Adsorption of ethanol onto phenol resin based adsorbents for developing next generation cooling systems

Ibrahim I. El-Sharkawy^{1,3,5}, Kutub Uddin^{2,5}, Takahiko Miyazaki^{1,5}, Bidyut Baran Saha^{2,5,*}, Shigeru Koyama^{1,2,5}, Hyun-Sig Kil², Seong-Ho Yoon^{2,4}, Jin Miyawaki^{2,4,6}

¹Faculty of Engineering Sciences, Kyushu University
Kasuga-koen 6-1, Kasuga-shi, Fukuoka 816-8580, Japan

²Interdisciplinary Graduate School of Engineering Sciences, Kyushu University
Kasuga-koen 6-1, Kasuga-shi, Fukuoka 816-8580, Japan

³Mechanical Power Engineering Department, Faculty of Engineering
Mansoura University, El-Mansoura 35516, Egypt

⁴Institute for Materials Chemistry and Engineering, Kyushu University
Kasuga-koen 6-1, Kasuga-shi, Fukuoka 816-8580, Japan

⁵International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University
744 Motoooka, Nishi-ku, Fukuoka 819-0395, Japan

⁶Research and Education Center for Advanced Energy Materials, Devices, and Systems
Kyushu University, Kasuga-koen 6-1, Kasuga-shi, Fukuoka 816-8580, Japan

*Author to whom correspondence should be addressed, E-mail:
saha.baran.bidyut.213@m.kyushu-u.ac.jp

Abstract

Adsorption characteristics of ethanol onto two promising adsorbents have been investigated for developing high performance adsorption chillers. These new adsorbents are based on spherical phenol resin treated with different mass ratios of KOH named as KOH4-PR and KOH6-PR. Experimental adsorption isotherm measurements show that the adsorption capacity of KOH4-PR/ethanol is as high as 1.43 kg kg⁻¹ whilst one kg of KOH6-PR able to adsorb nearly about 2 kg of ethanol. To the best of our knowledge, the studied adsorbents possess the highest ethanol uptake. Moreover, it is found that the KOH4-PR/ethanol pair has notably high adsorption

kinetics at the lower range of adsorption temperatures. Experimental measurements of adsorption uptake and adsorption uptake rate of the studied pairs have been analyzed and isosteric heats of adsorption have also been extracted.

Key words: adsorption cooling; adsorption isotherms; ethanol; kinetics, phenol resin

Nomenclature

D	diffusivity ($\text{m}^2 \text{s}^{-1}$)
D_{so}	pre-exponential constant ($\text{m}^2 \text{s}^{-1}$)
E	adsorption characteristics parameter (kJ Kg^{-1})
E_a	activation energy (kJ kg^{-1})
F	fractional uptake (-)
P	equilibrium pressure (kPa)
P_s	saturated pressure (kPa)
q_{st}	isosteric heat of adsorption (kJ kg^{-1})
R_g	gas constant ($\text{kJ kg}^{-1} \text{K}^{-1}$)
R_p	adsorbent particle radius (μm)
T	temperature (K)
t	time (s)
W	equilibrium adsorption uptake (kg kg^{-1})
w	instantaneous adsorption uptake (kg kg^{-1})
w_{in}	initial uptake (kg kg^{-1})

W_o maximum adsorption capacity (kg kg^{-1})

1. Introduction

According to International Institute of Refrigeration (IIR), approximately 15% of electricity produced in the world has been utilized for refrigeration and air-conditioning applications [1, 2]. The intensive use of air conditioning during summer periods, especially in hot climate regions, increases the peak of electricity demand. Adsorption cooling systems could be an effective option in reducing the peak electrical demand as they can be driven by solar energy or low-grade waste heat typically below 100 °C. Moreover, these systems are environmental friendly since they use natural refrigerants or alcohols such as water, ethanol and methanol. However, the main hindrance for spreading this technology is the poor system performance and bulkiness due to the limitation of sorption capacity of adsorbent material and the low heat transfer rate inside adsorber/desorber reactors. Therefore, characterization of innovative and promising pairs that possess high adsorption uptake and/or fast kinetics becomes a hot research topic for developing high performance adsorption cooling systems. The working pairs can be classified as pairs working at partial sub-atmospheric pressure which dominated mainly by silica gel/water, zeolite/water, activated carbon (AC)/methanol, activated carbon/ethanol and activated carbon fibers (ACFs) + ethanol or methanol. Pairs working at pressurized conditions such as AC/ammonia, AC/butane, AC /R134a and others. Adsorption characteristics of water onto two types of silica gel namely A and RD have been experimentally investigated using volumetric method [3]. The measurements have been conducted within a temperature ranges from 298 to 338 K and pressure ranges from 500 to 700 Pa. Adsorption isotherm data have been correlated

using Tóth equation and isosteric heat of adsorption has been evaluated. Aristov et al. [4] measured the water sorption equilibrium and specific heat of water onto a family of composite materials called selective water sorbents. These materials consist of a porous host matrix with open pores and a hygroscopic substance impregnated into its pores. Authors reported that composites based on CaCl_2 and LiBr as impregnated salts and different micro- and mesoporous silica gels as host matrices have adsorption capacity as high as 0.75 kg kg^{-1} . Solmuş et al. [5] measured adsorption isotherms of natural zeolite/water pair for adsorption cooling applications. The Dubinin–Astakhov adsorption isotherm model has been used to fit the experimental data. They reported that the maximum adsorption capacity of natural zeolite is about 0.12 kg kg^{-1} for adsorption temperature ranges from 40 to 150 °C and evaporation pressure from 0.87 to 7.38 kPa. El-Sharkawy et al. [6] measured adsorption uptake of ethanol onto two types of activated carbon fibers namely ACF(A-20) and ACF(A-15). It is reported that one kg of ACF (A-20) has the ability to adsorb about 0.797 kg of ethanol whilst ACF (A-15) has adsorption capacity of about 0.57 kg kg^{-1} . The study also addressed the effect of packing density of adsorbent on adsorption rate. El-Sharkawy et al. [7] studied the adsorption characteristics of ethanol onto parent and surface treated activated carbon powder. Adsorption isotherms and kinetics of ethanol onto three adsorbents namely, parent Maxsorb III, KOH-H_2 treated Maxsorb III, and H_2 treated Maxsorb III have been measured gravimetrically. It is found that the maximum adsorption uptakes of ethanol onto H_2 treated Maxsorb III, parent Maxsorb III and KOH-H_2 treated Maxsorb III are 1.23, 1.20 and 1.01 kg kg^{-1} , respectively. However, adsorption kinetics of KOH-H_2 treated Maxsorb III/ethanol pair is faster than other two studied pairs. Gordeeva and Aristov [8] presented the dynamic characteristics of methanol adsorption onto activated carbon ACM-

35.4 for enhancing the specific cooling power of the adsorption chillers. The study addressed the effects of adsorbent grain size, bed thickness and adsorption/desorption temperatures under conditions close to real adsorption chiller operations. Tamainot-Telto et al. [9] studied the adsorption characteristics of ammonia onto 26 different activated carbons. They used a thermodynamic cycle model coupled with adsorption isotherm equations to select the optimum pair based on the cooling/heating production and/or coefficient of performance. Adsorption characteristics of other pairs could be found elsewhere [10-19].

This study presents an experimental investigation of ethanol adsorption onto two newly developed phenol resin based adsorbents namely KOH4-PR and KOH6-PR. Adsorption isotherms and kinetics of the studied pairs have been measured gravimetrically using magnetic suspension adsorption measurement unit. Experimental data have been analyzed and correlated with popular adsorption isotherm and kinetic models. Adsorption uptakes of ethanol onto the studied adsorbents are found to be very promising which may lead to the design and development of high performance adsorption chillers.

2. Adsorbents

Spherical phenol resin samples, as provided BEAPS[®] series (Asahi Organic Chemicals Industry Co., Ltd.), have been used as a raw material. The BEAPS[®] which is a registered trademark of Asahi Organic Chemicals Industry Co., Ltd. has been carbonized at 600 °C for an hour in a flow of N₂ using a vertical electric furnace. For the preparation of activated carbons, potassium hydroxide (purity > 85.0%, Wako Pure Chemical Industries, Ltd.) is used as an activating agent. Weight ratio of KOH/carbonized BEAPS has set to 4 or 6. The mixture of KOH and the

carbonized BEAPS[®] has been put in a nickel container, and then the nickel container is located in a stainless steel container. After that, the stainless steel container is placed in an electric furnace vertically. The mixture is heat-treated up to 900 °C at a heating rate of 5 °C/min and maintained at 900 °C for one hour under N₂ flow. After the activation, the remaining KOH and salts formed during the heat treatment are removed by the washing with HCl solution for three times and deionized water once to adjust pH to be about 7. After washing, the collected samples are dried at 100 °C for 3 hours in an air oven and dried again at 150 °C for 12 hours in a vacuum oven. Figure 1 shows the SEM pictures of both KOH4-PR and KOH6-PR samples whilst their porosity and elemental composition are furnished in Table 1.

Laser Diffraction Particle Size Analyzer, SALD-2300, supplied by Shimadzu Corporation, Japan is used to measure the particle size distribution of adsorbent. Figures 2(a) and 2(b) show the particle size distributions for KOH4-PR and KOH6-PR samples. It can be seen from Figures 2(a) and 2(b) that both adsorbent samples have narrow particle size distributions and the normalized particle amount reaches 50% at particle diameters of 26 and 23 μm for KOH4-PR and KOH6-PR, respectively.

3. Experiments

Magnetic suspension adsorption measurement unit of type (MSB-VG-S2) supplied by BEL Japan, Inc. is used to measure adsorption isotherms and kinetics of the studied pairs. For each adsorption isotherm, the sample temperature is kept constant whilst evaporator temperature increases stepwise until reaches a relative pressure of about 0.9 or above. Experiments have been conducted at adsorption temperatures of 30, 40, 50, 60 and 70 °C. For the experimental

isotherm and kinetics measurement of both studied samples, a 47 mg of dry sample has been used. Ethanol of 99.5 % purity supplied by Osaka Kishida Chemical Co., Ltd., Japan is used as adsorbate. Experimental procedure has been described in detail by El-Sharkawy et al. [7].

4. Results and discussion

4.1 Adsorption isotherms

Figures 3(a) and 3(b) show adsorption isotherms of KOH4-PR/ethanol and KOH6-PR/ethanol pairs. It can be seen that one kg of KOH4-PR type adsorbent can adsorb 1.43 kg of ethanol whilst KOH6-PR has an adsorption capacity as high as about 2 kg kg⁻¹. Repeatability of the experimental adsorption isotherm data is also confirmed. To the best of our knowledge, adsorption uptakes of ethanol onto the two studied spherical phenol resin based adsorbents are significantly higher than adsorption capacity of ethanol onto any other adsorbent. It is worthy to mention that, adsorption capacity of KOH6-PR/ethanol pair is about 40% higher than that of KOH4-PR/ethanol pair. This is might be due to the difference between the micropore volumes of both adsorbents since KOH6-PR possesses micropore volume of 2.37 cm³ g⁻¹ whilst KOH4-PR has 1.85 cm³ g⁻¹.

The Dubinin-Raduskevich (D-R) equation (Eq. 1) is used to fit the adsorption isotherms of KOH4-PR/ethanol pair whilst the Dubinin-Astakhov (D-A) equation (Eq. 2) is found to be more suitable for KOH6-PR/ethanol pair. Numerical values of adsorption isotherm parameters are furnished in Table 2.

$$W = W_0 \exp \left[- \left(\frac{R_s T}{E} \ln \left(\frac{P_s}{P} \right) \right)^2 \right] \quad (1)$$

$$W = W_0 \exp \left[- \left(\frac{R_s T}{E} \ln \left(\frac{P_s}{P} \right) \right)^n \right] \quad (2)$$

Figures 4(a) and (b) show the adsorption/desorption isotherms for KOH4-PR/ethanol pair at adsorption temperatures of 30 and 70 °C, respectively. Similar plots for KOH6-PR/ethanol pair are shown in Figures 4(c) and 4(d). It is visible that, there is no hysteresis for the studied pairs.

4.2 Adsorption kinetics

Figures 5(a) and 5(b) show the plots of adsorption uptake and pressure versus time at adsorption temperature of 30 °C for both KOH4-PR/ethanol and KOH6-PR/ethanol pairs. As can be seen from Figure 5(a), adsorption uptake of ethanol onto KOH4-PR increases significantly at low relative pressure however, it increases marginally at relatively higher relative pressures. On the other hand, the increase in adsorption uptake of KOH6-PR/ethanol pair is almost constant within the whole experimental relative pressure span (see Fig. 5(b)). This is proven by the trend of adsorption isotherms of the studied pairs as can be seen from Figs. 3(a) and 3(b).

The diffusion time constant is evaluated using the Fickian diffusion model [20] for both pairs. As the studied adsorbents have spherical shapes (see Fig. 1), the fractional uptake can be calculated using Eq. 3 below [21],

$$F = \frac{w - w_{in}}{W - w_{in}} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left(- \frac{n^2 \pi^2 D t}{R_p^2} \right) \quad (3)$$

Equation (3) can be simplified to Eq. (4) at longer adsorption time [22],

$$F = 1 - \frac{6}{\pi^2} \exp\left(-\pi^2 \frac{D t}{R_p^2}\right) \quad (4)$$

The diffusion time constant (D/R_p^2) can be estimated by the linear plots of $\ln(1-F)$ versus time, t [22-24]. Figures 6(a) and 6(b) show the plot of $\ln(1-F)$ versus time at adsorption temperature of 30 °C and certain equilibrium pressures for KOR4-PR/ethanol and KOH6-PR/ethanol pairs. The diffusion time constant is evaluated at each adsorption step and the average values are evaluated at conditions suitable for adsorption chiller operation. Arrhenius equation (Eq. 5) is used to investigate the effect of temperature on the diffusion time constant. Arrhenius plots of the studied pairs are presented in Figure 7.

$$D = D_{so} \exp\left(\frac{-E_a}{R_g T}\right) \quad (5)$$

Therefore,

$$\ln\left(\frac{D}{R_p^2}\right) = -\ln\left(\frac{D_{so}}{R_p^2}\right) - \left(\frac{E_a}{R_g}\right)\left(\frac{1}{T}\right) \quad (6)$$

As can be seen from Fig. 7, the diffusion time constant of the KOH4-PR/ethanol pair is significantly higher than that of the KOH6-PR/ethanol pair at low adsorption temperature. Numerical values of (D_{so}/R_p^2) and activation energy of the studied pairs are furnished in Table 3. The diffusion time constants as a function of temperature for both KOH4-PR/ethanol and KOH6-PR/ethanol pairs are given by Eq. (7) and Eq. (8), respectively.

$$\ln\left(\frac{D}{R_p^2}\right) = -4.987 - \frac{898.16}{T} \quad (7)$$

$$\ln\left(\frac{D}{R_p^2}\right) = -1.349 - \frac{2133.3}{T} \quad (8)$$

As can be noticed from Eqs. (7) and (8), the diffusion time constant for KOH6-PR/ethanol pair strongly depends on the adsorption temperature.

4.3 Isosteric heat of adsorption

Isosteric heat of adsorption at constant adsorption uptake is commonly estimated using the Clausius-Clapeyron equation as given by Eq. (9) below;

$$q_{st}|_{CC} = -R_g \frac{\partial \ln P}{\partial (1/T)} \quad (9)$$

It is proven that the isosteric heat of adsorption depends on adsorption uptake and temperature. Here, the isosteric heats of adsorption of the studied pairs are estimated using Eq. (10) which was proposed by the same authors [25].

$$q_{st} = - \left(R_g T^2 \frac{\partial}{\partial T} \ln(P_s) + E \left\{ - \ln \left(\frac{W}{W_0} \right) \right\}^{1/n} \right) \quad (10)$$

Figures 8(a) and 8(b) show the plots of q_{st} versus adsorption uptake for KOH4-PR/ethanol and KOH6-PR/ethanol pairs at adsorption temperatures of 30, 50 and 70 °C. It is evident that the

isosteric heats of adsorption of the studied pairs decrease with the increase of adsorption uptake and q_{st} of KOH6-PR/ethanol pair is slightly lower than that of KOH4-PR/ethanol pair.

5. Conclusions

Adsorption equilibrium and kinetics of ethanol onto two spherical phenol resin based adsorbents treated with different mass ratios of KOH have been measured gravimetrically. Adsorption uptake of ethanol onto the studied adsorbents named KOH4-PR and KOH6-PR are as high as 1.43 kg kg^{-1} and 2 kg kg^{-1} , respectively. Results obtained from the present study show that adsorption capacity of KOH4-PR/ethanol and KOH6-PR/ethanol are higher than adsorption uptake of ethanol onto any other adsorbents available in the open literatures. Results of the adsorption kinetics show that KOH4-PR/ethanol pair has a higher kinetics compared to that of the KOH6-PR/ethanol pair at low adsorption temperatures. Isosteric heats of adsorption for both pairs have also been estimated using adsorption isotherm data. Results extracted from this study show that the newly developed adsorbents have promising adsorption characteristics with ethanol that may lead to the development of next generation of adsorption chillers.

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Fig. 8(a). Plot of q_{st} versus adsorption uptake for KOH4-PR/ethanol pair.

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KOH4-PR

KOH6-PR

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Table 1

Porosity and elemental composition of the studied adsorbents.

Adsorbent		KOH4-PR	KOH6-PR
Porosity	Total surface area [m ² g ⁻¹]	3060	2910
	External surface area [m ² g ⁻¹]	20	70
	Total pore volume [cm ³ g ⁻¹]	1.90	2.53
	Micropore volume [cm ³ g ⁻¹]	1.85	2.37
	Average pore width [nm]	1.25	1.78
Elemental composition	C [wt. %]	95.37	92.72
	H [wt. %]	0.05	0.10
	N [wt. %]	0.18	0.22
	O _{diff} [wt. %]	4.40	5.43
	O/C	0.035	0.044
	Ash [wt %]	-	1.53

Table 2

Isotherm parameters of ethanol adsorption onto KOH4-PR and KOH6-PR adsorbents.

Adsorbent	W₀ [kg kg⁻¹]	n [-]	E [kJ kg⁻¹]
KOH4-PR	1.43	2	128
KOH6-PR	1.98	1.5	90

Table 3

Parameters of Arrhenius equation of ethanol adsorption onto the KOH4-PR and KOH6-PR adsorbents.

Adsorbent	D_{so}/R_p^2 [s⁻¹]	E_a [kJ kg⁻¹]
KOH4-PR	6.83×10^{-3}	169
KOH6-PR	2.60×10^{-1}	391

Graphical Abstract

SEM picture of KOH4-PR adsorbent sample.

Profiles of adsorption uptake and pressure for KOH4-PR/ethanol pair at adsorption temperature 30 °C.