

Novel sorbents of ethanol “salt confined to porous matrix” for adsorptive cooling

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ABSTRACT In this work a new family of sorbents, specifically designed for ethanol sorption, is presented. The composites were synthesized by impregnation of matrices with an aqueous solution of different salts. The ethanol sorption capacity of composites, under conditions of typical adsorptive air conditioning cycle, was measured by using an express method based on the Polanyi principle of temperature invariance. Results obtained showed that several of the composites synthesised present ethanol sorption ability which is higher than that of known ethanol sorbents. The composite LiBr(30 wt.%)/SiO₂ appears to show the highest sorption capacity and an uptake variation $\Delta w = 0.56$ and 0.40 g/g per air conditioning and ice making cycles, respectively, that are much larger than those for conventional adsorbents. The correspondent thermodynamic cooling COP was estimated to be 0.66 and 0.61, which is comparable with typical cooling COP of the best water sorbents.

Keywords: adsorptive cooling, ice making, ethanol, host matrix, inorganic salt

1. INTRODUCTION

Environmentally benign adsorptive cooling (AC) is considered as a promising alternative to compression that [1-3]. Water and methanol are typical refrigerants for AC driven by low temperature heat like solar energy or waste heat of automotive engine. Methanol has high vapor pressure at working conditions of the AC cycle and low freezing point that make it promising for both the air conditioning and the ice making [4-6]. However, it is toxic and harmful for environment and so its usage is forbidden in some countries. For this reason an increasing attention has recently been paid to ethanol as a refrigerant [7-11]. Actually, ethanol has rather high vapor pressure at operating conditions of AC cycles (15-170 mbar) and low freezing point (-114 °C) in comparison with water vapor and, in contrast to methanol, it is not toxic.

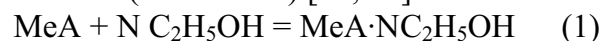
However, low latent heat of ethanol (840 J/g) strongly restricts an efficiency of AC cycles. In order to overcome this intrinsic drawback new adsorbent materials with advanced sorption properties adapted to particular AC cycle are welcome.

The typical ethanol adsorbent used in AC application is activated carbons [7, 11, 12]. However, they possess not sufficient sorption capacity [13, 14] resulting in low performance of the AC cycle. Li et al. showed that ice could not be produce in a solar assisted AC device with “activated carbon – ethanol” working pair due to low net sorption per cycle [7]. Some other materials have been proposed for ethanol sorption: a smectite modified with long-chain monofunctional ammonium cations [15], poly vinyl acetate [16], porous-clays heterostructures prepared by combining pillaring and templating approaches [17], microporous cadmium hexacyanoferrate(III)

[18], composite adsorbent NB [10] and activated carbon fibers (ACF) [8, 9]. However, the uptake difference per cycle does not exceed 0.10 g/g for the most of these sorbents. ACF materials demonstrate the largest sorption variation per cycle of 0.25-0.50 g/g.

Recently the new family of composites “salt inside porous matrix” (CSPMs) has been proposed for water and methanol sorption [19-22]. These materials were shown to combine advantages of solid porous adsorbents and liquid absorbents that results in high sorption capacity and low regeneration temperature. The salt was shown to be the primary sorbing component of the composites. The sorption mechanism on the CSPMs includes formation of solid crystalline solvates with monovariant sorption equilibrium. This gives stepwise sorption isobars and favours to large variation in sorption in narrow temperature range required for efficient AC units and, consequently, to high performance of AC cycles. It is worth noting that the sorption properties of CSPMs can be modified intently by the number of “tools” like changing chemical nature of the salt and the matrix, porous structure of the matrix, salt content and preparation conditions [23, 24]. It makes possible the design of the composite which properties fit the particular AC cycle.

This study was purposed on the extending of this approach to preparation of CSPMs for ethanol sorption specialized for adsorptive air conditioning and ice making cycles. It is based on the fact that a number of inorganic salts MeA can reversibly absorb ethanol by forming crystalline solvates (ethanolates) [25, 26]



The work consists of three parts. At the beginning a review of literature data on the system “salt-ethanol” was performed in order to choose salts promising for ethanol sorption. Then a number of the composites, based on the chosen salts and various matrices were synthesised. Their sorption

ability at typical operating conditions of AC cycle was scanned by an express method developed earlier [21]. Finally the properties of CSPMs, which showed the largest variation in sorption per cycle, were studied in more details by barometric method [27, 28] and the performance of both air conditioning and ice making cycles was estimated.

2. EXPERIMENTAL

2.1 Synthesis of CSPMs

Matrices of various chemical nature, namely, hydrophilic (silica, alumina) and hydrophobic (synthetic carbon Sibunit) ones with large pore volume were used. Another parameters affecting the sorption equilibrium of the composites varied during the composite preparation was the average pore diameter of the matrix (Table I). The composites were synthesized by a wet impregnation of the silica gel with an aqueous salt solutions followed by thermal drying at 473 K. The salt content in the composites was determined by weighting dry samples before and after impregnation.

Table I: Main characteristics of the matrices used: pore volume (V_p), surface area (S_{sp}), average pore diameter (d_{av}).

Matrix	V_p , cm ³ /g	S_{sp} , m ² /g	d_{av} , nm
Silica gel Davisil Gr. 646	1.2	300	15
Silica gel Grace SP2-8506	0.9	340	10
Sibunit	0.9	450	6-8
Alumina	0.7	220	6
ACF15	1.03	1900	2.2
ACF20	0.77	1400	2.2

2.2 Sorption measurements

Scanning of the sorption ability of the composites at the working conditions of AC cycles was performed by an express method based on the Polanyi principle of temperature invariance [29]. According to

this principle the equilibrium content of ethanol adsorbed w can be uniquely determined by the product $T \ln \frac{P}{P_s}$ [30]. For

the rich isoster of the cycle (stage 1-2 in Fig. 1), corresponding to $w = w_{\max}$, the following equation is true.

$$\Delta F(w_{\max}) = -RT_{\text{ads}} \ln \frac{P_{\text{ev}}}{P_s(T_{\text{ads}})} = -RT_2 \ln \frac{P_{\text{con}}}{P_s(T_2)} \quad (2)$$

whereas for the weak isoster (stage 3-4) ($w = w_{\min}$), the equilibrium can be written as

$$\Delta F(w_{\min}) = -RT_{\text{con}} \ln \frac{P_{\text{con}}}{P_s(T_{\text{con}})} = -RT_4 \ln \frac{P_{\text{ev}}}{P_s(T_4)} \quad (3)$$

The typical operating conditions for air conditioning cycle driven by low temperature heat (Fig. 1) are the following: the evaporator temperature $T_{\text{ev}} = 283$ K, condenser temperature and minimal temperature on the adsorption stage $T_{\text{con}} = T_{\text{ads}} = 303$ K and the maximum temperature at the desorption stage $T_{\text{des}} = 363$ K. Those for the ice making cycle are $T_{\text{ev}} = 270$ K, $T_{\text{con}} = 303$ K, $T_{\text{ads}} = 298$ K and $T_{\text{des}} = 363$ K. The free sorption energy corresponding to the rich isosters $\Delta F(w_{\max}) = 3.05$ and 4.34 kJ/mol for air conditioning and ice making cycles respectively, while the free sorption energy corresponding to the weak isoster $\Delta F(w_{\min}) = 8.26$ kJ/mol. Taking into account the expression $\Delta F(w_{\max}) = -RT_4 \ln (P_{\text{ev}}/P(T_4))$ the temperature T_4 can be calculated as 338 K.

The sorption measurements were carried out according to the following procedure. The air flow saturated with ethanol vapor up to the partial pressure $P_{\text{ev}} = 30$ mbar ($T_{\text{ev}} = 283$ K) or $P_{\text{ev}} = 12$ mbar ($T_{\text{ev}} = 270$ K) for air conditioning and ice making cycles, respectively, was passed through the adsorber filled with the dry composite at temperature $T_{\text{ads}} = 303$ and 298 K for air conditioning and ice making cycles, respectively, until the weight became constant. The desorption was carried out at the temperature of the adsorber $T_4 = 338$ K and ethanol pressure $P_{\text{ev}} = 30$ mbar. Methanol uptake after adsorption and desorption stages was measured as

$$w \left(\frac{g_{\text{EtOH}}}{g_{\text{sorbent}}} \right) = \frac{m}{m_0} \quad (4)$$

or as a number of methanol molecules adsorbed by one molecule of the salt

$$N \left(\frac{\text{mole}_{\text{EtOH}}}{\text{mole}_{\text{sorbent}}} \right) = \frac{\frac{m}{M_{\text{EtOH}}}}{\frac{m_0 C}{M_{\text{salt}}}} \quad (5)$$

The sorption per cycle Δw was calculated as the difference in the methanol uptakes at adsorption $w_{\min}$ and desorption stages $w_{\max}$.

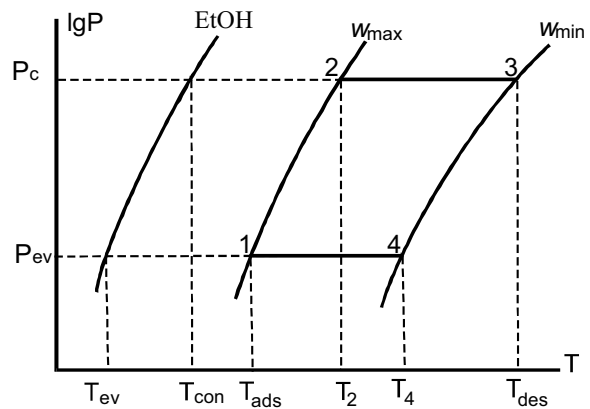


Figure 1: Thermodynamic cycle of adsorptive cooling.

Isosters of the ethanol sorption were measured by a barometric method [27]. About 50 g of the sorbent was placed inside a vacuum chamber and pumped to a residual pressure of 0.01-0.03 mbar at $T = 393$ K. Then the chamber was connected to an evaporator filled with a known amount of liquid ethanol, so that the sample was saturated with ethanol vapour up to a fixed uptake. Then the chamber was disconnected from the evaporator and the sample was kept at a closed volume for 24 hours in order to reach a uniform ethanol distribution inside the sample. Finally, the sample was heated to a fixed temperature and the ethanol equilibrium pressure over the sample was measured. During the experiment the temperature range was adjusted so that the pressure ranged from 1 to 160 mbar. During the heating phase a change in the ethanol uptake due to its partial desorption into a “dead” volume over the sample was estimated to be $\Delta n \approx (1 -$

$2 \cdot 10^{-3}$ mole C_2H_5OH /mole of the salt or $\Delta w \approx 10^{-3}$ g/g.

3. RESULTS AND DISCUSSION

3.1 Ethanol sorption ability

It is known that a number of inorganic salts react with ethanol forming solvates with different coordination number N from 0.5 to 6 (Table II) [25, 26]. The parameters of the solvate decomposition (ethanol pressure P_{dec} and temperature T_{dec}) vary in a wide range giving different values ΔF_{dec} . Taking into account the variation in ΔF at typical AC cycle using ethanol as refrigerant $3.05 \text{ kJ/mol} < \Delta F < 8.26 \text{ kJ/mol}$ (see Chapter 2.2) the salts, which ethanolates occur decomposition at proper ΔF_{dec} range, were chosen for the composite preparation. The number of the composites prepared for ethanol sorption as well as their main properties are presented in Table III.

Sorption ability of the composites measured at operating conditions of the air conditioning cycle is presented in Table 3 and Fig. 2 together with literature data for known ethanol adsorbents given for comparison [7-18]. Several new composites possess rather high sorption ability at the working conditions of the cycle. The most promising salts appear to be LiBr and LiCl, the adsorption uptake of the LiCl/SiO₂(Gr646) and LiBr/SiO₂(Gr646) composites reaches $w_{ads} = 0.4$ and 0.68 g/g , respectively. The major part of ethanol adsorbed can be removed during desorption stage giving the uptake variation per cycle as large as $\Delta w = 0.3\text{-}0.56 \text{ g/g}$. LiBr confined to pores of silica gel shows the best methanol exchange ($\Delta w = 0.56 \text{ g/g}$), which exceeds significantly that for the conventional adsorbents. LiBr/SiO₂ composite performs quite good sorption ability $w_{ads} = 0.45 \text{ g/g}$ ($N = 3.0$) and the uptake variation $\Delta w = 0.34 \text{ g/g}$ for ice making cycle.

Table II: Temperature (T_{dec}), pressure (P_{dec}), free sorption energy ΔF_{dec} of decomposition of ethanol solvates of various salts.

Salt	N, mole/ mole	T_{dec} , K	P_{dec} , mbar	ΔF_{dec} , kJ/ mol
CaCl ₂	3	30	10.2	5.77
CaBr ₂	3	30	14.3	4.92
CuCl ₂	2	30	57.0	1.44
MgCl ₂	6	30	4.2	8.00
CoCl ₂	3	30	7.3	6.62
CoBr ₂	3	30	13.8	5.01
Mg(NO ₃) ₂	6	30	19.1	4.19
Ca(NO ₃) ₂	2	30	36.6	2.56
CdCl ₂	1.5	30	53.3	1.60
CdBr ₂	1.5	30	65.9	1.08
SrBr ₂	0.5	20	20.0	3.94
LiCl	4	-	-	-
LiBr	4	-	-	-

Table III: Composition and properties of the composite sorbents synthesized: salt content (C), ethanol uptake after adsorption (w_{ads}), desorption (w_{des}) stages and uptake variation per cycle (Δw).

Composition	C, wt. %	Ethanol uptake		
		w_{ads} , g/g	w_{des} , g/g	Δw , g/g
LiCl/SiO ₂ *	24	0.41	0.08	0.33
LiCl/SiO ₂ **	16	0.22	0.07	0.15
LiBr/SiO ₂ *	30	0.68	0.12	0.56
CaCl ₂ /SiO ₂ *	23	0.2	-	-
CaBr ₂ /SiO ₂ *	22	0.26	0.11	0.15
SrCl ₂ /Al ₂ O ₃	15	0.05	-	-
NiBr ₂ /SiO ₂ *	44	0.07	-	-
Ca(NO ₃) ₂ /SiO ₂ *	27	0.18	0.14	0.04
Mg(NO ₃) ₂ /SiO ₂ *	22	0.27	0.12	0.15
MgSO ₄ /SiO ₂ *	24	0.08	-	-
CuSO ₄ /SiO ₂ *	17	0.08	-	-
LiBr/Sibunit	29	0.42	0.17	0.26
LiCl/ACF15	25	0.35	0.17	0.18
LiCl/ACF20	42	0.52	0.23	0.29

* – silica gel Davisil Grade 646

** – silica gel Grace SP2-8506

Ethanol is a weak polar compound and it absorbs better by hydrophobic adsorbents. So it can be expected that the replacement of the hydrophilic matrix (silica gel) with hydrophobic one (synthetic carbon Sibunit) would result in the increase in sorption ability. However, the LiBr/Sibunit composite sorbs ethanol worse than the silica based ones. This confirms our previous observation that the confined salt is a primary sorbing component of CSPMs, and the matrix is mostly a dispersing agent. Confinement of the salt to narrow pores ($d_{av} < 6-8$ nm) results in the decrease in the sorption ability of the salt-ethanol solution forming inside pores during sorption ($N_{ads} = 4.3$ and 2.7 for the silica and Sibunit based composites, respectively) that agrees well with our data on water sorption on CSPMs [30, 31].

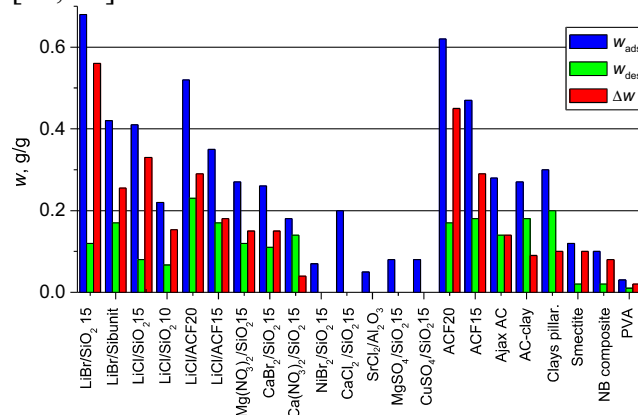


Figure 1: Ethanol sorption ability of the composites and solid adsorbents at operating conditions of air conditioning cycle ($T_{ev} = 283$ K, $T_{con} = T_{ads} = 303$ K, $T_{des} = 363$ K).

Among porous adsorbents an activated carbon fibers ACF20 performs the largest uptake variation ($\Delta w = 0.47$ g/g), which, however, remains lower than that for LiBr/SiO₂(Gr646) composite. The advantage of this composite becomes more pronounced in term of ethanol sorption per unit volume of the adsorbent. Sorption capacity related to unit volume w_{ads} for LiBr/SiO₂(Gr646) composite was 476 kg/m^3 , instead of 30 and 100 kg/m^3 for CF20 with the packing density $\rho = 50$ and 150 kg/m^3 [8]. Modification of ACFs by LiCl results in the enhancement of their

sorption ability related to 1 g of ACF, $w_{ads} = 0.89$ and 0.46 g/g for LiCl/ACF20 and LiCl/ACF15 composites, respectively. However, their sorption ability even decreases when related to the composite mass $w_{ads} = 0.52$ and 0.35 g/g.

The sorption ability of the composites based on $\text{Mg}(\text{NO}_3)_2$ and CaBr_2 under working conditions of the AC cycle is quite good $w_{ads} = 0.27$ g/g ($N_{ads} = 4$ and 5.2 , respectively). However, the large part of ethanol remains adsorbed after the desorption stage giving low variation in the uptake $\Delta w < 0.1$ g/g. The sorption ability of the composites based on $\text{Ca}(\text{NO}_3)_2$, NiBr_2 , CaCl_2 , SrCl_2 , MgSO_4 and CuSO_4 appears to be rather low $w_{ads} < 0.2$ g/g.

3.2 LiBr/SiO₂(Gr646) composite.

The sorption properties of LiBr/SiO₂(Gr646) composite, which appears to be the most promising for AC application were studied in more details (Figure 3). The results of isosteric measurements agree well with those obtained by the express-method. The uptake variation $\Delta w = 0.56$ and 0.40 g/g at operating conditions of air conditioning and ice making cycles, respectively.

Isosteric measurements allows an evaluation of the isosteric enthalpy and standard entropy of ethanol sorption (Table III) using the Van't Hoff equation [32]

$$\ln P_{\text{EtOH}} = \Delta H_{is}/RT - \Delta S^0/R \quad (6)$$

The isosteric enthalpy of sorption ranges from $\Delta H_{is} = -51 \pm 2$ to -45 ± 2 kJ/mol that is higher the ethanol latent heat ($\Delta L = 38.6$ kJ/mol). It trends to decrease with the increase in uptake that can be caused by weaker bonding between the salt ions and solvating ethanol molecules. The standard entropy remains almost constant $\Delta S^0 = -121 \pm 5$ J/(mol K).

3.3 Estimation of the cooling performance.

The data obtained allows an estimation of the coefficient of performance (COP) of AC cycles based on "LiBr/SiO₂(Gr646)

composite – ethanol” working pair considering the following approximations:

1) the equivalent specific heat C_p of each composite is calculated as a sum of the specific heat of the host matrix, of the salt and of the ethanol adsorbed [33],

$$C_{p_{eq}}(w) = C_{p_{salt}}C_{salt} + C_{p_{SiO_2}}(1 - C_{salt}) + wC_{p_{EtOH}} \quad (7)$$

The specific heat of the composites at stage 2-3 in the cycle (Fig. 1) was calculated for the average ethanol uptake $w_{av} = (w_{min} + w_{max})/2$;

2) the latent heat of ethanol is calculated considering the medium temperature of the working cycle;

3) the desorption enthalpy is calculated as a medium value of those for various uptake.

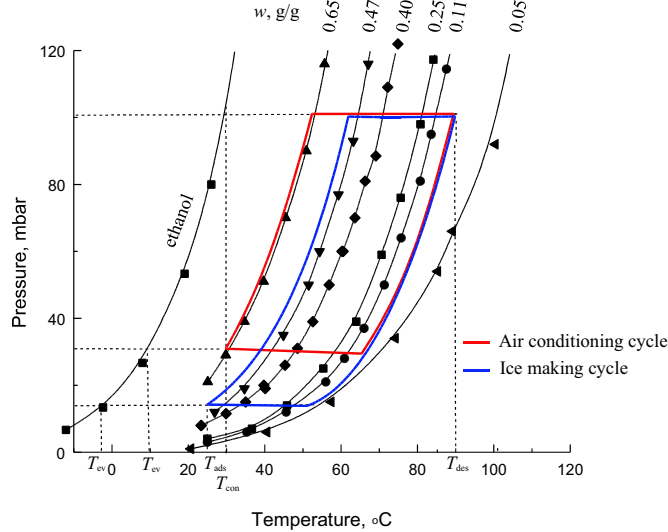


Figure 3: Isosters of ethanol sorption on LiBr/SiO₂(Gr646) composite.

The COP of a basic cooling cycle is the ratio of the vaporization heat Q_{ev} to the heat supplied to the adsorbent during the isosteric heating 1-2 (Q_{ish}) and desorption 2-3 (Q_{des}) (see Figure 1). The contribution of the sensible heat of liquid ethanol flowing from the condenser to the evaporator is neglected.

$$COP = \frac{Q_{ev}}{(Q_{ish} + Q_{des})} \quad (8)$$

Taking into account that

$$Q_{ev} = \Delta L \Delta w; \quad Q_{ish} = C_p(w_{max})(T_2 - T_{ads}); \\ Q_{des} = \Delta H \Delta w + C_p(w)(T_{con} - T_2) \quad (9)$$

the COP of the cycle can be written as:

$$COP = \frac{\Delta L \Delta w}{\Delta H \Delta w + C_p(w_{max})(T_2 - T_{ads}) + C_p(w)(T_{con} - T_2)}$$

(10), where ΔL is the latent heat of ethanol and ΔH is the desorption heat. The obtained COP = 0.66 and 0.61 for air conditioning and ice making cycles, respectively, that is comparable with COP of the best water adsorbents. Thus the design of the composite with the sorption properties adapted to the operating conditions of particular AC cycles allows the overcoming the intrinsic drawback of ethanol, namely, a low vaporization heat.

4. CONCLUSION

New ethanol sorbents “salt in mesoporous solids” were presented. Measurements of the ethanol sorption properties of these composites, under operating conditions of typical AC cycles, showed that LiCl and LiBr based composites present the net ethanol sorption ability $\Delta w = 0.3-0.56$ g/g which is higher than that of common ethanol sorbents. LiBr/SiO₂ composite is likely to be the most promising due to the maximum variation in sorption $\Delta w = 0.56$ and 0.40 g/g, per air conditioning and ice making cycles, respectively. The estimated COP is 0.66 and 0.61 for air conditioning and ice making cycles that makes the composite very attractive for adsorption cooling driven by low grade heat. Such a good performance is due to advanced sorption properties of the composite fitted to the particular cycle requirements.

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