



Structure and adsorption properties of the composites ‘silica gel–sodium sulphate’, obtained by sol–gel method



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HIGHLIGHTS

- The composites ‘silica gel–sodium sulphate’ are synthesized by sol–gel method.
- Their sorption properties exceed Na_2SO_4 and silica gel mechanical mixture.
- The composites hydration is controlled with water transport on matrix pore system.
- The composite heat energy storage capacity is 0.36 kWh/kg or 258 kWh/m³.

ARTICLE INFO

Article history:

Received 24 June 2013

Accepted 7 December 2013

Available online 17 December 2013

Keywords:

Composite sorbent

Adsorption

Heat storage material

Heat accumulating capacity

ABSTRACT

The composite sorbents ‘silica gel–sodium sulphate’ synthesized by sol–gel method are studied. The sorption capacity of the composite ‘silica gel–sodium sulphate’ is stated to be 0.85 g/g that exceeds noticeably the linear superposition of the sorption capacity of the silica gel and sodium sulphate mechanical mixture which is 0.66 g/g. The composite ‘silica gel–sodium sulphate’ composition and structure are studied by IR-spectroscopy and wide-angle X-ray scattering. The changing of adsorption properties of crystalline Na_2SO_4 at their allocation in silicon–oxygen matrix is shown to be result from dispersion up to nanoscale. The experimental setup for effectiveness tests of synthesized composite sorbents is developed. The correlation of their composition, structure, water adsorption kinetic and operating characteristic as heat storage material is shown. The heat storage capacity is stated to be 0.36 kWh/kg (258 kWh/m³).

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1. Introduction

The world community initiatives on reducing of carbon dioxide and other hothouse gases emission stimulate not only search of alternative energy sources, improvement of high temperatures industrial application, but also heat energy storage including lower temperature potential. An adsorptive technology for energy storage and transformation is one of the engineering solution at that. Nevertheless, current status of technical level in the field cannot be considered as wholly satisfactory, in particular, because of poor properties of adsorbent materials [1]. Recently attempts of their improvement are focused on existing sorbent selection [2,3], as well as on synthesis and investigation of composites ‘salt inside porous matrix’ [1,4,5] due to their enhanced sorption capacity to common working fluids. Silica gel [6,7], alumina [4], $\gamma\text{-Al}_2\text{O}_3$ [8],

vermiculite [9,10], carbon sibeunit [8], attapulgite [10], burnt clay [11] are used as porous matrix at that, and salts are lithium bromide and magnesium chloride [4], calcium chloride [5,11], barium chloride [8], lithium chloride [10], lithium nitrate [12], binary mixtures of LiCl and LiBr, BaCl_2 and BaBr_2 , CaCl_2 and CaBr_2 [7]. Adsorptives are water vapor [4,5,12], ammonia [7,9], methanol [7]. It is obvious, that heat energy storage is based on three different mechanisms founded on heat capacity of solid medium, phase transition, and adsorption. The salt reacts with water vapor that results in the formation of a complex crystalline hydrate of the salt. Further adsorption leads to the complex dissolution and transformation to the ‘salt–sorbate’ solution. The composite sorbents ‘salt inside porous matrix’ are promising to get wide spread occurrence in low-temperature heat energy accumulating devices [7,12], adsorption cooling [9], heat and moisture regeneration in ventilation and conditioning systems [6,10], gas drying and extraction of potable water from the atmosphere [4,11]. The main advantages of composite sorbents are not only possibility of controllable modification of their sorption properties, but also low charging temperature and

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Nomenclature

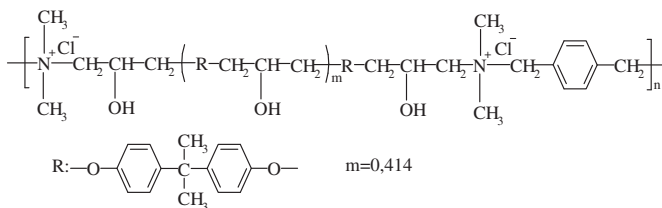
m_v	water mass
m_s	sorbent mass
P_{H_2O}	partial pressure of water vapor
I	intensity
2θ	angle

high heat storage capacity. For instance, regeneration temperature of the composite LiNO₃/vermiculite (SWS-9V) is lower than 65–70 °C, it being demonstrated heat storage capacity of 900 kJ/kg [12], i.e. 0.25 kWh/kg. Thus, their production is concerned with applying of quite complex and rather expensive technologies of porous media dry and wet impregnation by crystalline hydrate solutions [8,11] that inhibit their commercialization in energy storage devices. This problem can be solved by synthesis of composite ‘silica gel–crystalline hydrate’ with sol–gel method [13]. Sorption capacities sol–gel sorbents CaCl₂/silica gel reach 1 kg of water/kg of adsorbent, and regeneration temperature of these materials is 200 °C [13].

The aim of this paper is to investigate properties of the composite ‘silica gel–sodium sulphate’ synthesized with sol–gel method (composition, structure, water vapor sorption kinetics) and its operating characteristics as heat energy storage material.

2. Experimental

The composite sorbents ‘silica gel–sodium sulphate’ synthesized from tetraethoxysilane, Na₂SO₄, ethanol (as a solvent) and hydrochloric acid (as a catalyst) were chosen as the objects of investigation. Polyionenes served as organic modifiers. The structural formulas of polyionenes are given below:



The modifying organic additives ($8 \cdot 10^{-4}$ mol) were preliminarily dissolved in water and then were introduced portion wise into the sols containing tetraethoxysilane (1.0 mol), Na₂SO₄ (1.0 mol), ethanol (4.0 mol), and hydrochloric acid (0.01 mol). Thereafter, the sols were stored in closed vessels to gelation. The resulting gels were dried under vacuum for 8 h to the formation of coarse-grained powders. Detailed synthesis technique is described in Ref. [14]. Diameters of the composite sorbents granules are in the range of 0.5–1.5 mm.

The DTA analysis was conducted on a Paylic–Paylic–Erdy derivatograph Q-1500J, using a heating rate of 10 °C/min, from room temperature to 900 °C. The samples mass is 200 mg. IR-spectrum are registered on UR – 20. Wide-angle X-ray scattering (WAXS) profiles of the samples were collected in the transmission mode using a DRON-2,0 diffractometer. Nickel-filtered Cu-K_α radiation (radiation wavelength $\lambda = 0.154$ nm) was produced by an IRIS-M7 generator at an operating voltage of 30 kV and a current of 30 mA. The scattering intensities were measured using a scintillation detector scanning in 0.2° steps over the range of angles of 2–40°. The diffraction curves obtained were normalized for the intensity of the primary beam and the scattering volume by the usual

technique [15]. The setup described in Ref. [16] is applied for investigation of water sorption kinetics.

The effectiveness tests of synthesized composite sorbents are carried out on the experimental setup (Fig. 1). The setup includes heat-insulated cored tubing (1), filled with heat accumulating material, through which fan by compressor (3). The heater (4) for regulating air temperature is located behind compressor. Thermopairs (T) for air temperature control are set into the setup in the inlet, in the outlet, along heat accumulator zones and on the outside. Mass changing during test is registered by balance (5). Temperature is controlled by thermopairs TP-2088/5 (producer scientific production plant «ELEMER», Russia). The sensor EE23, model C (producer E+E Elektronik Ges.m.b.H., Austria) is used for air humidity control. Air speed is controlled by high-precision industrial sensor EE75, model C (producer E+E Elektronik Ges.m.b.H., Austria).

Airflow is maintained of 50 m³/h, and its humidity is 70–80%. The heat energy storage material mass is 3.5 kg until adsorption.

Quantity of heat adsorption of water with the composite sorbent could be calculated by trivial formula

$$Q = Cp \cdot \rho \cdot \Delta T \cdot V \quad (1)$$

where $Cp = 1012$ is medium isobaric heat capacity of dry air, kJ/(kg K); $\rho = 0.825$ is medium air density, kg/m³; ΔT is air temperature difference on the input and output of tubing with heat energy storage material, K; $V = 90$ is volume of air passed through the composite sorbent, m³.

A theoretical value of heat storage capacity could be calculated by formula [17]

$$Q = \Delta h \cdot \rho \cdot C \cdot \left(\frac{1000}{Mw} \right) \cdot \left(\frac{1}{3600} \right) \quad (2)$$

where $\Delta h \cong 60$ is heat adsorption of water vapor, kJ/mol; $\rho = 720$ is packed density of the composite sorbent, kg/m³; C is maximum moisture-holding capacity; $Mw = 18$ is the molar mass of water, g/mol.

3. Results and discussion

The composite mass loss after desorption during heating to 140–1000 °C is 2%. The composite mass loss after air moisture adsorption is observed at 32–100 °C to be 46%. DTA analysis decahydrate sodium sulphate, anhydrous sodium sulphate and silica gel KSM dried to constant mass at 100 °C which can be considered as confirmed difference of investigated composite sorbents and

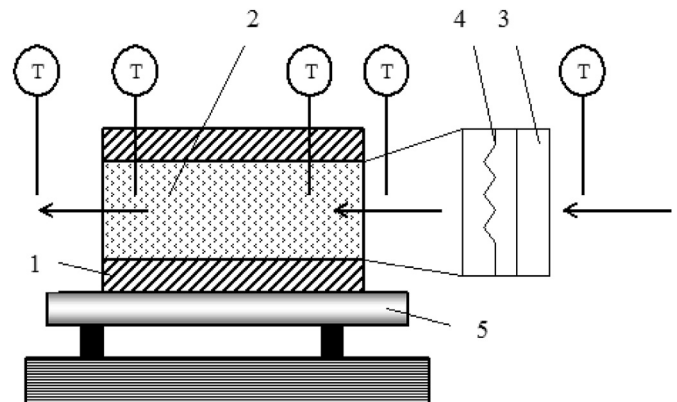


Fig. 1. Experimental setup for heat storage material investigation.

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