}^2/\text{m}^3$ for 5 PPI al foam), the adsorption capacity increases. Compared to the solid aerogel block an aerogel coating on the 10 PPI aluminum foam can adsorb about 20 % extra moisture for the same dry mass of the desiccant at 90 % relative humidity. It is important to note that when the silica aerogel is coated on flat aluminum plate the adsorption capacity increases only about 2%, which is not a very significant increase. However the coating on the metal foams show about 7% and 20% increase for 5 PPI and 10 PPI metal foam respectively.

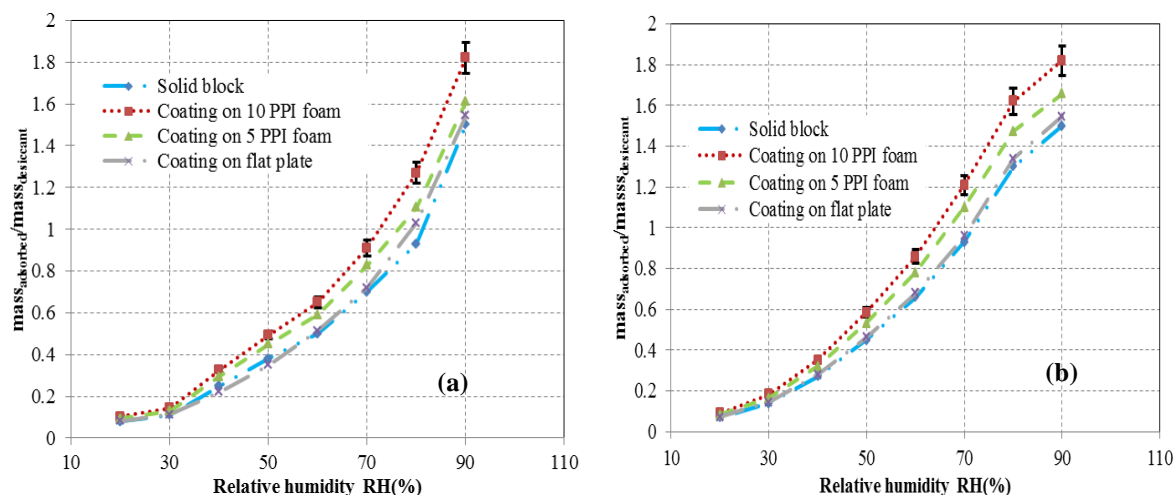


Figure 11: Effect of substrate on (a) adsorption and (b) desorption isotherms

4. CONCLUSIONS

- 1- The Sol-Gel process was used to prepare a variety of silica aerogels. Different types of acidic and basic catalysts were used in the process. The wet samples were dried using a supercritical drying process with CO_2 . Scanning Electron Microscopy was performed to analyze the pore structure of different aerogels.
- 2- It was found that the catalyst used in the process significantly affects the microstructure.
- 3- Adsorption/desorption isotherms are used to characterize the adsorption capacity of desiccants. Equilibrium sorption and desorption experiments were conducted using the Dynamic Vapor Sorption instrument, where the dry or wet samples were exposed to incremental increase/decrease in environmental humidity.
- 4- The resulted equilibrium isotherms indicated that adsorption capacity of the silica aerogels depends on the microstructure of the porous media which is affected by the catalysts used in the sol-gel process. The silica aerogel prepared by hydrofluoric acid and ammonium hydroxide showed the capacity of about 150 % the dry mass of the desiccant.
- 5- All adsorption/desorption isotherms were of type IV and showed hysteresis, which was observed due the capillary condensation phenomena.
- 6- The adsorption/desorption capacity of the desiccant is highly affect by the operation temperature. An increase of about 15 % was observed when the temperature was increased from 15°C to 35°C .
- 7- The adsorption capacity of silica aerogels when coated on metal foams was found to be almost 20 % higher compared to the adsorption capacity of solid desiccant blocks. Hence the substrate type significantly affects the adsorption capacity of the coating.

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