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ADSORPTION AND DESORPTION OF *M*-XYLENE FROM SUPERCRITICAL CARBON DIOXIDE ON ACTIVATED CARBON

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ABSTRACT

The equilibrium loadings of *m*-xylene from supercritical carbon dioxide on activated carbon are reported. The experimental data were obtained by measuring the outlet concentration of *m*-xylene eluted from a column packed with activated carbon until the effluent reached the input concentration. The Freundlich isotherm expression was found to correlate the experimental data satisfactorily. In a second step, the regeneration by supercritical carbon dioxide of activated carbon loaded with *m*-xylene was investigated. The experimental data demonstrated that the adsorptive capacities of the regenerated activated carbon for *m*-xylene after

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many cycles were close to those of the virgin carbon and remained stable. The effects of temperature, pressure, and flow rate on regeneration efficiency were also studied.

INTRODUCTION

Emissions of organic vapors from industrial and commercial sources need to be controlled because they are a source of photochemical smog and nuisance odors. Several methods, such as condensation, absorption, adsorption, contact oxidation, and incineration, are used for removal and/or recovery of organic vapors. The adsorption method in which granular activated carbon is used possesses many advantages for removal and recovery of organic vapors in low concentration. For example, pure product can be recovered for reuse at low energy costs.

In other respects, supercritical carbon dioxide possesses several special characteristics and physicochemical properties. For example, it is nonflammable; nontoxic; relatively inexpensive; has a relatively high mass-transfer rate; and has adjustable extraction power, dependent on density, for organic compounds. For all these reasons, it has proved to be an efficient solvent for regenerating activated carbon loaded with organic compounds. Adsorption under supercritical fluids has been studied by a number of researchers (1–18), notably in applications of supercritical chromatography (7) or to regenerate adsorbents by desorption with supercritical fluids. The studies described in the literature concern mainly pollutants such as toluene (4,6,8–10), benzene (5,10–13), nitro-aromatic compounds (14), heavy molecular-weight organics (15,16), ethylacetate (17), and dichlorodiphenyltrichloroethane (DDT) (18). Activated carbon was the adsorbent used in most of these studies.

To design large-scale supercritical desorption processes, it is necessary to understand in which way dynamic desorption is influenced by process variables such as mass transfer effects and equilibrium considerations. The description of adsorption equilibrium is essential in all desorption models (19–21), and the existing experimental database of supercritical adsorption equilibria is limited. Moreover, most of the data have been modeled with one of the three common adsorption isotherm models, i.e., the Langmuir, the Freundlich, and the Toth. In supercritical adsorption processes, the adsorption equilibrium is influenced by the system temperature and by the supercritical fluid density. So the variation of the parameters in isotherm models as a function of both temperature and density limits the applicability of the equations when they are used for fitting experimental data. Up to now, due partly to insufficient data, the density and temperature dependence of the isotherm parameters have not been established.

In this study, the *m*-xylene, chosen as a representative product of the volatile organic compound, was adsorbed on activated carbon. Adsorption isotherms were measured for two fixed values of the CO_2 density ($\rho = 0.62$ and 0.7 g/cm^3). The experimental data obtained were correlated with two adsorption isotherm models: the Langmuir equation and the Freundlich model. According to the previous work of Squires (22), it was concluded that the adsorption of CO_2 in the selected experimental conditions could be neglected.

Another main objective of this paper was to describe the regeneration of spent, activated carbon loaded with *m*-xylene by supercritical carbon dioxide at different operating conditions. The effects of temperature, pressure, and flow rate on regeneration efficiency were investigated.

EXPERIMENTAL PROCEDURE

m-xylene was used as the adsorbate in this study because it is frequently used as the solvent in petrochemical and polymer industries. Moreover, it is often a constituent of steam exhaust.

Products and Material

m-Xylene (98% purity) was purchased from Aldrich (St. Quentin Fallavier, France), and CO_2 (99.95% purity in volume) was supplied from KWD-Hydrogas Deutschland (Germany).

The AC 40 activated carbon evaluated in this research was obtained from supplier CECA S.A. (Paris La Défense, France). Brunauer Emmet Teller surface area and total pore volume of the granular, activated carbon sample used in this study were measured and are summarized in Table 1.

Experimental Setup

The experimental setup, based on the principles of frontal analysis chromatography, was used to determine the adsorption isotherms and desorption profiles of *m*-xylene from activated carbon by supercritical CO_2. In this technique, a step change in the concentration of the solute is imposed at the inlet of the bed (the pressure and the temperature were chosen to produce a monophasic system in accordance with literature data (23)), and the response of this bed to the step change

Table 1. Adsorbent Properties

Type	Supplier	Geometry	Total Pore Volume	Surface Area
AC 40 carbon bed	CECA	pellets 4 mm	560 mm^3/g	1300 m^2/g

is monitored to obtain a breakthrough curve. Analysis of these breakthrough curves enables the construction of the adsorption isotherm. The desorption profile of *m*-xylene from activated carbon is determined by passing pure carbon dioxide through the activated carbon and monitoring the effluent concentration until it falls below detection limits.

The schematic diagram of the experimental setup is presented in Fig. 1. Virgin activated carbon was packed in a stainless steel tube with stainless steel fresh on either side to confine the carbon in the column. The sample was dried in an oven at about 423 K. After drying, about 6 g of the prepared activated carbon were packed in the column to constitute a fixed bed. Glass beads of 3 mm diameter were also packed above and below the activated carbon packing. With these pre- and postpacking sections, a uniform flow distribution in the adsorber may be achieved according to the observations of Tan and Liou (3).

The column adsorption was immersed in an insulated constant temperature oven. The temperature was controlled with an average stability of ± 0.1°C. Purified CO_2 (99.95%) was sent into the fixed bed by a compressed-air driven, high-pressure pump (HASKEL). A secondary high-performance liquid chromatography pump (Gilson 305) was used to pump the pollutant (*m*-xylene) into the fixed bed. The effluent concentration was monitored on-line using a UV detector, and the breakthrough profile of the solute was recorded. The micro-metering valves were used to control flow and to release the pressure of supercritical CO_2 to col-

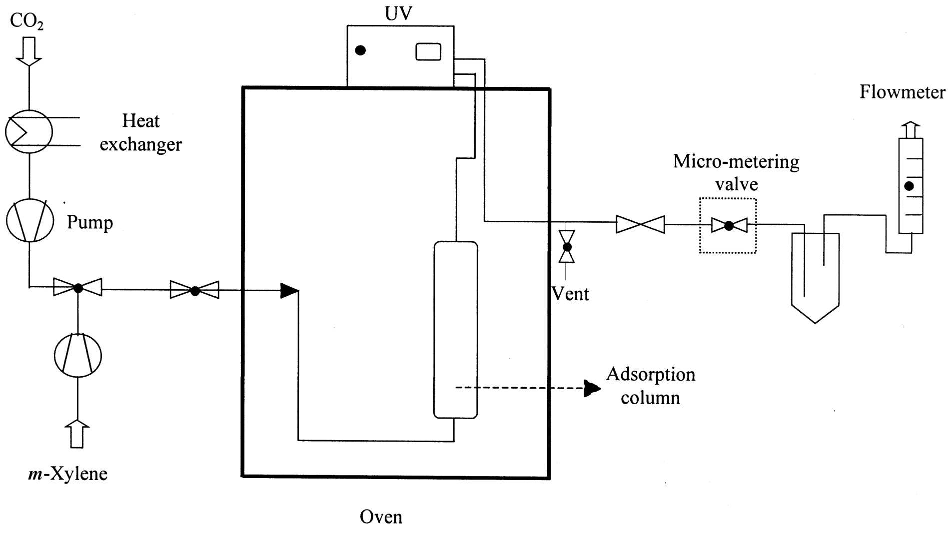

Figure 1. Schematic diagram of the experimental apparatus for adsorption/desorption measurements.

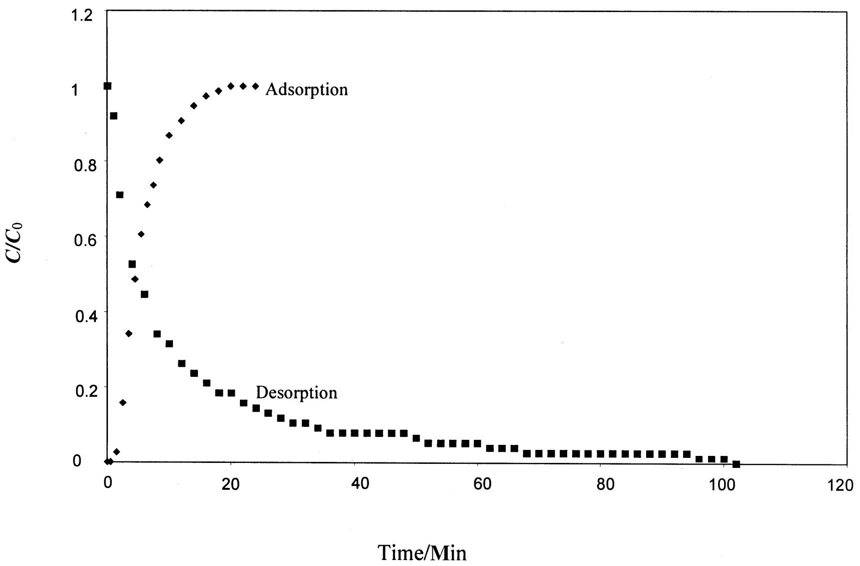

Figure 2. Example of a measured breakthrough curve. In the ordinate axis, C_0 is *m*-xylene concentration entering the column and C is the exiting concentration. In this figure, the parameters were fixed as follows: T = 323.15 K, P = 150 bar, CO_2 flow rate = 9 g/min, $C_0 = 0.492$ mol/m^{-3}.

lect the solutes under atmospheric pressure. The CO_2 flow rate was measured by a flowmeter. The plot of outlet *m*-xylene concentration versus time, also called the breakthrough curve, yielded the equilibrium data (Fig. 2).

RESULTS AND DISCUSSION

Adsorption

The adsorption isotherms at 313.15 and 323.15 K for *m*-xylene on activated carbon in supercritical carbon dioxide when the CO_2 densities were fixed at 0.622 and 0.7 g/cm^3 are presented in Fig. 3. Because the concentration of the *m*-xylene in carbon dioxide was small, the density of the supercritical mixture was regarded as that of pure carbon dioxide. The corresponding temperatures and pressures for these densities, obtained by interpolating the tabulated values provided by Angus et al. (24), are listed in Table 2.

These adsorption isotherm data indicate that equilibrium loading of *m*-xylene on activated carbon decreases when the temperature increases. This temperature-dependent behavior is analogous to that for gas- or liquid-phase adsorption.

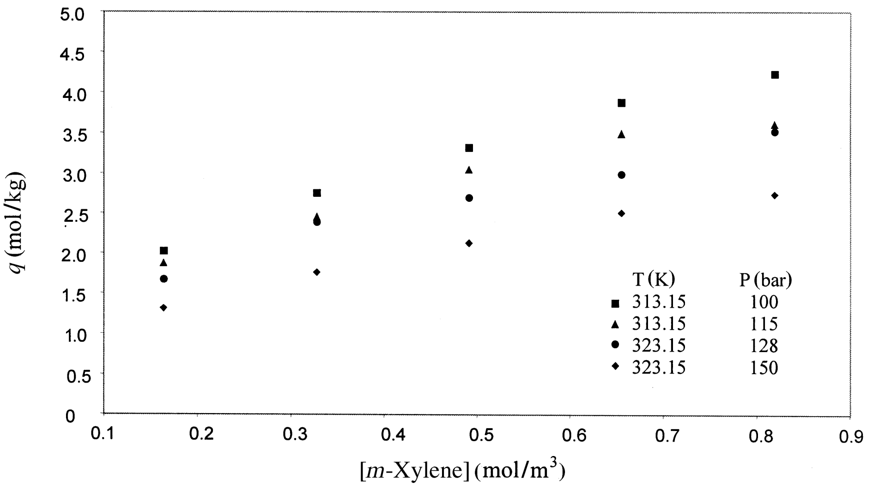

Figure 3. Adsorption isotherms of *m*-xylene on activated carbon at $\rho_{CO2} = 0.622$ g/cm^3 and 0.7 g/cm^3.

To describe mathematically the adsorption isotherms at fixed densities, some well-known relationships, the Langmuir expression (25) and the Freundlich equation (26), were tested. The Langmuir isotherm is still the simplest and most useful isotherm for describing both physical and chemical adsorption. It is a two parameter model usually written as

$$\frac{q}{q_s} = \frac{bC}{1 + bC}$$

where q is the adsorbed quantity (mole of *m*-xylene/kg of activated carbon), and C is the concentration of the adsorbate in the bulk gas phase. The two parameters q_s and b are, respectively, the saturation limit and the Langmuir constant. In this study, a nonlinear curve-fitting procedure was used to determine numerically b and q_s. The parameters obtained are summarized for each temperature in Table 3 and

Table 2. Operation Temperatures and Pressures with Constant CO_2 Densities

	$\rho = 0.622$ g/cm^{-3}	$\rho = 0.7$ g/cm^{-3}
T (K)	P (bar)	P (bar)
313.15	100	115
323.15	128	150

Table 3. Langmuir Adsorption Equilibrium Constants of *m*-Xylene

ρ_{CO_2} (g/cm^3)	T (K)	P (bar)	q_s (mol/kg)	b (m^3/mol)
0.622	313.15	100	5.95	2.831
0.622	323.15	128	4.39	3.829
0.7	313.15	115	4.97	3.294
0.7	323.15	150	3.89	2.745

the corresponding calculated adsorption isotherms are shown in Fig. 4. From this figure, it is possible to conclude that the Langmuir model is not well adapted to the description of *m*-xylene adsorption on activated carbon. For this reason, the Freundlich isotherm model was tested as an apt description of the *m*-xylene system. The Freundlich isotherm is an empirical expression used to describe the linearity of adsorption capacity as a function of adsorbate concentration when adsorption data are plotted on a log-log scale. The Freundlich equation is represented as:

$$q = KC^{1/n}$$

where K and n are empirical constants.

The two constants may be determined by plotting 1n C in the abscissa and 1n q on the ordinate and finding the slope (equal to $1/n$) and ordinate intercept (equal to 1n K) of the best-fit line through the experimental points (Fig. 5). The parameters are summarized in Table 4, and the corresponding calculated adsorption isotherms are shown in Fig. 6. From this figure, it is possible to conclude that

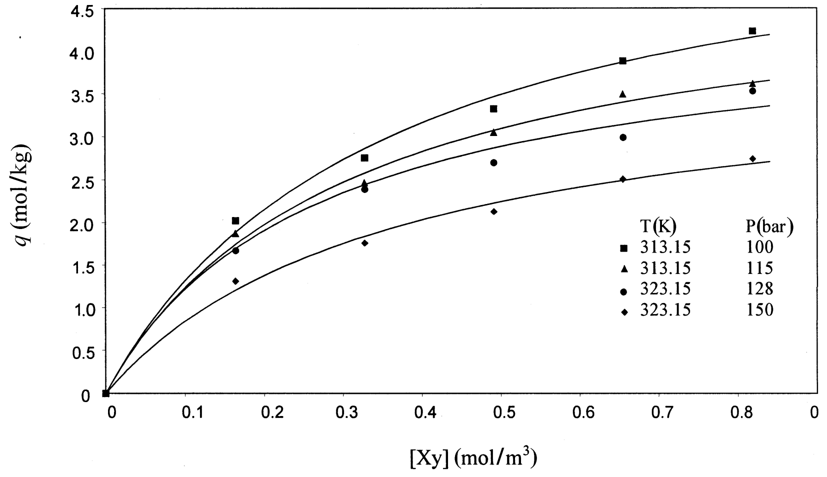

Figure 4. Correlation of the experimentally determined adsorption isotherms with the Langmuir model.

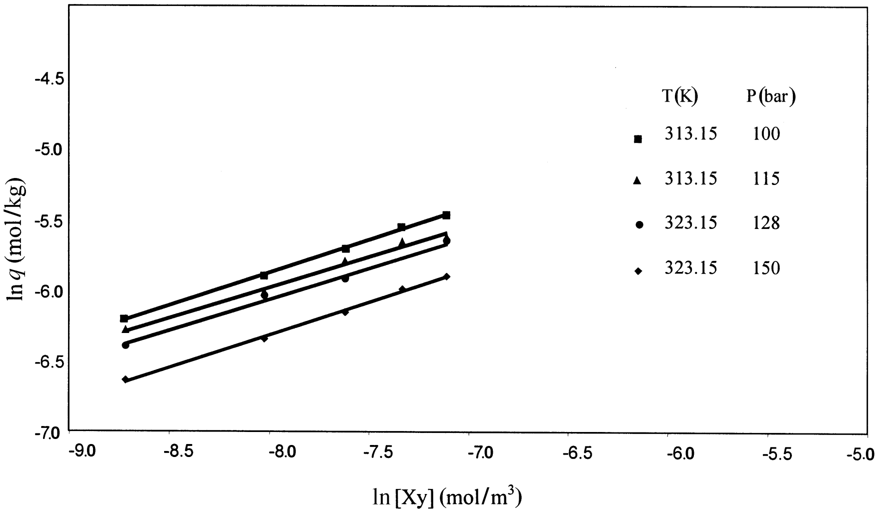

Figure 5. Plot ln q versus ln [Xy].

the Freundlich model is well adapted to the description of *m*-xylene adsorption on activated carbon.

Figures 3–6 illustrate that the adsorptive capacity decreases with increasing density when the temperature and concentration are constant. This indicates that at higher density (or higher pressure) the interaction forces between *m*-xylene and CO_2 molecules are larger than the bonding forces between *m*-xylene and the activated carbon surface.

Desorption

The conclusions related to adsorption illustrate that a higher regeneration efficiency of the activated carbon loaded with *m*-xylene should be obtained when the density is increased. For this reason, desorption experiments were conducted. The experimental results are shown in Table 5. For each experiment, the regener-

Table 4. Freudlich Adsorption Equilibrium Constants of *m*-Xylene

ρ (g/cm³)	T (K)	P (bar)	*K*	*n*
0.622	313.15	100	0.114	2.160
0.622	323.15	128	0.045	2.729
0.7	313.15	115	0.081	2.308
0.7	323.15	150	0.074	2.149

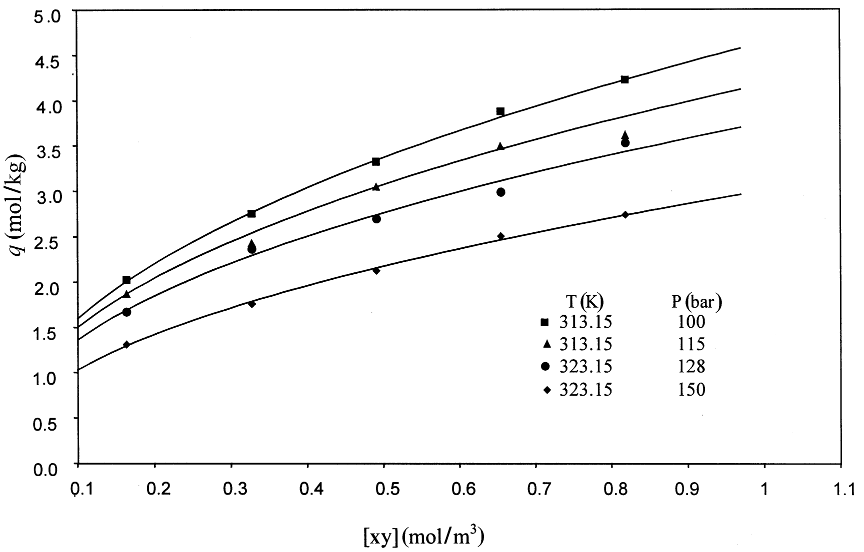

Figure 6. Correlation of the experimentally determined adsorption isotherms with the Freundlich model.

Table 5. Output of Desorption for Each Experiment

Experiment 1*		Regeneration Efficiency (%)	Experiment 2*		Regeneration Efficiency (%)
[Xy] (mol/m^3)	q_{ads} (mol/kg^1)		[Xy] (mol/m^3)	q_{ads} (mol/kg^1)	
0.164	1.671	96	0.164	1.308	94
0.327	2.389	90	0.327	1.756	93
0.491	2.698	95	0.491	2.126	95
0.655	2.989	94	0.655	2.506	96
0.818	3.525	91	0.818	2.740	91

* 323.15K, 128 bar

* 323.15K, 150 bar

Experiment 3*		Regeneration Efficiency (%)	Experiment 4*		Regeneration Efficiency (%)
[Xy] (mol/m^3)	q_{ads} (mol/kg)		[Xy] (mol/m^3)	q_{ads} (mol/kg)	
0.164	2.024	93	0.164	1.869	94
0.327	2.753	96	0.327	2.402	95
0.491	3.322	92	0.491	3.045	91
0.655	3.879	95	0.655	3.489	93
0.818	4.228	94	0.818	3.610	95

* 313.15K, 100 bar

* 313.15K, 115 bar

ation efficiencies, defined as the fraction of the loaded amount to be desorbed at a fixed time, were determined. The values given in Table 5, which are all higher than 90%, were obtained after a period of 90 min. Indeed, after such a period, only pure CO_2 was exiting the column.

In the present study, each adsorption experiment and the subsequent desorption experiment were considered as one cycle. In Fig. 7, each cycle included an adsorption step at 313.15 K and 115 bar for 0.065 mol/m^3 of *m*-xylene solution and a desorption step at 313.15 K and 115 bar. The results illustrate that the adsorptive capacity of the regenerated activated carbon drops slightly during the first three cycles, but after the third cycle, adsorptive capacity becomes quite stable and is approximately 85% of that of virgin activated carbon. This phenomenon was also observed by Kander and Paulaitis (27) and Tan and Liou (3).

Flow Rate Effect

Because the interphase mass-transfer coefficient is a hydrodynamic property, desorption is influenced by the flow rate of the regenerating fluid. Figure 8 illustrates the importance of the interphase mass-transfer resistance during the regeneration stage. The importance of the flow rate on regeneration of activated car-

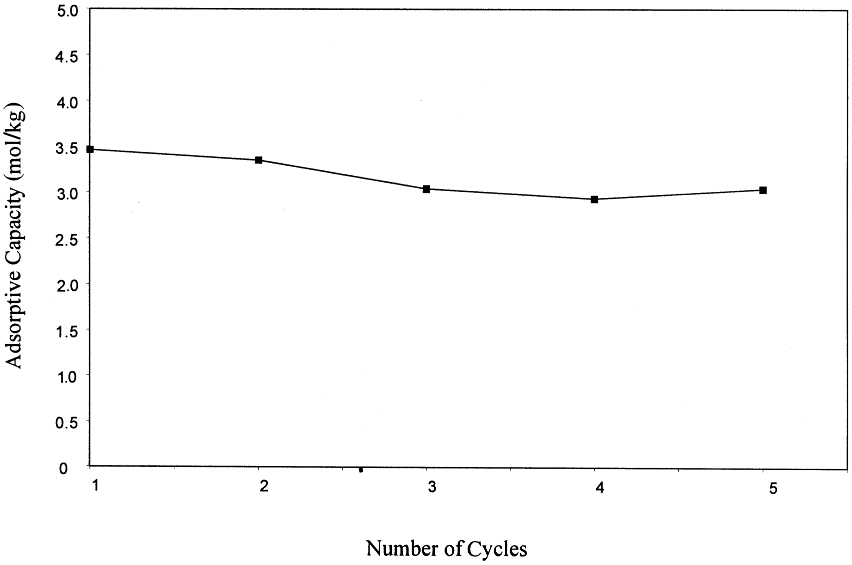

Figure 7. Adsorptive capacities of regenerated, activated carbon during the beginning cycles.

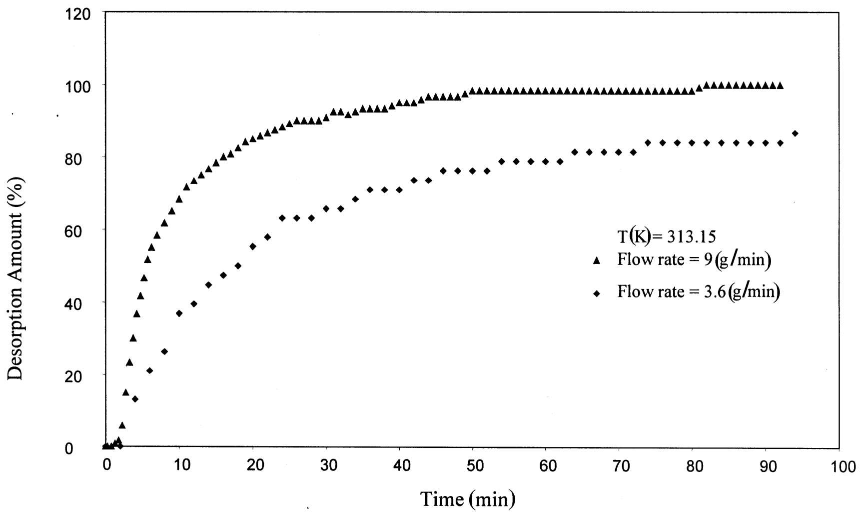

Figure 8. Flow rate effects on regeneration at 100 bar.

bon was studied at T = 313.15 K and P = 100 bar. At a low flow rate of 3.6 g/min, the desorption amount was smaller than at the higher flow rate of 9 g/min. This result suggests the existence of a film transfer resistance at low flow rate. From this study, it may be concluded that to increase the flow rate of CO_2, less desorption time is required.

Pressure Effect

Figure 9 shows the effect of pressure at 313.15 K when the CO_2 regeneration flow rate was fixed at 9 g/min. Though the regeneration period in each experiment was much more than 1.5 h, only the regeneration data at the beginning times are reported in this figure. Nevertheless, it can be seen that more than 80% regeneration for all of the runs could be achieved within the first 40 min. From Fig. 9, it can be concluded that the higher the operating pressure, the higher the regeneration efficiency. This trend was also observed by Tan and Liou (4,5). This pressure effect may be due to the increase of the CO_2 density.

Temperature Effect

The effect of temperature was studied at 100 bar when the CO_2 flow rate was fixed at 9 g/min. Figure 10 shows the effect of the temperature on desorp-

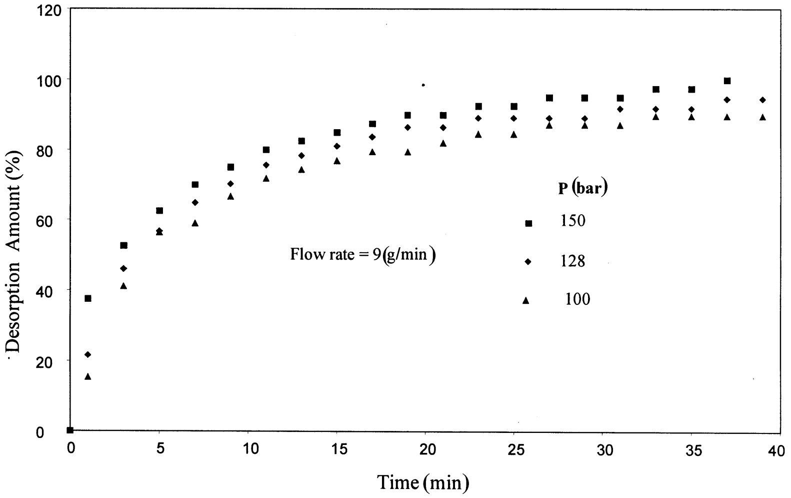

Figure 9. Pressure effect on regeneration at 313.15 K.

tion. At 100 bar, the greatest desorption was obtained at the lowest temperature. The regeneration efficiency decreases with increasing temperature, but desorption is clearly not as sensitive to temperature when temperatures are higher than 323.15 K.

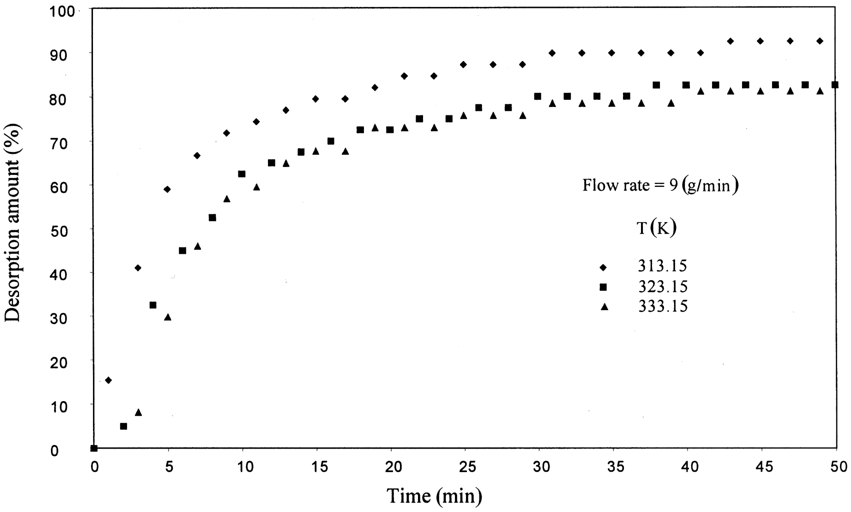

Figure 10. Temperature effect on regeneration at 100 bar.

CONCLUSION

Continuous measurements using the apparatus shown in Fig. 1 were conducted to provide the equilibrium loadings of *m*-xylene from supercritical carbon dioxide on activated carbon. The data obtained were correlated with two models described in the literature. From this study, it can be concluded that the Freundlich model leads to a precise correlation of the data. The equilibrium data showed that, when the temperature increased, the amount adsorbed was reduced. This phenomenon is analogous to that under gas- or liquid-phase operation.

In a second experimental step, regeneration of activated carbon loaded with *m*-xylene by supercritical carbon dioxide was studied. The experimental data showed that the adsorptive capacity of the regenerated activated carbon was, after many cycles, still close to that of the virgin carbon and remained stable after several regeneration cycles.

The effects of temperature, pressure, and flow rate of carbon dioxide on regeneration efficiency were examined. The higher operating pressure was more favorable for regeneration; this is probably due to the increase of CO_2 density.

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