



Performance study of composite silica gels with different pore sizes and different impregnating hygroscopic salts



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HIGHLIGHTS

- Composite silica gels were prepared for solid desiccant cooling systems.
- Morphologies of composite desiccants were different from those of pure silica gels.
- Salt category and pore size affected the sorption performance of composite desiccants.
- Performance curves of composite materials were divided into three sections.

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ABSTRACT

Composite solid desiccant materials are fabricated by impregnating hygroscopic salts into pores of different silica gels. In this paper, sorption characteristics of composite desiccants with different pore sizes (namely, 2–3 nm, 7–8 nm and 9–10 nm) of silica gels and different salts in terms of LiCl, LiBr and CaCl₂ are tested and analyzed. Researches on nitrogen adsorption suggest that, due to impregnated salt particles, composite desiccants are different from those of pure silica gels and have smaller surface area and pore volume. Sorption isotherms and sorption kinetics with respect to different composite desiccant materials are tested and fitted with the Dubinin–Astakhov (D–A) equation and the Linear Driving Force (LDF) model, respectively. Results showed that both the pore size of silica gel substrates and categories of impregnated salts affect the sorption characteristics of composite desiccants. Microporous silica gel with pore size of 2–3 nm is not fit for preparing composite desiccants due to decreased water sorption quantity and sorption rate. Type B and mesoporous silica gels can be a promising host matrix of composite desiccant owing to enhanced water sorption quantity and favorable sorption rate. In addition, composite desiccants employing LiCl have the best sorption properties, while CaCl₂ and LiBr follow next.

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1. Introduction

With the rapid development of economy and society, large amount of energy is consumed for cooling in our modern life and industry. Solid desiccant cooling technology, which can handle sensible and latent heat loads independently together with the merits of energy saving and environmental protection, has received increased attention (Daou et al., 2006a; La et al., 2010). The dehumidification performance of desiccant cooling systems largely depends on the sorption properties of desiccant materials (Collier, 1989; Golubovic et al., 2006). As one of the most common desiccant materials, silica gel is widely utilized in dehumidification due to its high porosity, stable sorption characteristics and low cost (Tashiro et al., 2004; Wang et al., 2009). However, it

has the disadvantage of low water exchange within a typical dehumidification cycle because the water sorption mainly occurs at very high relative pressures (Yu et al., 2013). To solve this problem, some researchers have focused on the modification of its texture by introducing inorganic hygroscopic salts, alumina, hydrophilic organic network, etc. to improve its affinity for water (Chung and Chung, 1998; Fang et al., 2005; Vradman et al., 2005; Gong et al., 2010). Among these, impregnation of hygroscopic salts has been proved by numerous researchers to be a simple and effective way to improve the water sorption capacity of silica gels (Aristov et al., 2006; Daou et al., 2006b; Gordeeva et al., 1998; Saha et al., 2009; Simonova and Aristov, 2005).

Aristov et al. (2002) developed composite adsorbents from microporous and mesoporous silica gels with the support of CaCl₂, respectively. Large water sorption amount as high as 0.7 g g⁻¹ was obtained for composite samples made from mesoporous silica gels. Jia et al. (2007) developed a composite desiccant by impregnating LiCl into the pores of silica gel. Results showed that the dehumidification amount of

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composite desiccant wheel under practical operation increased by 20–40% over the conventional silica gel one. Later, Simonova et al. (2009) fabricated a composite desiccant by impregnating silica gel with saturated aqueous solution of $\text{Ca}(\text{NO}_3)_2$. This material had reasonable water sorption exchange of $0.15\text{--}0.17\text{ g g}^{-1}$ at a low desorption temperature ($75\text{--}80\text{ }^\circ\text{C}$). Subsequent work by Aristov et al. (2012) demonstrated that a much lower heat source ($65\text{--}75\text{ }^\circ\text{C}$) was possible for the regeneration of SG (KSK)/ LiNO_3 composite adsorbent.

Quite recently, Bu et al. (2013) prepared a series of composite adsorbents by soaking silica gels of different pore sizes into different CaCl_2 solutions with varied mass concentrations in the range of 10–50 wt%. Experiment results showed that water adsorption capacity and adsorption rate of composite samples were affected by both pore size of silica gel and confined CaCl_2 content. Similar work by Gordeeva et al. (2003) and Yu et al. (2014) also demonstrated that water adsorption quantity of composite silica gels improved with the increasing of mass content of confined salt. In addition to the salt concentration, categories of hygroscopic salts also play a role in the water adsorption quantity of silica gel-based composites (Gordeeva et al., 2006; Gordeeva and Aristov, 2012; Mrowiec-Bialon et al., 1999). Li et al. (2010) developed a series of composite adsorbents by impregnating CaCl_2 , ZnCl_2 , MgCl_2 , LiCl , KCl and NaCl into the pores of mesoporous silica gels, respectively. Results showed that composite silica gels prepared from CaCl_2 or LiCl had higher sorption capacities and better water sorption rates at relative humidity less than 80%. Cortés et al. (2012) manufactured composite adsorbents by impregnating mesoporous silica gel into aqueous solutions of LiBr , MgCl_2 and CaCl_2 . Water sorption isobars showed that composite samples employing CaCl_2 had the best water uptake, while LiBr and MgCl_2 followed next.

It can be seen that numerous investigations have been conducted by impregnating inorganic hygroscopic salt into silica gel as host in recent years. The pore size of silica gel substrates and categories of immersed salts can affect the sorption performance of composite desiccant materials. According to Hall et al. (2012), by correlating hygrothermal function properties with microstructural characteristics (pore volume, surface area, pore size, etc.), certain required property of a candidate material for any given applications may be achieved. However, there is few literature data available on the comprehensive discussion and comparison of composite silica gels with different pore sizes and different impregnating hygroscopic salts under practical conditions of solid desiccant systems. Furthermore, publications describing the relevance of sorption performance of composite silica gels with their microstructural characteristics are almost non-existent. Therefore, this paper presents the study of some key parameters (microstructural characteristics and sorption performance) regarding the development of a series of silica gel-based composite desiccants with the support of different hygroscopic salts. Firstly, surface area and pore parameters (pore volume, pore size and pore size distribution) were characterized by N_2 adsorption at 77 K. Then water sorption isotherms and sorption kinetics of composite samples were conducted and investigated. The Dubinin–Astakhov (DA) equation and Linear Driving Force model (LDF) were used to simulate the sorption properties of these desiccants. Appropriate impregnated salts and silica gels were determined based on the above measurements and analysis. Finally, dehumidification performance of composite desiccant in a novel solid desiccant cooling component named desiccant coated heat exchanger (DCHE) was evaluated.

2. Material and methods

2.1. Synthesis of composite silica gels

The materials used in this paper mainly included three types of silica gels with different pore sizes, three hygroscopic salts (i.e.

Table 1

Surface area and pore parameters of different silica gels.

Type	Designation	d_p (mm)	S_{BET} ($\text{m}^2\text{ g}^{-1}$)	V_t ($\text{cm}^3\text{ g}^{-1}$)	d_{av} (nm)
Microporous	SGA	0.2–1.0	706	0.4	2.3
Type B	SGB	0.2–1.0	487	0.82	7.5
Mesoporous	SGC	0.2–1.0	395	0.93	9.4

LiCl , LiBr , CaCl_2) and deionized water. The commercial silica gels were used as host and purchased from Qingdao Meigao Chemical Co., Ltd., and three hygroscopic salts were purchased from Sino-pharm Chemical Regent Co., Ltd. The parameters of silica gels such as particle size (d_p), BET surface area (S_{BET}), total pore volume (V_t) and average pore diameter (d_{av}) are listed in Table 1. It shall be noted that the symbols SGA, SGB and SGC denote microporous, type B and mesoporous silica gels, respectively.

These composite desiccants were prepared by impregnating dry silica gels with each saturated aqueous solution of hygroscopic salt for 24 h; then a vacuum filter was utilized to obtain the composite samples from the solutions. After that, they were dried at $120\text{ }^\circ\text{C}$ for at least 4 h until the weight remained unchanged. By weighing these dry samples before and after the impregnation, salt contents in the composites were determined. Mass concentrations of confined salt solutions were 45.5 wt%, 62.5 wt% and 42.7 wt% for LiCl , LiBr and CaCl_2 , respectively. In this paper, nine composite desiccants with different silica gels and hygroscopic salts were manufactured. The samples are named as “Silica gel/immersed salt”, which demonstrate the constitution of composite desiccant material. For example, “SGC/ LiCl ” refers to composite desiccant obtained by impregnating LiCl solution into the pores of mesoporous silica gels.

2.2. Test of surface area and pore parameters

The surface area and pore parameters such as specific surface area, total pore volume, average pore size and pore size distributions of composite desiccants were tested and analyzed by Micromeritics gas adsorption analyzer (ASAP 2020). This analyzer utilizes the principle of static volumetric technique (Pierotti and Rouquerol, 1985) to obtain nitrogen sorption isotherms, then applies multiple gas sorption laws such as BET equations (Classics Brunauer et al., 1938), BJH method (Barrett et al., 1951) and Density Functional Theory (DFT) (Parr, 1983) to obtain information on surface area and pore parameters of a solid material.

Test samples were first degassed at $120\text{ }^\circ\text{C}$ for at least 8 h in a vacuum to remove impurities. Then, dry samples were analyzed at $-196\text{ }^\circ\text{C}$, controlled by liquid nitrogen. The experimental error of ASAP apparatus is mainly caused by temperature and pressure transducers. The accuracy of temperature transducer is $\pm 0.02\text{ }^\circ\text{C}$ and the testing errors of pressure ones are about 0.1%.

2.3. Test of sorption kinetics

The dynamic water sorption quantities of composite desiccants were measured in a thermo-humidistat chamber, with pure silica gels as reference. This constant temperature and humidity chamber has a space of 3 m (length) \times 3 m (width) \times 2.45 m (height). It can supply constant air condition with deviations of $\pm 0.5\text{ }^\circ\text{C}$ for temperature and $\pm 3\%$ for relative humidity. The defined sorption operation condition was set at $20\text{ }^\circ\text{C}$ and a relative pressure of 0.7. The water sorption amount was denoted as per water uptake on per unit mass of the composite desiccants (i.e. g g^{-1}).

The experiment steps were as follows: (1) heating test samples in an oven at $120\text{ }^\circ\text{C}$ for at least 4 h to remove moisture; (2) weighing dry samples in an electronic balance with

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