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Comparison of Adsorption of Ethyl Acetate on Activated Carbon and Molecular Sieves 5A and 13X

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Adsorption behavior of ethyl acetate is studied on a 5A molecular sieve at four temperatures. The Langmuir, Freundlich, and Langmuir−Freundlich isotherm model parameters and overall effective mass transfer coefficient using the uptake curve method are evaluated. The results of the 5A molecular sieve are compared with those of the 13X molecular sieve and activated carbon reported in the literature. Results show that the isotherms are of Brunauer type I and are well-fitted by the Langmuir isotherm for all the adsorbents. 13X shows adsorption capacity between activated carbon and 5A, where 5A shows the minimum. Enthalpy of adsorption and overall effective mass transfer coefficient are of the order of 10^5 J·kg^{-1} and 10^{-4} s^{-1}, respectively.

Introduction

Effective design of an adsorber requires quantitative information on equilibrium adsorption capacity, type of adsorption isotherm model suitably representing the system, isotherm model parameters, enthalpy of adsorption, and overall effective mass transfer coefficient.[1] Ethyl acetate, an important solvent used in petrochemical and polymer industries, is one of the toxic volatile organic compounds (VOCs).[2,3] Toxicity of ethyl acetate is not so alarming, but the removal/recovery of a mixture of toxic compounds containing ethyl acetate by adsorption needs the above quantitative information on ethyl acetate.[1] A few available reported literature provide information on the adsorptive capacity of fibrous carbon cloth and crushed activated carbon for ethyl acetate from inert gas[4] and from aqueous solution.[5] Gales et al.[6] studied hysteresis in the cyclic adsorption of ethyl acetate on activated carbon. Gales et al.[7] experimentally investigated the possibility of recovering ethyl acetate along with ethanol and acetone from a nitrogen stream. Patil et al.[8] reported the equilibrium adsorption capacity of activated carbon for ethyl acetate. But very little information is available on the removal or recovery of ethyl acetate from air using molecular sieves. We have already reported adsorption behavior of ethyl acetate on molecular sieve 13X based on equilibrium adsorption capacity, suitable isotherm model, enthalpy of adsorption, and overall effective mass transfer coefficient.[1] The 5A molecular sieve has comparable pore size (5 Å) with the molecular size of ethyl acetate (4.95 Å); therefore, it is important also to study the adsorption behavior of ethyl acetate on 5A molecular sieve. This paper discusses the adsorption behavior of ethyl acetate on molecular sieve 5A based on equilibrium adsorption capacity, suitable isotherm model, enthalpy of adsorption, and overall effective mass transfer coefficient; compares the results with our reported values for 13X and activated carbon; and finally analyzes the performances of the adsorbents toward adsorption of ethyl acetate. Thus the present study effectively summarizes the important properties related to adsorption of ethyl acetate on different adsorbents that include mesoporous activated carbon, molecular sieve 13X having pore diameter much greater than molecular size of ethyl acetate, and molecular sieve 5A having pore size comparable with the molecular size of ethyl acetate.

Table 1. Physical Properties of Adsorbents Supplied by E-merck India

	adsorbents		
parameters	5A (granular)	13X (granular)	activated carbon (granular)
average particle diameter/mm	1.58	1.58	1.50
pore diameter/Å	5	10	10 to 50
particle bulk density/kg·m^{-3}	650	600	500
BET surface area/m^2·g^{-1}	960	1000	1200
macroporosity	0.35	0.37	0.38

Experimental Studies

Experimental Setup. The adsorption isotherm setup (Figure 1) consisted of a U-tube containing a known mass (approximately 1 g) of preheated (at 300 °C overnight and thereafter cooled) adsorbent (the physical properties are given in Table 1); a constant temperature water bath; the air pretreatment column packed with glass wool, activated alumina, and silica gel; saturator(s) filled with ethyl acetate (99 % extra pure); and knockout pot(s) to avoid any entrainment of ethyl acetate droplets with air. The Knockout pot is an empty vessel, placed after the saturators, through which ethyl acetate laden air is passed so that any droplets of ethyl acetate is separated out from the airstream.

Experimental Procedure. Compressed air from the compressor was passed through the air-regulating valve to supply air without fluctuations to the bottom of the pretreatment column. This column reduces moisture content, carbon dioxide content, and particulates when air is passed through it. The pretreated air was then passed through saturator(s) and knockout pot(s) successively. Metered amounts of air (through calibrated rotameter) and the ethyl acetate mixture were then sent through the U-tube. The concentration of ethyl acetate was varied by maintaining different flow rates as well as different levels of ethyl acetate in the saturator(s). The inlet concentration of ethyl

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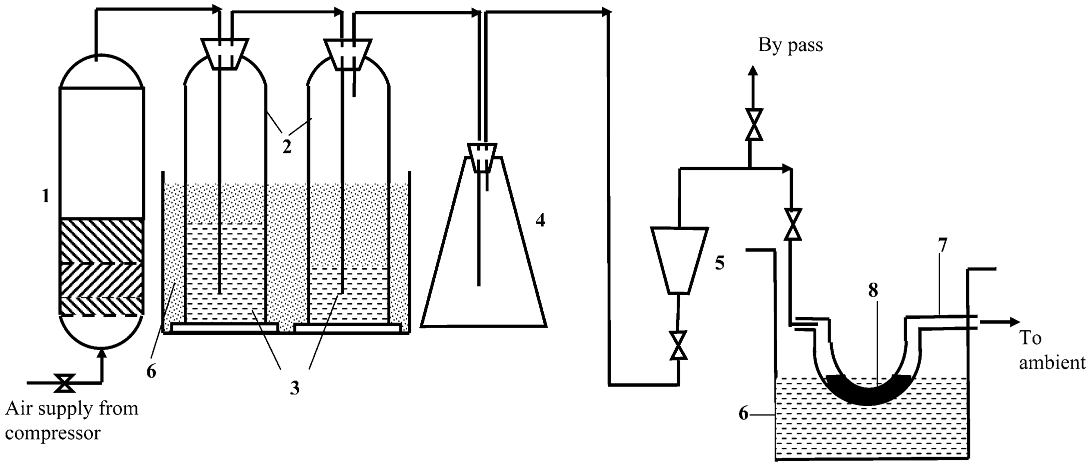

Figure 1. Experimental setup for isotherm study: 1, dehydration column packed with glass wool, activated alumina, and silica gel; 2, saturator; 3, ethyl acetate level; 4, knockout pot; 5, flow meter; 6, constant temperature water bath; 7, U-tube filled with adsorbent; 8, adsorbent.

acetate vapor in air was measured several times, using gas chromatography at the start of the experiment and at the end of the experiment. The average concentration was taken as inlet concentration. Mass change of the U-tube containing molecular sieve was measured after regular time intervals using an electronic Metler balance. The experiment was continued until there was no change in mass of the U-tube with molecular sieve (i.e., till equilibrium). The possibility of experimental error (if any) might be associated with the measurement of mass of the sample by electronic balance and concentration of the adsorbate by the gas chromatograph. Utmost care was taken in these two cases. However, at the initial stages of the experiments, a few experiments were repeated to check the reproducibility of the data**.** Sincere effort was taken to make the experimental data as accurate as possible.

Data Analysis. The concentration of ethyl acetate vapor in the inlet stream was measured with the help of gas chromatography (GC) with the AT 1000 Poropack Q column and a flame ionization detector (FID). The concentration of ethyl acetate was calculated using the following two steps such as calculation of ethyl acetate concentration in the vapor from ethyl acetate bottle using Antoine equation and calculation of ethyl acetate concentration in the syringe after dilution (i.e., the ethyl acetate final concentration). The estimated value of experimental uncertainty in measuring the concentration was ± 3.67 %. Further details are discussed elsewhere.[1,8]

Theoretical Analysis

Adsorption Isotherm. The Freundlich, Langmuir, and Langmuir−Freundlich isotherm equations are expressed by eqs 1 to 3, respectively:

$$q^* = Kc^n \tag{1}$$

$$\frac{q^*}{q_s} = \frac{bc}{1 + bc} \tag{2}$$

$$\frac{q^*}{q_s} = \frac{bc^n}{1 + bc^n} \tag{3}$$

where q^* is the amount adsorbed at equilibrium; q_s is the saturation capacity of adsorbent; K is the Freundlich constant; b is the Langmuir constant; c is the concentration of ethyl acetate in air; and n is the Freundlich exponent.

In the present study, all the three isotherm models are used to fit the experimental equilibrium data of ethyl acetate on 5A molecular sieve to investigate the suitability of the isotherm models for the adsorbate−adsorbent system of study. The experimental equilibrium data are fitted with different isotherm models, and the obtained model parameters K, b, q_s, and n are compared with those obtained for 13X molecular sieve and activated carbon.

Temperature dependency of b is calculated by the Van't Hoff equation:[9]

$$b = b_0 e^{-\Delta_{\text{abs}}H/RT} \tag{4}$$

where b_0 is the preexponential factor in the van't Hoff equation; $\Delta_{\text{abs}}H$ is the enthalpy of adsorption; R is the universal gas constant, and T is the temperature.

Enthalpy of Adsorption. The Clausius−Clapeyron equation[9] after integrating for constant adsorbed phase concentration, q (while $\Delta_{\text{abs}}H_s$ is independent of temperature) and converting pressure in terms of fluid phase concentration of adsorbate takes the form

$$\ln c = \text{constant} - \Delta_{\text{abs}}H_s/RT \tag{5}$$

where $\Delta_{\text{abs}}H_s$ is the isosteric enthalpy of adsorption. Enthalpy of adsorption is calculated by two ways. The first one is by the van't Hoff equation (eq 4) using the values of Langmuir constant obtained by the Langmuir model. The other one, the isosteric enthalpy of adsorption, is calculated by eq 5 using the plot of ln c versus $1/T$ for constant q^*. The results obtained by the two methods are compared.

Mass Transfer Coefficient. For most particle shapes, representation as an equivalent sphere is an acceptable approximation, and transport may therefore be described by a diffusion equation, written in spherical coordinate with the assumption of constant diffusivity as[9]

$$\frac{\partial q}{\partial t} = D_c\left(\frac{\partial^2 q}{\partial r^2} + \frac{2}{r}\frac{\partial q}{\partial r}\right) \tag{6}$$

The solution of this equation with suitable initial and boundary conditions[9] for the uptake curve is given by the familiar expression:

$$\frac{q_t}{q^*_\alpha} = 1 - \frac{6}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp\left(-\frac{n^2\pi^2 D_c t}{R_p^{\,2}}\right) \tag{7}$$

where q_t is the amount adsorbed at any time t; q^*_α is the amount

Table 2. Equilibrium Capacities for Ethyl Acetate in Activated Carbon and Molecular Sieves Q Flow Rate

				q^*/kg·kg^{-1}								
	c/kg·m^{-3}			30 °C	35 °C		45 °C			55 °C		
$10^6\,Q$/m^3·s^{-1}	highest value	lowest value	average value	AC	5A	13X	AC	5A	13X	AC	5A	13X
1.49	0.028	0.026	0.027	0.41	0.181	0.238	0.3	0.121	0.17	0.24	0.10	0.135
1.49	0.049	0.0455	0.047	0.55	0.24	0.294	0.42	0.18	0.251	0.356	0.155	0.218
6.5	0.074	0.0715	0.073		0.27	0.345		0.21	0.285		0.19	0.274
0.86	0.13	0.11	0.12		0.29	0.416		0.24	0.336		0.22	0.324
4.0	0.29	0.26	0.27	0.66	0.33	0.508	0.525	0.291	0.387	0.43	0.265	0.35
1.49	0.44	0.42	0.43		0.352	0.55		0.324	0.415		0.285	0.36
6.5	0.58	0.55	0.56	0.7	0.363	0.583	0.553	0.343	0.435	0.456	0.288	0.376
4.0	0.615	0.585	0.60	0.705	0.366	0.584	0.555	0.345	0.44	0.457	0.291	0.377

adsorbed at infinite time; D_c is the intracrystalline diffusivity; and R_p is the particle radius.

Equation 7 can be used to find the diffusional time constant, D_c/R_p^2 [9] where q_t/q_α^* is the fractional approach to equilibrium. This expression (eq 7) converges rapidly in the long time region since the higher terms of the summation become vanishingly small. For fractional uptakes greater than 70 %, only the first term may be retained to obtain[9,10]

$$1 - \frac{q_t}{q_\alpha^*} \approx \frac{6}{\pi^2} \exp\left(-\frac{\pi^2 D_c t}{R_p^2}\right) \tag{8}$$

In the long time region, a plot of $\ln(1 - q_t/q_\alpha^*)$ versus t should be linear with slope $-\pi^2(D_c/R_p^2)$ and intercept $\ln(6/\pi^2)$. Such a plot provides, in principle, a simple method of both checking the conformity of an experimental uptake curve with the diffusion equation and determining the diffusional time constant. Overall mass transfer coefficient in linear driving force rate equation is expressed as $k = 15(D_c/R_p^2)$, where k is overall mass transfer coefficient. The validity of this approximation has been confirmed for many different initial and boundary conditions.[10]

Results and Discussion

Adsorption Isotherm. Isotherm experiments were performed for several concentration levels of ethyl acetate on molecular sieve 5A at four different temperatures; 35 °C, 40 °C, 45 °C, and 55 °C. Four isotherms, Brunauer type I, at the four different temperatures of the experiment are presented in Figure 2. It can be observed from the figure that with anincrease in temperature the equilibrium capacity decreases. This establishes the fact that higher temperature does not favor adsorption. Equilibrium capacities for ethyl acetate on 5A, 13X, and activated carbon along with operating conditions for the experiments are presented in Table 2. Figure 3 is a sample plot indicating the different isotherm models' prediction of the experimental data for ethyl acetate adsorption on 5A. It is obvious from the figure that while both Langmuir and Langmuir−Freundlich models show very good agreement with the experimental data, the Freundlich model shows a large deviation. The detailed statistical analysis in the form of average absolute deviations (AAD) for the 5A molecular sieve is presented in Table 3. To determine isotherm constants by fitting experimental equilibrium data into said isotherm models, eight data points are used. The statistical analysis also shows that standard deviation is the least in case of the Langmuir model, lower in case of the Langmuir−Freundlich model, and much higher in case of the Freundlich model at all different temperatures. Similar observations regarding temperature dependency and model fitting were reported by the present authors in cases of ethyl acetate adsorption on activated carbon[8] and 13X.[1] Different isotherm model parameters for ethyl acetate adsorption on 5A, 13X, and activated carbon are presented in Table 4. It can be observed from the table that the saturation capacity decreases with increase in temperature for 5A and 13X molecular sieves and activated carbon for all three models. It is also observed from the table that the Freundlich constant, K, and the Langmuir constant, b, decrease with an increase in temperature for all adsorbents. The decrease in values of K and b with an increase in temperature implies the exothermicity of the adsorption process. The

Table 3. Statistical Analysis (in terms of AAD[a]) of Fitting of Experimental Isotherm Data to Different Isotherm Models for Ethyl Acetate Adsorption in E-merck 5A Molecular Sieve

model	35 °C	40 °C	45 °C	55 °C
Freundlich	0.0087	0.0091	0.0083	0.0113
Langmuir	0.0037	0.0047	0.0034	0.003
Langmuir−Freundlich	0.0044	0.0081	0.0037	0.0036

[a] AAD, average absolute deviation.

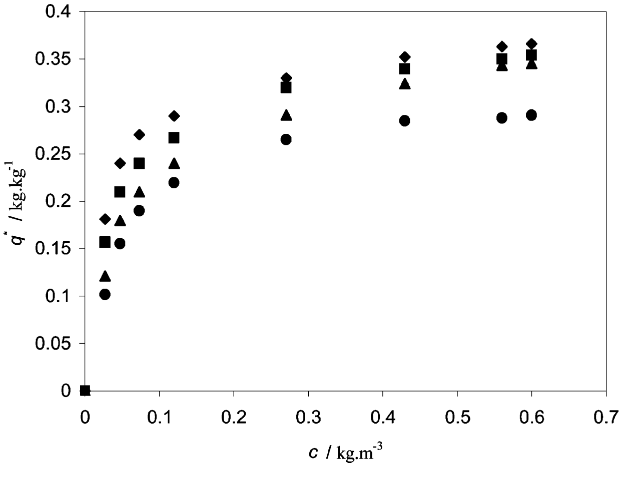

Figure 2. Ethyl acetate adsorption isotherms at different temperatures on 5A molecular sieve: ◆, 35 °C; ■, 40 °C; ▲, 45 °C; ●, 55 °C.

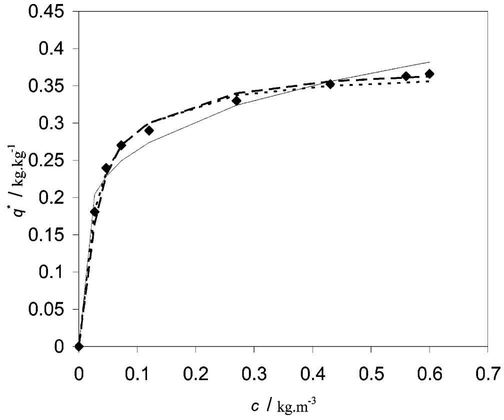

Figure 3. Comparison of different isotherm models' predictions with experimental equilibrium data of ethyl acetate adsorption on 5A molecular sieves at 35°C: ◆, experimental; —, Freundlich; - - -, Langmuir.

Table 4. Model Parameters for Ethyl Acetate Adsorption on Activated Carbon and Molecular Sieves

		30 °C	35 °C		45 °C			55 °C		
isotherm model	parameter	AC	5A	13X	AC	5A	13X	AC	5A	13X
Langmuir	q_s/kg·kg^{-1}	0.73	0.372	0.587	0.584	0.364	0.467	0.487	0.326	0.440
	b/m^3·kg^{-1}	50.92	35.70	22.8352	41.99	18.87	21.8729	39.58	17.50	18.6258
Freundlich	K	0.78	0.423	0.6893	0.634	0.42	0.5104	0.52	0.37	0.4422
	n	0.146	0.20	0.2745	0.169	0.30	0.2402	0.171	0.30	0.2241
Langmuir−Freundlich	q_s/kg·kg^{-1}		0.372	0.587		0.364	0.467		0.326	0.440
	b/m^3·kg^{-1}		58.58	29.1306		22.86	19.9869		13.57	11.9816
	n		1.19	1.0933		1.08	0.9650		0.90	0.8299

Table 5. Enthalpies of Adsorption for Ethyl Acetate on Activated Carbon and Molecular Sieves

system studied	10^{-5} ($-\Delta_{abs}H$)/J·kg^{-1} Van't Hoff equation (Langmuir model)
ethyl acetate−activated carbon	0.97
ethyl acetate−5A molecular sieve	3.21
ethyl acetate−13X molecular sieve	1.32

decrease in saturation capacity with an increase in temperature (this is in contrast to the results obtained by Gales et al.[6] for ethyl acetate adsorption on activated carbon) is observed. This contradiction is explained below.

In the original Langmuir formulation, the saturation limit was assumed to coincide with saturation of a fixed number of identical surface sites and as such it should be independent of temperature. In fact a modest decrease in saturation capacity with temperature is generally observed and is indeed to be expected, if the saturation limit corresponds with filling of the micropore volume, rather than with the saturation of a set of surface sites.[1] Figure 4 compares the adsorption isotherms obtained experimentally at 45 °C for 5A, 13X, and activated carbon. The figure indicates all as Brunauer type I. It can be observed from the figure that the adsorption capacity of ethyl acetate in activated carbon and 13X molecular sieve is higher than 5A molecular sieve at all concentrations and at all temperatures. This may be attributed to the fact that the comparable pore size of 5A molecular sieves with the molecular diameter of ethyl acetate provides steric hindrance to the movement of the chain of ethyl acetate into pores of 5A leading to lesser adsorption capacity. The same phenomenon is not true for 13X. The pore size in 13X is comparatively larger, allowing more layers of ethyl acetate adsorption onto 13X as compared to 5A. In case of activated carbon, pore size is not an issue because of its wide pore size distribution. Also the surface of activated carbon is basically nonpolar (although a slight polarity may arise from surface oxidation). It tends to be more organophilic[9] as compared to molecular sieves. Therefore, the saturation capacity of ethyl acetate adsorption in case of activated carbon is more than both the molecular sieves under study. The saturation capacity of ethyl acetate adsorption on activated carbon at 303 K in present study is 0.73 kg·kg^{-1}. Gales et al.[6] in their adsorption study on BASF activated carbon reported the saturation capacity in terms of adsorbed phase concentration at saturation limit. According to them, the adsorbed phase concentration at saturation limit is 0.326 kg·kg^{-1} in the temperature range of 288 K to 353 K. The adsorptive capacities of the fibrous carbon cloth and crushed activated carbon for ethyl acetate from inert gas were 0.223 kg·kg^{-1} and 0.175 kg·kg^{-1}, respectively.[4] Also the adsorptive capacity of the activated carbon for ethyl acetate from aqueous solution was 0.15 kg·kg^{-1}.[5] The above comparisons show that the present results are somewhat closer to the results presented by Gales et al.[6] The existing differences may be attributed to the type and properties of the activated carbon used from the different sources.

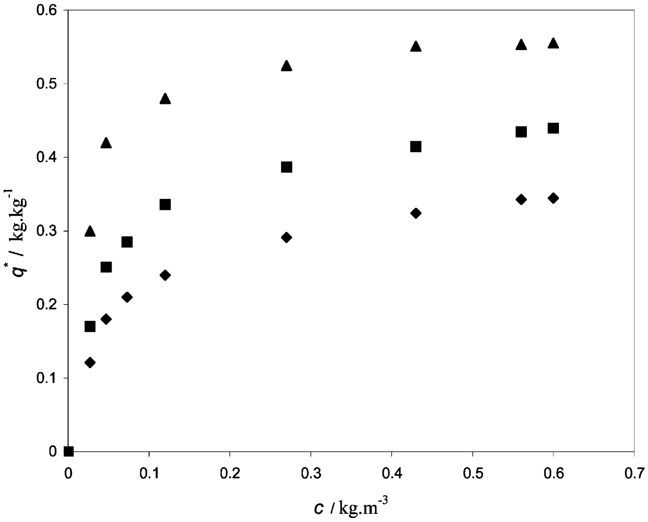

Figure 4. Comparison of adsorption isotherms of ethyl acetate in activated carbon, 5A, and 13X molecular sieves at 45 °C: ◆, 5A; ■, 13X; ▲, activated carbon.

Enthalpy of Adsorption. Enthalpy of adsorption values of ethyl acetate in 5A, 13X, and activated carbon are calculated from the Langmuir constant values using van't Hoff equation.[9] Table 5 reflects that enthalpies of adsorption values in all the three adsorbents are of the order of 10^5 J·kg^{-1}, of which 5A shows the maximum of the three. Isosteric enthalpies of adsorption, calculated by interpolating adsorption isotherms at four different values of equilibrium adsorption capacity, in-

Table 6. Overall Effective Mass Transfer Coefficients in Activated Carbon and Molecular Sieves

				10^4 k/s^{-1}								
	c/kg·m^{-3}			30 °C	35 °C		45 °C			55 °C		
10^6 Q/m^3·s^{-1}	highest value	lowest value	average value	AC	5A	13X	AC	5A	13X	AC	5A	13X
1.49	0.028	0.026	0.027	2.20	1.27	1.47	2.98	1.82	1.19	4.73	2.38	1.90
1.49	0.049	0.0455	0.047	3.37	0.84	1.19	1.44	1.47	1.32	5.90	1.67	
6.5	0.074	0.0715	0.073		1.16	1.30		1.46	1.13		2.07	0.68
0.86	0.13	0.11	0.12		1.19	0.81		1.52	1.24		1.04	1.52
4.0	0.29	0.26	0.27	1.32	0.91	1.1	2.00	1.16	1.06	2.03	1.34	0.76
1.49	0.44	0.42	0.43		1.32	1.69		1.62	1.00		2.1	2.12
6.5	0.58	0.55	0.56	1.28	1.32	1.11	2.08	1.44	1.62	3.42	1.10	1.64
4.0	0.615	0.585	0.60	1.45	1.34	1.24	1.90	1.19	2.25	2.31	1.47	0.89

creases nonlinearly with an increase in saturation adsorption capacity (not presented here), but all the values lie in the order of 10^5 J·kg^{-1}. Detailed analysis for ethyl acetate on 13X is presented elsewhere.[1] In contrast to the ideal Langmuir model, in reality enthalpy of adsorption varies with coverage depending upon surface heterogeneity and sorbate−sorbate interaction. The commonly observed increase in enthalpy of adsorption with nonpolar sorbates with coverage (particularly on zeolites A and X) is commonly attributed to the effect of intermolecular attraction forces. But in case of strongly polar molecules, enthalpy of adsorption decreases with coverage and that depends on the number and nature of cations present in zeolites.[9] Thus, ethyl acetate being a weekly polar compound, the observed variation of isosteric enthaply of adsorption with equilibrium capacity is in line with the above discussion resembling the case of nonpolar sorbate adsorption in zeolite.[1]

Overall Effective Mass Transfer Coefficient. Table 6 shows the overall effective mass transfer coefficient for ethyl acetate adsorption in all three adsorbents including the operating conditions such as flow rate, temperature of study, and concentration of ethyl acetate. It is observed from the table that the values of k are of the order of 10^{-4} s^{-1} and lie mostly in the range of (1.0 to 2.5) × 10^{-4} s^{-1} except a few cases when k is slightly away from this range. The model used is the simplest model, and the values of k thus obtained are of the same order of magnitude with the values used for validation of adsorption breakthrough data reported in our paper.[3] Apart from the model simplicity, application of a time-dependent, very precise weight-measuring device, the assumption of insignificant additional diffusional resistance associated with the system that consists of not a single particle but a few pellets/granules where several microparticles are assembled together and the assumption of insignificant vapor phase concentration distribution in the U-tube in the direction of vapor flow could be the reasons of such inconsistency in some cases.[1] However, from the values of overall mass transfer coefficient, k, obtained by the uptake curve method, it may be concluded that k is independent of temperature, concentration, and flow rate for all the adsorbents in the range of experimental conditions studied. Further, independency of k on flow rate apparently leads to the conclusion that pore diffusion is predominant for the present conditions of study, which can possibly be confirmed through further experimentation.[1,8]

Conclusions

Adsorption behavior of ethyl acetate on molecular sieve 5A based on equilibrium adsorption capacity, suitable isotherm model, enthalpy of adsorption, and overall effective mass transfer coefficient is studied. The results are compared with our reported values for 13X and activated carbon. Ethyl acetate adsorption in molecular sieve 5A follows the Brunauer type I adsorption isotherm and is well-described by the Langmuir isotherm model. Among the three adsorbents studied, the adsorption capacity of 13X is between activated carbon and 5A. The overall effective mass transfer coefficient is not influenced by the fluid flow rate, and its value is in the order of 10^{-4} s^{-1}. Enthalpy of adsorption value is in the order of 10^5 J·kg^{-1}.

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