

# Composite silicone-SAPO-34 foams: experimental characterization for open cycle applications

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**Abstract.** In this work, novel composite silicone-SAPO-34 foams have been prepared and experimentally characterized for application in desiccant open cycles. Water adsorption isotherms of several samples have been measured by a gravimetric dynamic vapour sorption analyser at 30°C and 70°C up to the relative humidity  $RH=75\%$ , representing typical process and regeneration air conditions in desiccant evaporative cooling cycles. Adsorbent foams manufactured with 20%, 40% and 60% weight fraction of SAPO-34 have been compared with the pure SAPO-34 powder. Results highlighted that the prepared foams adsorb a significant amount of water, according to the initial mass fraction of zeolite used in the compound. Moreover, the tested foams exhibited sufficiently fast water sorption rate for practical application in a desiccant open cycle system.

## 1 Introduction

Desiccant evaporative cooling cycles can significantly contribute to develop Heating, Ventilating and Air Conditioning (HVAC) systems characterized by high energy efficiency and low environmental impact [1, 2]. At present, attention is given to the development of new sorption compounds, in particular aimed at reducing the regeneration temperature [3]. In addition, there is an increasing interest in developing new composite foams, which can be effectively used in heat driven cycles for air dehumidification and cooling processes. In literature, an innovative silicone-zeolite composite foam has been investigated for heat pump closed cycle application [4]. Foamed materials, due to their intrinsic high surface area per unit mass, could allow to obtain a large amount of zeolite coating per unit of volume, and at the same time the foam porosity could act as a preferential pathways for the vapour diffusion. This approach allows improving the amount of active material embedded into the adsorbent heat exchanger. Being the zeolite a compound typically used in open cycle desiccant wheels [5], in this work several samples of silicone-SAPO-34 composite foams have been prepared and morphological and water adsorption properties have been investigated. More specifically, adsorption isotherms transient water uptake have been measured by a gravimetric adsorption apparatus in typical operating conditions of such systems.

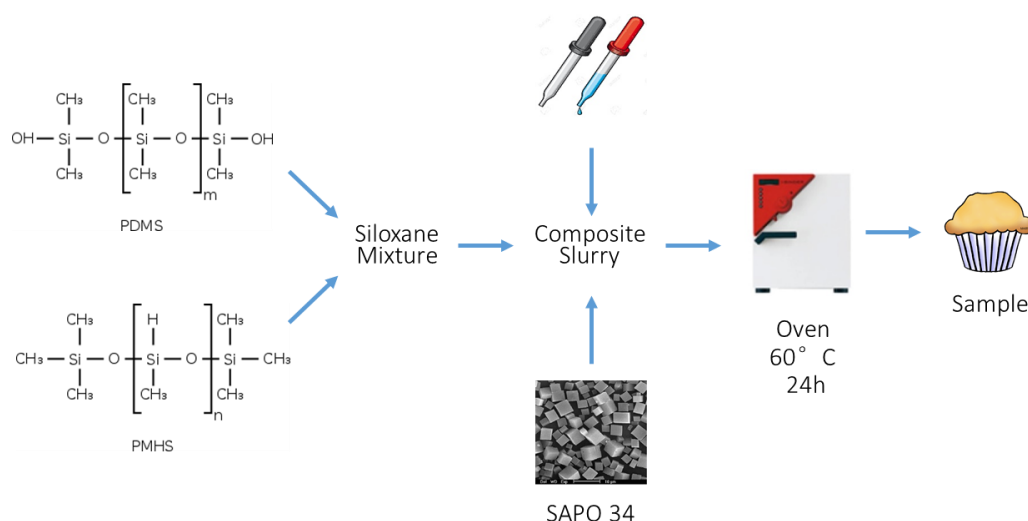
## 2 Sample preparation

Adsorbent foams were produced following a direct foaming approach, according to the following procedure: SAPO-34 filler was dispersed under high shear mixing in polydimethylsiloxane (PDMS) and polymethylhydrosiloxane (PMHS) mixture (PDMS/PMHS weight ratio 1:2). Water and ethanol were added to reduce the slurry viscosity. Finally, a catalyst was added under vigorous mixing, for about 15s. The so obtained mixture was poured into a cylinder mould to obtain blowing. Foaming was started putting the sample into an oven kept at a controlled temperature (60 °C) for 24 hours to obtain a complete compounds reaction. Three foam samples of SAPO-34-silicone foams have been produced (Fig. 1): the SAPO-34 mass fraction is respectively 20% (Z20), 40% (Z40) and 60% (Z60).



Fig. 1. View of the three SAPO-34-silicone foams

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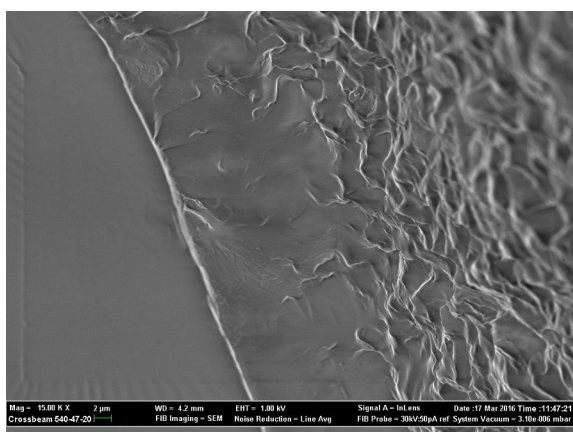


**Fig. 2:** Scheme of preparation steps of the silicone-SAPO-34 composite foam

Figure 2 schematically shows the preparation procedure; more details are reported in [4]. The samples were codified by means of the code: Z plus a number, where Z means zeolite foam while the number indicates the percentage of the SAPO-34 added to the matrix (i.e. Z20 designates the composite foam filled by 20% of SAPO-34). The obtained foams were cut in order to get cubic samples with edge of about 20 mm.

Porous structure of the composite foam is obtained by a combined action of crosslinking and foaming reactions of the siloxane constituents. The foaming phenomenon is due to a combined chemical and physical blowing. The former is due to hydrogen evolution generated by the chemical reaction among siloxane compounds: The latter is due to the evaporation of the solvent during the curing stage.

Finally, foam morphology was evaluated by ion beam scanning electron microscope (ZEISS SEM/FIB crossbeam 540). Figure 3 shows a SEM image of a bubble



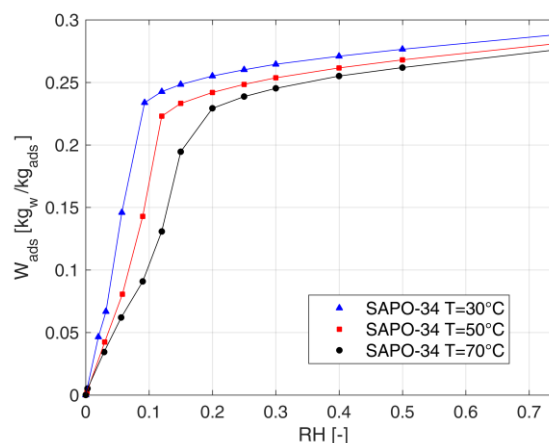
**Fig. 3:** SEM image of bubble wall cross-section in Z60 sample

wall section of a Z60 foam. An optimal interaction between the zeolite filler and the silicone foam can be observed. The sample is defect-free and without voids confirming the good interconnection of the zeolite crystals in the silicone matrix.

### 3 Experimental results

Water adsorption isotherms of the adsorbent foams have been measured by a gravimetric adsorption apparatus (Aquadyne DVS). In addition, a sample of pure SAPO-34 has been investigated and compared to literature results. According to equipment technical specifications, the reference state weight is measured at 80°C, in nitrogen atmosphere and at ambient total pressure.

Firstly, water adsorption isotherm of pure SAPO-34 have been measured at different temperature: at 30°C, 50°C and 70°C. As shown in Fig. 4, according to literature [5], pure zeolite shows a sharp uptake rise at low relative



**Fig. 4:** Adsorption isotherm of pure SAPO-34 powder at different temperature

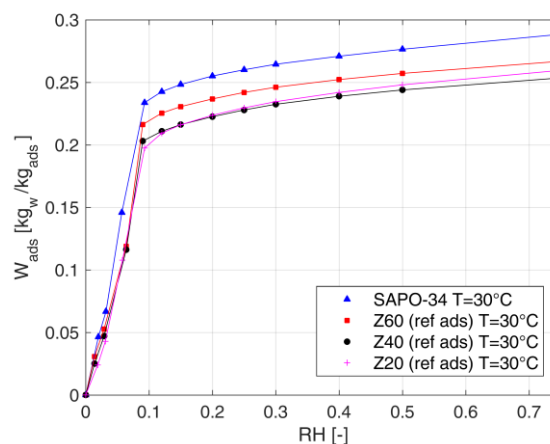
humidity ( $RH < 15\text{-}20\%$ ), which is favourable for open cycle applications. In addition, the higher the temperature the lower the amount of adsorbed water.

Secondly, the water adsorption isotherm of Z20, Z40 and Z60 have been measured at  $30^\circ\text{C}$  and  $70^\circ\text{C}$ , which represent common dehumidification and regeneration conditions adopted in open air cooling cycles. In Fig. 5 and 6 the obtained results for pure zeolite and the three compounds are shown. It is possible to highlight that all the Z20, Z40 and Z60 foams keep the same isotherm shape of the pure sorption material both at  $30^\circ\text{C}$  and  $70^\circ\text{C}$ .

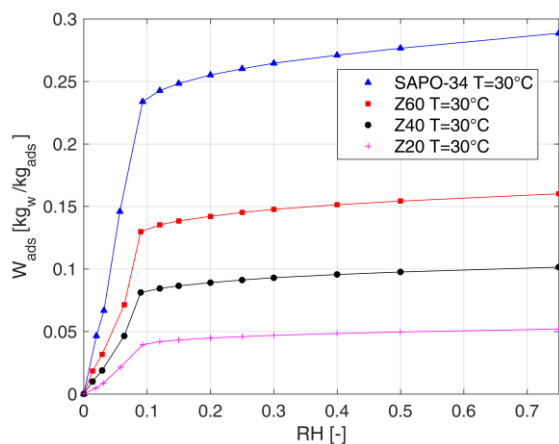
The reduced adsorption capacity is in agreement with the zeolite mass fraction. The silicone does not participate in the adsorption process and, therefore, the water uptake capacity is reduced compared to pure desiccant. Anyway, as shown in Figs. 7 and 8, if the adsorption capacity is referred to the dry SAPO-34 mass of each sample instead of to the total dry mass (silicone and zeolite), adsorption isotherm curves of pure desiccant and of foams are very close: the maximum measured reduction in the sorption capacity is around 14%. Note that Z20 performs slightly better than Z40 and Z60, even if the SAPO-34 mass fraction is lower, mainly due to the manual manufacturing process that leads to not perfectly homogenous samples. Based on aforementioned considerations, it is possible to

state that the silicone matrix does not limit the water adsorption phenomena significantly.

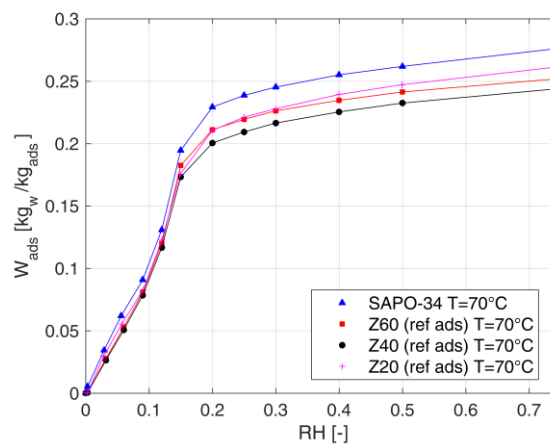
Similar conclusions could be obtained also observing the transient water vapour uptake. In Fig. 9, the transient water vapour uptake curves are shown for Z40 and Z60 foams at  $70^\circ\text{C}$ . The two composites respond to relative



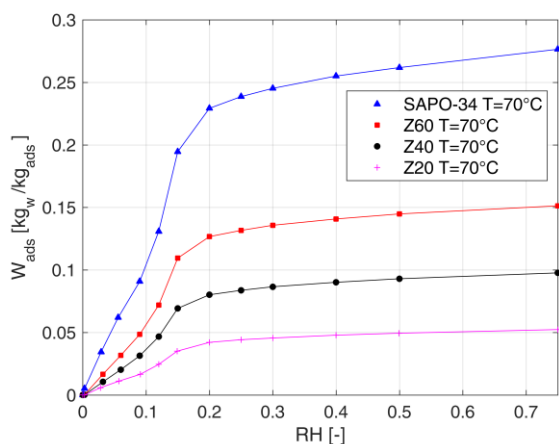
**Fig. 7:** Adsorption isotherm of pure SAPO-34 and prepared Z20, Z40 and Z60 foams at  $30^\circ\text{C}$  (reference mass equal to SAPO-34 content)



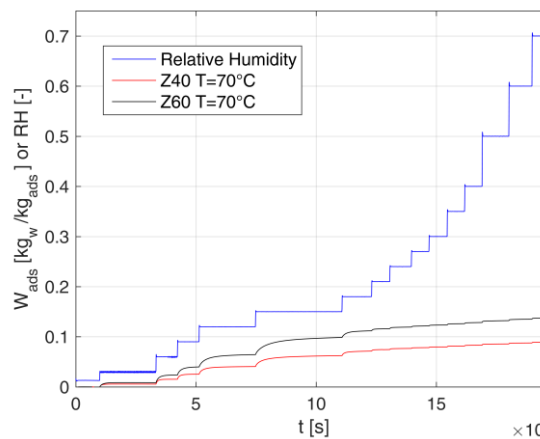
**Fig. 5:** Adsorption isotherm of pure SAPO-34 and prepared Z20, Z40 and Z60 foams at  $30^\circ\text{C}$



**Fig. 8:** Adsorption isotherm of pure SAPO-34 and prepared Z20, Z40 and Z60 foams at  $70^\circ\text{C}$  (reference mass equal to SAPO-34 content)



**Fig. 6:** Adsorption isotherm of pure SAPO-34 and prepared Z20, Z40 and Z60 foams at  $70^\circ\text{C}$



**Fig. 9:** Transient water vapour uptake of prepared Z40 and Z60 foams at  $70^\circ\text{C}$

humidity step variation in a similar way and independently of the zeolite mass fraction.

The careful evaluation of the influence that such composite foams and related measured properties could have on performance of desiccant HVAC devices will be subject of a future paper.

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## 4 Conclusions

Based on first obtained results, the investigated foams are a promising compound that can be used to develop new desiccant components for open cycle systems. It is shown that silicone does not reduce significantly steady state and transient adsorption capacity of pure zeolite.

Further studies will deal with experimental analysis and modelling of actual components based on proposed Silicone-SAPO-34 foams, in order to effectively evaluate potential uses and advantages of proposed composite in real devices.

## Nomenclature

### Acronyms

DEC	Desiccant Evaporative Cooling
Z20	Silicone Foam, 20% weight of SAPO-34
Z40	Silicone Foam, 40% weight of SAPO-34
Z60	Silicone Foam, 60% weight of SAPO-34

### Symbols

$RH$	Relative humidity [-]
$t$	Time [s]
$T$	Temperature [°C]
$W$	Average water content [ $\text{kg}_v/\text{kg}_{\text{ads}}$ ]

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