



Experimental study on the kinetics of water vapor sorption on selective water sorbents, silica gel and alumina under typical operating conditions of sorption heat pumps

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Abstract

A selective water sorbent (SWS) is a composite material consisting of a porous host matrix and a hygroscopic substance (commonly an inorganic salt) impregnated into its pores. This work presents an experimental investigation for the kinetics of water vapor sorption on two host materials; namely mesoporous silica gel and alumina in comparison with the two composites SWS-1L and SWS-1A formed by impregnating these two host matrices with CaCl_2 . Moreover, the kinetics of water vapor sorption on microporous silica gel have been also investigated. The measurements have been carried out on 3 g samples of loose pellets on an isothermal wall under three different operating conditions of sorption heat pumps. The results obtained evidence a remarkable increase in the differential water loading of both SWS-sorbents over their host materials. However, and due to the increased diffusion resistance to water sorption resulting from the salt impregnation, the kinetics of water sorption into the host matrices is faster than that into the two SWS-composites. Moreover, SWS-1L is found to be faster than SWS-1A in sorbing water vapor. The differential water loading on microporous silica is about twice that on mesoporous silica and alumina, but the sorption kinetics are a little bit slower. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Alumina; Calcium chloride; Porous media; Selective water sorbent; Silica gel; Sorption heat pumps; Sorption kinetics; Vapor diffusion

1. Introduction

A family of new working materials, so called selective water sorbents (SWSs), has been recently presented by Aristov et al. [1] for sorption air conditioning and heat pumps. These materials are composites “hygroscopic salt inside porous matrix” that demonstrate an intermediate behaviour between solid adsorbents, salt hydrates and liquid absorbents. The SWS sorption properties can be controllably modified, as depicted by the group of authors [1–4], by varying (a) porous

structure of the host matrix, (b) chemical nature of the impregnated salt and (c) the amount of the salt inside the pores. The study of thermodynamic properties of these materials has shown that composites based on CaCl_2 and LiBr as impregnated salts and silica gels and alumina as host matrices can operate with cycles whose maximum temperature is about 100 °C lower than that of the zeolite/water pair with COP values considerably higher than those obtainable by zeolite/water at the same temperature of the heat source.

Despite of such promising thermodynamic parameters ultimate conclusions on the feasibility of these materials for sorption heat pumping can be only drawn after the dynamic analysis of SWS behaviour under real operating conditions of sorption heat pumps (SHPs). So far the dynamic properties of the SWSs are scantily

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Nomenclature

d	differential operator
D	diffusion coefficient ($\text{m}^2 \text{s}^{-1}$)
E	energy (J)
L	solution layer thickness (m)
m	mass (kg)
\dot{m}	mass flow rate (kg s^{-1})
p	pressure (kPa)
R	gas constant ($\text{kJ kg}^{-1} \text{K}^{-1}$)
S	internal surface area of the porous sorbent (m^2)
t	time (s)
T	temperature (K)
V	volume (m^3)
x	water loading (g/100 g)

Greek symbols

Δ	difference operator
Φ	pore diameter (nm)
τ	time (s)

χ dimensionless differential water loading

Subscripts

act	activation
amb	ambient
av	average
con	condenser
des	desorber
max	maximum
MC	measuring Cell
min	minimum
0	initial value
s	sorbed
sor	sorber
sp	specific
S, dry	dry sample
VV	vapor vessel
WV	water vapor
∞	final value

known. Recently the thermal conductivity of several SWSs has been measured by Aristov et al. [5] and Tanashev and Aristov [6] at atmospheric pressure, and by Freni et al. [7] and Krainov [8] under real conditions of SHPs. The kinetics of water desorption from the composite “CaCl₂ in silica pores” have been measured by Tokarev and Aristov [9]. The heat for the desorption was supplied to the sorbent layer by a flux of hot air passing through the layer at the atmospheric pressure.

The sorption of water vapor into a porous sorbent results in the release of the so called “heat of sorption”. Accordingly, the temperature of the sorbent particle rises, which reduces its sorptive capacity. Therefore, the heat of sorption has to be removed before a further vapor can be transferred into the sorbent. Thus, the rate of vapor sorption is mainly controlled by both heat and mass transfer mechanisms within the porous bed. The investigation of the combined heat and mass transfer characteristics accompanied with such a sorption process is, therefore, very important in designing and optimizing the operation of SHPs.

A porous sorbent can be utilized in different forms in designing the sorber/desorber heat exchanger of a SHP. Investigations carried out on sorption beds using, for example, zeolite pellets [10–12] showed that the initial adsorption rate is high due to the large surface area of the zeolite bed. However, the poor thermal conductivity of zeolite beds and the poor contact between the zeolite pellets and the heat exchanger wall results in a steep drop in the adsorption rate. This leads to long cycle times and, consequently, to a low thermal output of the heat pump. In order to overcome this difficulty, Lang

et al. [13] have proposed a design of the adsorber heat exchanger, in which a mono-layer of loose zeolite pellets is applied on the surface of the fins of a finned tube heat exchanger, so that each pellet layer will have a contact with the underlying metal surface. This design has proven itself to be effective during the investigations of a multi-modular adsorption heat pump, carried out by Stricker [14].

As the kinetics of water sorption/desorption on SWSs under the real operating conditions of a SHP have not yet been studied, this work gives the first presentation of the kinetics of water sorption on two host materials; namely, mesoporous silica gel and alumina in comparison with the two composites SWS-1L and SWS-1A formed by impregnating these host matrices with CaCl₂. Besides, the kinetics of water vapour sorption on microporous silica gel have also been studied. The measurements have been carried out on 3 g samples of loose pellets on an isothermal wall under three different operating conditions of SHPs.

2. The sorption heat pump process

The ideal SHP process can be simply presented on the so-called vapor pressure diagram of SWS-water as shown in Fig. 1. The process takes place between two pressure levels and two water loading levels. The ambient temperature dictates the low pressure according to the saturation vapor pressure of the refrigerant (here water), while the heating net temperature fixes the condenser or the high pressure.

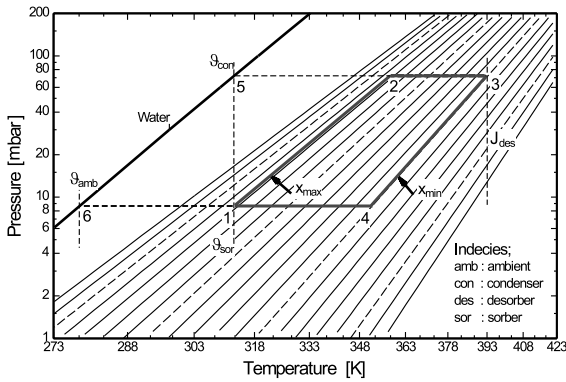


Fig. 1. Schematic presentation of the ideal SHP process on an SWS-water vapor pressure diagram.

The intersecting point of the high pressure with the available desorbing temperature decides the minimum water loading x_{min} , while the cross section point of the low pressure with the sorption or the heating net temperature fixes the maximum water loading of the cycle. The difference between the maximum and minimum water loadings is defined as the differential water loading (Δx).

$$\Delta x = x_{max} - x_{min} \tag{1}$$

For a certain set of operating temperatures, the higher the differential water loading, the higher the COP of the SHP-process [15–18]. This means that the maximum COP is to be expected from the sorbent, which attains the maximum differential water loading. It is, therefore, the first aim of this work to compare the attainable differential water loadings on the tested sorbents under typical operating temperatures of SHPs. However, the results of the numerical simulations carried out by Westerfeld [19] as well as by Miltkau and Dawoud [20] prevailed that, the average heating power of the SHP depends mainly on the time required for the selected sorbent to sorb this differential water loading, or, in other words, on the kinetics of the sorption process. This justifies the main goal of this work, which is namely to compare the kinetics of water vapor sorption on the tested sorbents.

3. Experimental apparatus, procedure, and materials

3.1. Apparatus

The sorption kinetics test-rig consists, as depicted in Fig. 2, mainly of two compartments. The first is the measuring cell, in which a sample of 3 g dry sorbent is placed as loose pellets. The temperature of the holding

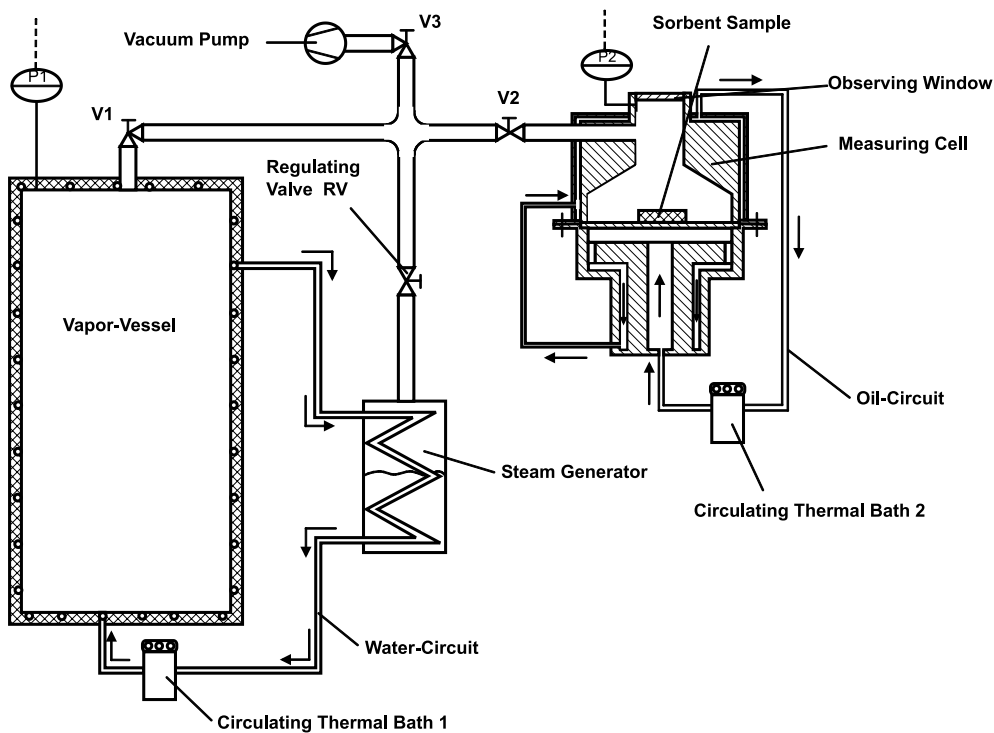


Fig. 2. Test-rig for measuring the kinetics of water vapor sorption.

surface of the sample can be adjusted and controlled using an oil circuit coupled to the circulating thermal bath 2. The second compartment is a constant volume vapor vessel. The temperature of this vessel is controlled using a water circuit coupled to the circulating thermal bath 1.

3.2. Experimental procedure

In order to activate the sorbent sample, the measuring cell is heated to 150 °C and evacuated, in the same time, for 2 h using a vacuum pump. During this desorption process, the regulating valve has to be kept closed, while the valves V1–V3 have to be opened. The remaining water loading x_0 , at the end of this activation process, has been estimated by performing thermogravimetric tests on each sample under identical boundary conditions to this activation process (150 °C and 10^{-2} mbar for 2 h). The uncertainty associated with the determination of the remaining water loading amounts to ± 0.003 g/100 g. The valves V2 & V3 are then closed and the measuring cell is cooled down to the required sorption temperature. The temperature of the vapor vessel is adjusted to the required sorption temperature, and then charged with water vapor, from the steam generator, up to the required starting pressure for the sorption process. The temperature of the connecting piping and valves has to be adjusted and controlled to the required sorption temperature.

The sorption process starts once the valve V2 is opened, resulting in decreasing the pressure in the vapor vessel. The pressure variation, being measured using the two pressure transducers P1 & P2 is used to determine the amount of the adsorbed water vapor on the sorbent sample. The accuracy of the pressure measuring system, being applied, amounts to ± 0.15 mbar. The temperature

controllers of the circulating thermal bathes have an accuracy of ± 0.2 °C. Table 1 gives the three boundary conditions of the investigated sorption processes. The sorption process lasts 3 h in order to approach the equilibrium conditions related to the sorption temperature and the final water vapor pressure $p_\infty(\text{H}_2\text{O})$. The data required for evaluating the sorption process are to be scanned and stored every second during the first hour as well as during the last 5 min of the sorption period.

3.3. The tested materials

Some characteristics of the host matrices and composites being tested are given in Table 2. The SWS-composites as well as the alumina have been supplied by the Boreskov Institute of Catalysis, Russia. The microporous silica gel has the commercial serial no. 720, while the mesoporous silica gel has the number 560. Both are fabricated and delivered by the Swiss company Uetikon.

4. Thermodynamical analysis of the experimental test-rig

A water vapor mass balance on the measuring cell of the sorption kinetics test-rig yields:

$$\frac{dm_{\text{WV,MC}}}{dt} = |\dot{m}_{\text{WV,VV}}| - \dot{m}_{\text{WV,S}} \quad (2)$$

Assuming an ideal gas behavior for the water vapor during the whole sorption process, both the rate of water vapor flow from the vapor vessel to the measuring cell $\dot{m}_{\text{WV,VV}}$ and the time rate of variation of the mass of the vapor phase in the measuring cell $dm_{\text{WV,MC}}/dt$ can be obtained according to the following Eqs. (3) and (4).

$$\begin{aligned} |\dot{m}_{\text{WV,VV}}| &= \left| \frac{\Delta m_{\text{WV,VV}}}{\Delta t} \right| \\ &= \frac{m_{\text{WV,VV}}(t) - m_{\text{WV,VV}}(t + \Delta t)}{\Delta t} \\ &= \frac{(p_1(t) - p_1(t + \Delta t))V_{\text{VV}}}{R_{\text{WV}}T_{\text{VV}}\Delta t}, \end{aligned} \quad (3)$$

$$\begin{aligned} \frac{dm_{\text{WV,MC}}}{dt} &= \frac{(m_{\text{WV,MC}}(t + \Delta t) - m_{\text{WV,MC}}(t))}{\Delta t} \\ &= \frac{(p_2(t + \Delta t) - p_2(t))V_{\text{MC}}}{R_{\text{WV}}T_{\text{MC}}\Delta t}. \end{aligned} \quad (4)$$

Table 1
The boundary conditions of the investigated sorption processes

Boundary condition	Sorption temperature (°C)	Initial pressure, $p_0(\text{H}_2\text{O})$ (mbar)	Initial relative humidity (%)
1	50	60	48.6
2	50	40	32.4
3	35	40	71.1

Table 2
Some characteristics of the host matrices and composites being tested

Sorbent	S_{sp} (m ² /g)	Φ_{av} (nm)	Salt content (wt.%)	Pellet diameter (mm)	Pellet length (mm)
SWS-1L	230	14.5	33.7	1–3	–
SWS-1A	125	6.8	28.0	2.8	3–5
Alumina A1	180	7.1	–	2.8	3–5
Microporous silica gel	700	2	–	1–3	–
Mesoporous silica gel	500	15	–	1–3	–

The adjusted sorption temperature has been assigned to the water vapor within both the vapor vessel (T_{VV}) and the measuring cell (T_{MC}). The released heat of sorption, from the sorbent sample, is thought to heat water vapor in its direct proximity. This heated vapor is then being directly sorbed such that the temperature of the bulk of water vapor within the measuring cell might be assumed constant. Combining Eqs. (2)–(4) gives the mass rate of flow of the sorbed water vapor on the sorbent sample $\dot{m}_{WV,s}$ according to Eq. (5).

$$\begin{aligned} \dot{m}_{WV,s} &= \frac{\Delta m_{WV,s}}{\Delta t} \\ &= \frac{(p_1(t) - p_1(t + \Delta t))V_{VV}}{R_{WV}T_{VV}\Delta t} \\ &\quad - \frac{(p_2(t + \Delta t) - p_2(t))V_{MC}}{R_{WV}T_{MC}\Delta t}. \end{aligned} \quad (5)$$

Rewriting Eq. (5) in (6) gives an expression for the amount of the sorbed water vapor on the sorbent sample.

$$\Delta m_{WV,s} = \frac{|\Delta p_1|V_{VV}}{R_{WV}T_{VV}} - \frac{\Delta p_2V_{MC}}{R_{WV}T_{MC}}. \quad (6)$$

Knowing the mass of the dry sorbent sample $m_{S,dry}$ as well as the starting water loading x_0 , the time variation of the water loading can be calculated due to Eq. (7).

$$x = x_0 + \sum_{t=0}^t \frac{\Delta m_{WV,s}}{m_{S,dry}}. \quad (7)$$

In order to compare the results of measuring the sorption kinetics under different operating conditions, it is reasonable to represent the time variation of the water loading in a dimensionless form. This may occur by defining the dimensionless differential water loading χ as the ratio between the instantaneous differential water loading to the maximum differential water loading

achievable at each operating condition of the sorption process on the sorbent sample.

$$\chi(t) = \frac{x(t) - x_0}{x_\infty - x_0}. \quad (8)$$

5. Results and discussion

Figs. 3–5 give the kinetic curves of water sorption on the sorbents studied in a “water loading vs. time” presentation. The maximum half-time of water sorption $\tau_{\chi=0.5} = 4.85$ min. was measured for SWS-1A at $p_0(\text{H}_2\text{O}) = 60$ mbar and $T = 50$ °C. For this sorbent, this time appears to be only slightly dependent on both temperature and the initial vapour pressure (Tables 3–5). The fastest sorption ($\tau_{\chi=0.5} = 1.0$ min) was found for pure mesoporous silica at $p_0(\text{H}_2\text{O}) = 40$ mbar and $T = 50$ °C. The sorbents with a half-time of water sorption of a few minutes observed under operating conditions of a SHP can be considered as quite suitable for designing SHPs with a short working cycle. In Tables 3–5, the given final water loading x_∞ is the water loading at the end of the sorption period (3 h). For the sake of comparison we present, in the last column of these tables, calculated values of the equilibrium water loading x_{eq} for the SWS-1L and SWS-1A at the final sorption conditions, that means for the sorption temperature and the final vapor pressure $p_\infty(\text{H}_2\text{O})$. For these calculations, we made use of the sorption equilibrium data of these sorbents given in the form of the equilibrium water loading vs. the relative humidity by Aristov et al. [3]. Both experimental and calculated data are close to each other, hence, after long times (here 3 h) the sorbent indeed reaches the true sorption equilibrium.

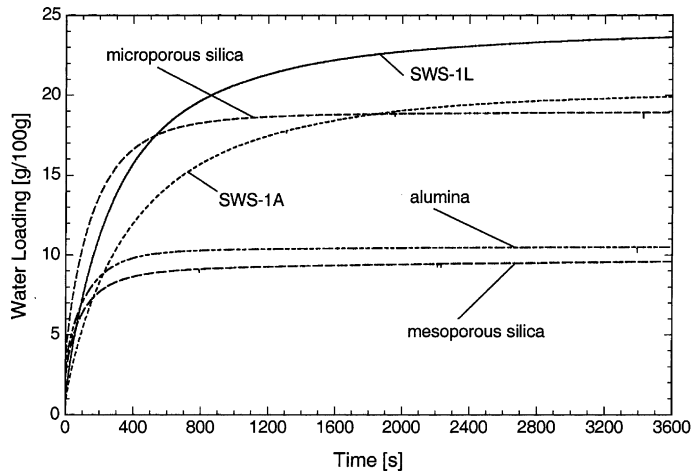


Fig. 3. A comparison between the sorption kinetics of water vapor on the tested sorbents at a starting pressure of 60 mbar and an sorption temperature of 50 °C.

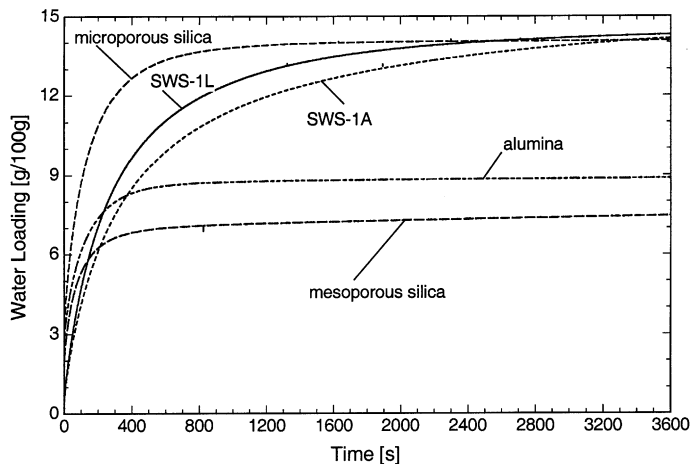


Fig. 4. A comparison between the sorption kinetics of water vapor on the tested sorbents at a starting pressure of 40 mbar and an sorption temperature of 50 °C.

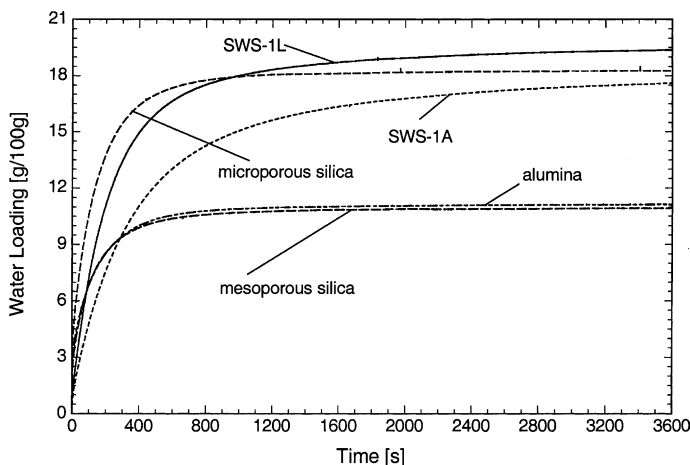


Fig. 5. A comparison between the sorption kinetics of water vapor on the tested sorbents at a starting pressure of 40 mbar and an sorption temperature of 35 °C.

Table 3

A comparison of the sorption kinetics characteristics of the tested sorbents at a starting pressure of 60 mbar and an sorption temperature of 50 °C

Sorbent	Time (min) $\tau_{\gamma=0.5}$	Time (min) $\tau_{\gamma=0.9}$	x_0 (g/100 g)	x_{∞} (g/100 g)	$x_{\infty} - x_0$ (g/100 g)	x_{eq} (g/100 g)
SWS-1L	3.95	23.82	0.17	24.23	24.06	23.4 ± 1.0
SWS-1A	4.85	25.68	0.12	20.31	20.19	20.0 ± 0.4
Microporous silica	1.80	8.52	2.57	19.01	16.44	–
Mesoporous silica	1.13	8.62	1.54	9.68	8.14	–
Alumina	1.23	6.47	2.16	10.62	8.46	–

Unmodified host matrices adsorb water faster than the composites obtained by impregnating these matrices with calcium chloride (Tables 3–5), thus the presence of the salt inside pores slows down the rate of water vapor mass transfer. At least two possible effects can be responsible for this rate moderation:

(a) The mean free path of water molecules at $P(\text{H}_2\text{O}) = 35\text{--}60$ mbar and $T = 35\text{--}50$ °C is larger than 50 μm , which is much greater than the mean pore diameter of silica or alumina (Table 2). It means that collisions between a water molecule and the pore wall are much more frequent than

Table 4

A comparison of the sorption kinetics characteristics of the tested sorbents at a starting pressure of 40 mbar and an sorption temperature of 50 °C

Sorbent	Time (min) $\tau_{\chi=0.5}$	Time (min) $\tau_{\chi=0.9}$	x_0 (g/100 g)	x_{∞} (g/100 g)	$x_{\infty} - x_0$ (g/100 g)	x_{eq} (g/100 g)
SWS-1L	3.83	25.88	0.17	14.64	14.47	15.5 ± 1.0
SWS-1A	4.83	31.57	0.12	14.62	14.50	14.3 ± 0.4
Microporous silica	1.58	9.23	2.57	13.44	11.53	–
Mesoporous silica	1.00	9.27	1.57	7.58	6.04	–
Alumina	1.18	6.18	2.16	8.93	6.77	–

Table 5

A comparison of the sorption kinetics characteristics of the tested sorbents at a starting pressure of 40 mbar and an sorption temperature of 35 °C

Sorbent	Time (min) $\tau_{\chi=0.5}$	Time (min) $\tau_{\chi=0.9}$	x_0 (g/100 g)	x_{∞} (g/100 g)	$x_{\infty} - x_0$ (g/100 g)	x_{eq} (g/100 g)
SWS-1L	2.77	14.75	0.17	19.58	19.42	19.0 ± 1.0
SWS-1A	4.50	24.63	0.12	17.95	17.83	18.0 ± 0.4
Microporous silica	1.63	7.92	2.57	18.3	15.73	–
Mesoporous silica	1.35	7.67	1.54	11.02	9.48	–
Alumina	1.57	8.32	2.16	11.24	9.08	–

the collisions between the molecules (Knudsen diffusion). After the collision with the wall, the molecule remains on the surface for some time τ that is defined by the activation energy of desorption E_{act} and the temperature T as $\tau \sim \exp(E_{\text{act}}/RT)$. According to the calorimetric studies carried out by Pankratev et al. [21], the heat of water adsorption on composites “an inorganic salt confined to a porous matrix” is found to be higher than on the pure host matrix. This means that water molecules are bound with the confined salt more stronger than with the pure matrix. Of course, this can increase the activation energy of desorption and slows down the water diffusion.

- (b) The formation of the solution film near the external pellet surface as it was found by Koptug et al. [22] for the process of water sorption from the air flux passing around the SWS-1L and SWS-1A cylindrical pellets. As a result, this solution film creates a strong mass transfer resistance for vapor to penetrate to the internal part of the pellet. The diffusion of water through the solution film towards the pellet centre or the salt molecule could be a limiting stage of water sorption. In this case the sorption rate is mostly determined by the film properties and slightly depends on vapor pressure and temperature that is in fact observed experimentally, especially for SWS-1A. If one considers the salt diffusion coefficient D in the concentrated aqueous solution of calcium chloride to be equal to 1.3×10^{-5} cm²/s, as given by Baron [23], the brief estimation of the typical diffusion time through the solution layer of thickness $L = 0.1$ cm

can be done $t \approx L^2/2D = 400$ s ≈ 7 min that is close to the observed values of $\tau_{\chi=0.5}$.

Regardless the sorption boundary conditions, the process is definitely faster on SWS-1L compared to that on SWS-1A. The mean pore diameter of the alumina host matrix is twice smaller than that of the silica (Table 2), hence, in small alumina pores the diffusion coefficient in the confined salt solution is expected to be smaller in respect to that in the larger silica pores.

From Figs. 3–5 as well as Tables 3–5, it is to be concluded that, the differential water loading on microporous silica gel is 70% to 100% higher than that obtained on mesoporous silica gel and alumina, according to the sorption boundary conditions. Comparing the measured half-times of water sorption $\tau_{\chi=0.5}$ on these sorbents reveals, however, that the sorption kinetics on microporous silica gel are a little bit slower. The differential water loadings obtained on the tested SWS-composites are found to be 22% to 46% higher than those obtained on microporous silica gel.

As the water sorption process is accompanied by a significant heat release, the temperature of the sorbent is going up while water is being sorbed. As measured by Freni et al. [7], the dissipation of the sorption heat is slow under vacuum conditions, and the increase in the SWS-pellet temperature can reach 15–40 °C. Accordingly, the studied process of water sorption is likely to be neither isobaric nor isothermal, and for extracting kinetic parameters from the experimental curves no simple kinetic models can be used and more sophisticated mathematical modelling is strictly necessary. This will be

the subject of our future research activities. At such non-isothermal condition the difference in the sorption rate between the samples is smaller compared to the rate that would be observed at a constant sorption temperature. In fact, the increase in the pellet temperature, which is higher for a more active sorbent, moderates the sorption rate due to the decrease in the equilibrium uptake at higher temperature.

Nevertheless, the experimental procedure used can be considered as a proper one for comparative studies of different sorbents under the operating conditions of the real cycle of SHPs. Moreover, it is tried, in these experiments, to simulate the sorbent heat exchanger proposed by Lang et al. [13], where it is suggested to make use of one layer of loose sorbent pellets in a direct contact to each heat exchanger fin without a binder. The authors supposed that this structure “combines a large mass transfer with sufficient heat transport characteristics”. Indeed, from the data presented in the paper one can conclude that this statement is true at least regarding the mass transport of water vapour inside the single pellets of the sorbents studied. The sorption rate is highest for the non-impregnated mesoporous silica, however the similar silica impregnated with calcium chloride has a much greater final water loading.

6. Conclusion

The kinetics of water vapor sorption on two host materials; namely mesoporous silica gel and alumina in comparison with the two composites SWS-1L and SWS-1A formed by impregnating these two host matrices with CaCl_2 have been experimentally investigated. In addition, the kinetics of water vapor sorption on microporous silica gel have been investigated. The measurements have been carried out on 3 g samples of loose pellets on an isothermal wall under three different operating conditions of SHPs.

The salt impregnation results in an increase of about two to three times in the differential water loading on the SWS-composites compared to their host materials. Due to the increased diffusion resistance to water sorption, resulting from the salt impregnation and the formation of a sorbed solution film on the outer surface of the SWS-pellets, the kinetics of water sorption into the host matrices is faster than that into the two SWS-composites. The time required to reach 50% of the final differential water loading on the host materials is found to be about 25% of the time required for the SWS-composites. However, the measured half time for the SWS-composites of about 4 to 5 min, is fairly adequate for applying SWS to SHP applications. SWS-1L is found to be faster than SWS-1A in sorbing water vapor. It needs nearly 60% to 80% of the time required for SWS-1A to reach 50% of the final differential water loading. The

differential water loading on microporous silica is about twice that on mesoporous silica and alumina, but the sorption kinetics are a little bit slower.

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