Modelling of Gas Phase Adsorption on Activated Carbon
I. Experiment and Equilibrium Model*

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Received 1 April 2004

Dedicated to the 80th birthday of Professor Elemír Kossaczký

The course of breakthrough curve of acetone and cumene vapours mixture from a stream of nitrogen on a fixed bed of activated carbon Dezorex DB1 was measured at ambient temperature. During the process a competitive adsorption occurs, whereby acetone as the less adsorbing component was displaced by cumene. Mathematical models describing the course of equilibrium adsorption for isothermal and nonisothermal conditions were developed and used. The modified Langmuir isotherm for a two-component mixture was applied to describe the adsorption equilibrium. Experimental breakthrough curve was compared to the curves computed by equilibrium isothermal and nonisothermal models.

The breakthrough point of acetone on a fixed bed of activated carbon of a length 12 cm occurred after 55 min of contact time. According to the equilibrium nonisothermal model and isothermal model the breakthrough time of about 80 min and 95 min was calculated, respectively. The model breakthrough curves are much steeper. It can be expected that after involving the resistance against mass transfer into the model the shape of the model breakthrough curves will not be so steep.

Adsorption as a unit operation is applied not only in chemical technology but also in the environmental protection. The need for environmental and human health protection triggered development of procedures for neutralization of majority of common pollutants. In the case of volatile compounds diluted in e.g. air to a very low concentration their removal by adsorption entrapment seems to be the most efficient. A lot of studies concerning the modelling and prediction of adsorption process, adsorption equilibrium, and breakthrough curves, or mass transfer resistances were published recently. O’Connor and Mueller [1] described modelling of competitive adsorption of chlorinated volatile compounds. They predicted breakthrough curves through the use of thermodynamic properties. Tsai et al. [2] and Miyake et al. [3] investigated the breakthrough curves of organic chlorinated compound vapours on carbon adsorbents. Pentchev et al. [4] described the dynamics of nonisothermal adsorption in a packed bed of biporous zeolites. The authors measured and modelled breakthrough curves of one-component adsorption from a stream of inert gas. They compared adiabatic, near-adiabatic and nonadiabatic behaviour resulting from the fact that the operation mode might play an important role in adsorption dynamics, particularly for cases with significant heat of adsorption and high adsorptive concentration. Rivero et al. [5] presented analysis and modelling of styrene drying by means of adsorption onto activated alumina. To get experimental breakthrough curves the authors carried out fixed-bed experiments with different flow rates and using different bed lengths. They used a linear adsorption isotherm. Three extensive databases of breakthrough curves were analyzed by Wood [6] to quantify the skew of breakthrough curves. Takeuchi et al. [7] published the process of removal of single-component chlorinated hydrocarbon vapours by a new type of activated carbon. The authors measured the adsorption equilibria and breakthrough curves on a fixed bed. Martin et al. [8] described the results of study of dynamic adsorption of SO2 traces from air on activated carbons. In work [9] Subra et al. published the experimental results of adsorption of a mixture of thirteen terpenes from supercritical carbon dioxide on silanized silica. The authors continue in the investigation with a simpler system than a mixture of thirteen

solute. Sheik et al. [10] reported equilibrium and kinetic parameters for methane and nitrogen on a new type of active carbon. The pure component adsorption data are used as input data in the mathematical modelling of binary breakthrough experiments using an axial dispersion model. Tantet et al. [11] published the breakthrough study of adsorption and separation of sulfur dioxide from wet gas using hydrophobic zeolites. The Langmuir—Freundlich and extended Langmuir—Freundlich equilibrium models were used to predict equilibrium properties for the single-component and binary systems. Breakthrough curves for binary systems were calculated using kinetic and equilibrium data of single-component systems. Takeuchi et al. [12] describe experiments of fixed-bed adsorption of butanol vapours and their mixtures with high silica zeolites. For the cases of single-component adsorption, the adsorption equilibria obey the Langmuir isotherm. The Langmuir equation was found to represent the binary equilibrium data of high silica zeolites of the ZSM-5 type. The dynamics of the fixed-bed adsorption may be predicted with the use of a mass-transfer-zone method. Takeuchi et al. [13] performed fixed-bed adsorption studies on the use of activated carbon fibre for solvent vapour recovery from air. Breakthrough curves and equilibrium relationships were obtained for single- and binary-solvent vapour systems of ethyl methyl ketone, toluene, and cyclohexanone, respectively. Heinen et al. [14] investigated the hydrophobicity of activated carbons by competitive adsorption of water and toluene vapours. Chern and Chien [15] and Xiu and Li [16] presented comparison of measured and predicted one-component breakthrough curves on activated carbon adsorbent in a liquid phase.

The aim of this contribution is to compare the experimental measured breakthrough curve with the equilibrium isothermal and nonisothermal model and to verify the possibility of using the equilibrium models at low adsorptive concentrations.

THEORETICAL

Generally, modelling of multicomponent adsorption is a quite complex task. In this study, a general model of adsorption cleaning of natural gas [17] was adopted. This model was further simplified for the case of an equilibrium isothermal and equilibrium nonisothermal adsorption.

Sorption in a fixed bed of adsorbent is a complex unsteady process in which laws of hydrodynamics, mass and heat transfer in fluid and solid phase, adsorption equilibria, and heat effects accompanying the entrainment of a substance on a solid surface are valid. For the description of such a complex process some simplifying assumptions must be introduced.

In general, both solid and fluid phases are considered to be continua being in a mutual contact in the direction of flow on a constant area related to the volume unit of adsorbent. Later on, it is assumed that the fluid flows straightforwardly through the column, i.e. without axial mixing, and its axial velocity does not change from place to place. Furthermore, concentration profiles in the direction of flow are considered to be continuous functions, which are insignificantly influenced by the position of individual particles of the bed. And finally, void fraction of the bed is assumed constant with respect to time and space and the fluid occupies a constant volume in the bed (in the interparticle space), i.e. the void fraction is independent of temperature and pressure.

The last given assumption is rarely fulfilled. The first and the third assumptions introduce one disadvantage, as the mass transfer coefficients and the coefficients of resistance against fluid flow cannot be determined directly.

Equilibrium Isothermal Model

When developing an equilibrium model, it is supposed that the system is at any time and at each point in equilibrium. By neglecting the axial dispersion, material balances get the following form

\[ w \frac{\partial c_i}{\partial z} + \varepsilon \left( \frac{\partial c_i}{\partial \tau} \right)_z = - (1 - \varepsilon) \left( \frac{\partial x_i}{\partial \tau} \right)_z \rho_p \]  

(1)

Adsorption equilibrium was described by the Langmuir equation

\[ x_i = \frac{k_i c_i}{1 + b_i c_i + b_j c_j} \quad i, j = 1, 2 \]  

(2)

or by the modified Langmuir equation

\[ x_i = \frac{k_i c_i}{1 + b_i c_i} \quad i, j = 1, 2 \]  

(3)

Initial and boundary conditions for the first adsorption cycle are

\[ \tau = 0 \quad c_i = 0 \quad x_i = 0 \quad i, j = 1, 2 \]  

(4)

For the end of computation

\[ c_2 = c_{2e} \]  

(5)

Equilibrium Nonisothermal Model

Likewise in the case of equilibrium isothermal model, also when developing the equilibrium nonisothermal model the system is supposed to be in equilibrium at any time and at each point. It is also assumed that the adsorptive concentration in the bulk
of stream and in particle pores is the same and the
temperature of the bulk of stream and of a particle is
equal. Thus

\[ p_i(z, \tau) = p_i'(z, \tau) \quad T_g(z, \tau) = T_p(z, \tau) \quad i = 1, 2 \quad (6) \]

The adsorptive concentration is expressed by partial
pressure of adsorptive. Then by neglecting accumu-
lation of adsorptives in an interparticles space, the
material balance has the following form

\[
\frac{\dot{n}}{P} \left( \frac{\partial p_i}{\partial z} - \frac{p_i}{T_g} \frac{\partial T_g}{\partial z} \right) = \rho_w \frac{\partial x_j}{\partial \tau} \quad i = 1, 2 \quad (7) \]

Equilibrium relations adopt the form

\[ x_i = a_i \exp(-b_i T_g) : \]

\[
\left( \frac{T_g^{1/2} k_i \exp(r_i/T_g) p_i}{(T_g^{1/2} + k_i \exp(r_i/T_g) p_i)(T_g^{1/2} + k_j \exp(r_j/T_g) p_j)} \right) \quad i, j = 1, 2 \quad (8) \]

By neglecting heat accumulation in the gas phase,
and heat transfer resistance in the adsorbent and the
fluid phase, and between the solid and fluid phases
the following form of heat balances was developed

\[
\frac{\dot{n} R T_g}{P} \rho_c \frac{\partial T_g}{\partial z} + \rho_w c_p \frac{\partial T_g}{\partial \tau} + \frac{4}{d_i} \dot{q}_w = \rho_w \left[ (-\Delta H_i) \frac{\partial x_i}{\partial \tau} + (-\Delta H_j) \frac{\partial x_j}{\partial \tau} \right] \quad i, j = 1, 2 \quad (9) \]

Heat transfer between the adsorber wall and gas
could be expressed as follows

\[
\dot{q}_w = k_w (T_g - T_w) = \frac{d_i^2 - d_i^3}{4d_i} c_{po} \rho_o \frac{\partial T_w}{\partial \tau} + \dot{q}_{dis} \quad (10) \]

Heat losses into the surroundings are

\[
\dot{q}_{dis} = u_1 (T_g - T_o) \quad (11) \]

Initial and boundary conditions for the first ad-
sorption cycle

\[ \tau = 0 \quad p_i = 0 \quad x_i = 0 \quad \text{for} \quad i = 1, 2 \quad T_g = T_w = T_0 \]

\[ \tau > 0 \quad z = 0 \quad p_i = p_{io} \quad \text{for} \quad i = 1, 2 \quad T_g = T_{go} \quad (12) \]

and the end of computation

\[ z = z_o \quad p_2 = p_{2o} \quad (13) \]

**EXPERIMENTAL**

Acetone with purity of 99.99 mass % and cumene,
99.57 mass %, were supplied by Slovnaft, a.s. Bratisla-
va. Nitrogen was purchased from Slovnaft, a.s. Bratis-
la. Prior to the use, it was deprived of humidity
by passing through silica gel and 5A molecular sieve
beds.

Cylindrical pellets of activated carbon Dezorex
DBI used in this study were purchased from SLZ
Hnušťa – Likier. Declared parameters of activated
carbon pellets are summarized in Table 1. Prior to the
first use, activated carbon was boiled in distilled water

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average diameter, d_{ave}/mm</td>
<td>1.5</td>
</tr>
<tr>
<td>Average length, l_{ave}/mm</td>
<td>2.56</td>
</tr>
<tr>
<td>Bulk density, \rho_o/(kg m^{-3})</td>
<td>494</td>
</tr>
<tr>
<td>Void fraction, \varepsilon</td>
<td>0.36</td>
</tr>
</tbody>
</table>

**Table 1.** Properties of Activated Carbon Pellets

Fig. 1. Apparatus for the measurement of breakthrough curves
for two-component vapour mixtures: 1. manometer; 2. valve; 3. adsorber filled with silica gel; 4. adsorber filled with
molecular sieve; 5, 9, 13. needle valves; 6, 10, 14. flow meters; 7, 11. saturators; 8, 12. reservoirs of liquid;
15, 16. U-manometers; 17, 19—21. adsorbers filled with
activated carbon; 18. samples withdrawal point.
and dried in air. Then, its activation at 130°C during three hours followed.

Experimental investigation of the course of adsorption of acetone—cumene mixture from a nitrogen stream in a fixed bed of activated carbon Dezorex®DB1 was carried out at ambient temperature in the laboratory equipment, which is schematically drawn in Fig. 1. The adsorber 17 where the breakthrough curve was measured had the internal diameter of 26.3 mm and the activated carbon bed height was 12 cm.

Carrier gas from a pressure vessel passed adsorbers 3 and 4 to be deprived of humidity and the dry gas was divided into three streams. The first two streams were saturated with vapours of acetone and cumene by the passage through thermostated saturators 7 and 11, respectively. Then, the three carrier gas streams were put together and passed or bypassed through the adsorber 17. In the place 18 samples of the gas mixture were withdrawn. If the gas mixture bypassed the adsorber 17, the initial concentration of adsorbed species could be determined. Adsorbers 19—21 were used to determine the overall amount of hydrocarbons in gas. The pressure in the apparatus was measured by manometer 15. The pressure loss in adsorber 17 was measured by manometer 16. The analyses of samples were made on a gas chromatograph. Experimental conditions, at which the measurement was carried out, are summarized in Table 2.

**Table 2. Experimental Conditions**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient temperature/°C</td>
<td>20.8</td>
</tr>
<tr>
<td>Temperature in acetone saturator/°C</td>
<td>13</td>
</tr>
<tr>
<td>Temperature in cumene saturator/°C</td>
<td>33</td>
</tr>
<tr>
<td>Total pressure $P$/Pa</td>
<td>103 674</td>
</tr>
<tr>
<td>Pressure loss $\Delta P_{loss}$/Pa</td>
<td>530</td>
</tr>
<tr>
<td>Partial pressure of acetone $p_1$/Pa</td>
<td>131</td>
</tr>
<tr>
<td>Partial pressure of cumene $p_2$/Pa</td>
<td>137</td>
</tr>
<tr>
<td>Concentration of acetone $c_{1o}/(g m^{-3})$</td>
<td>6.8</td>
</tr>
<tr>
<td>Concentration of cumene $c_{2o}/(g m^{-3})$</td>
<td>3.1</td>
</tr>
<tr>
<td>Superficial velocity $w/(m s^{-1})$</td>
<td>0.138</td>
</tr>
<tr>
<td>Flow rate of gas $\dot{V}/(dm^3 min^{-1})$</td>
<td>4.489</td>
</tr>
<tr>
<td>Mass of adsorbent $m_A/g$</td>
<td>28.35</td>
</tr>
</tbody>
</table>

The following form of mass balance was adopted for the chosen experimental system

$$\dot{V}c_{i\alpha} \tau = \dot{V}c_{i\alpha} \int_0^\tau \frac{c_i}{c_{i\alpha}} (\tau) d\tau + m_{i\tau}, \ i = 1, 2 \quad (14)$$

The left-hand side of this equation represents the amount of component entering the adsorber at a time $\tau$. The first term on the right-hand side corresponds to the amount of component at the outlet of adsorber at time $\tau$, i.e. the area below the experimental breakthrough curve. The difference between the inlet and outlet amounts represents the adsorbed amount of component $m_{i\tau}$.

Comparison of experimental and calculated adsorbate amounts is summarized in Table 3. The experimental value of the adsorbate amount was obtained as the difference of adsorbent mass before and after the measurement.

The experimental breakthrough curve points for both adsorptives are presented in Fig. 2. The suitabil-

**Table 3. Comparison of Experimental and Calculated Adsorbate Amounts**

<table>
<thead>
<tr>
<th>Adsorbate amount/(g kg$^{-1}$)</th>
<th>Acetone</th>
<th>Cumene</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experiment</td>
<td>3.56</td>
<td>230.25</td>
<td>233.81</td>
</tr>
<tr>
<td>Area under the experimental curve</td>
<td>1.38</td>
<td>205.33</td>
<td>206.71</td>
</tr>
<tr>
<td>Area under the isothermal model curve</td>
<td>3.10</td>
<td>469.70</td>
<td>472.80</td>
</tr>
<tr>
<td>Equilibrium equation (eqn (2))</td>
<td>1.46</td>
<td>217.40</td>
<td>218.86</td>
</tr>
</tbody>
</table>

Fig. 2. Comparison of experimental (thick line) and calculated breakthrough curves: isothermal (thin solid line) and nonisothermal (thick dashed line) equilibrium model. Experimentally estimated relative concentration of acetone (■) and cumene (○).

**RESULTS AND DISCUSSION**

The left-hand side of this equation represents the amount of component entering the adsorber at a time $\tau$. The first term on the right-hand side corresponds to the amount of component at the outlet of adsorber at time $\tau$, i.e. the area below the experimental breakthrough curve. The difference between the inlet and outlet amounts represents the adsorbed amount of component $m_{i\tau}$.

Comparison of experimental and calculated adsorbate amounts is summarized in Table 3. The experimental value of the adsorbate amount was obtained as the difference of adsorbent mass before and after the measurement.

The experimental breakthrough curve points for both adsorptives are presented in Fig. 2. The suitabil-
ity of drawing a line through experimental points can be verified by the mass balance (14).

Thin solid line in Fig. 2 represents the breakthrough curve calculated by the equilibrium isothermal model. Adsorbate mass computed by this model should correspond to the equilibrium state, i.e. to the value of adsorbate concentration calculated from the isotherm equation. For the sake of completeness the equilibrium values were calculated using both eqns (2) and (3). The latter provided more accurate results for this concentration of adsorptives. Parameters of the used equations are given in [18].

Generally, the course of adsorption is influenced by the flow rate of the gas phase and/or the adsorptive concentration, total pressure, and temperature. In the presented study the ratio of partial pressures of adsorptives was approximately 1 : 1. During the experiment course, replacement of acetone by cumene was observed and the acetone concentration reached almost double the value of its initial concentration. This documents the existence of a competitive adsorption, in which cumene replaces adsorbed acetone.

Proposed model, a combination of mass balance and equilibrium equation was solved by the predictor—corrector method. The results of computation using the equilibrium nonisothermal model are depicted in Fig. 2 by a thick dashed line.

For the equilibrium model one can expect higher breakthrough times and higher concentrations. In terms of application of the adsorption course models, the breakthrough point of the first component is significant. Breakthrough point of acetone, which is the less adsorbing component, occurred after 55 min of running the experiment. Using the equilibrium isothermal and nonisothermal model, the calculated breakthrough point appeared at about 95 min and 80 min, respectively. The isothermal model does not consider a change of temperature caused by the release of adsorption heat. At low adsorptive concentrations one can assume a withdrawal of this heat by a passing gas. Applying the nonisothermal model, which accepted the temperature increase, an earlier breakthrough point of the replaced component – acetone was found. The computed temperature of gas at the adsorber output was max. 4°C higher than the input one without an expressive maximum. At a higher adsorptive concentration and/or smaller flow rate one could expect a higher temperature increase.

If compared with the experimental curve, the shape of the modelled breakthrough curves is much steeper, which is caused by the absence of mass transfer resistances in the model. It is obvious that after introducing the mass transfer resistance in the gas phase and in adsorbent particles also the shape of the model curve will change.

Suitability of the equation describing equilibrium substantially influences the size of the area under the model curve. If experimental points of the mixture equilibrium are at disposal, the task is easier. If, however, we go out from the equilibrium of pure components, one should select such a form of equilibrium relations which satisfies the real equilibrium state, that can be verified for example by the adsorbate amount.

Acknowledgements. This paper was created within the Project No. 1/0063/03. The authors are grateful to the Scientific Grant Agency of the Ministry of Education of the Slovak Republic and Slovak Academy of Sciences for financial support of this project.

SYMBOLS

<table>
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<th>Symbol</th>
<th>Description</th>
<th>Unit</th>
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<tbody>
<tr>
<td>(a_i)</td>
<td>equilibrium relation parameters, eqn (8)</td>
<td>kmol kg(^{-1})</td>
</tr>
<tr>
<td>(b_i)</td>
<td>equilibrium relation parameters, eqns (2) and (3)</td>
<td>m(^3^) kmol(^{-1})</td>
</tr>
<tr>
<td>(c_i)</td>
<td>equilibrium relation parameters, eqn (8)</td>
<td>m(^3^) kg m(^{-3})</td>
</tr>
<tr>
<td>(c_{i0})</td>
<td>initial adsorptive concentration</td>
<td>kmol m(^{-3}), kg m(^{-3})</td>
</tr>
<tr>
<td>(c_p)</td>
<td>specific heat of the fluid phase</td>
<td>J kg(^{-1}) K(^{-1})</td>
</tr>
<tr>
<td>(c_{pb})</td>
<td>specific heat of the solid phase</td>
<td>J kg(^{-1}) K(^{-1})</td>
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<tr>
<td>(c_{po})</td>
<td>specific heat of the adsorber jacket</td>
<td>J kg(^{-1}) K(^{-1})</td>
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<td>(d_1)</td>
<td>internal adsorber diameter</td>
<td>m</td>
</tr>
<tr>
<td>(d_2)</td>
<td>external adsorber diameter</td>
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<tr>
<td>((-\Delta H_i))</td>
<td>adsorption heat</td>
<td>J mol(^{-1})</td>
</tr>
<tr>
<td>(k_i)</td>
<td>equilibrium relation parameters, eqns (2) and (3)</td>
<td>m(^3^) kg m(^{-3})</td>
</tr>
<tr>
<td>(k_w)</td>
<td>convective heat-transfer coefficient</td>
<td>W m(^{-2}) K(^{-1})</td>
</tr>
<tr>
<td>(m_{\text{ad}})</td>
<td>adsorbate amount</td>
<td>g</td>
</tr>
<tr>
<td>(\dot{n})</td>
<td>gas molar flow rate</td>
<td>kmol s(^{-1})</td>
</tr>
<tr>
<td>(\dot{\phi})</td>
<td>gas flux, eqn (7)</td>
<td>kmol s(^{-1}) m(^{-2})</td>
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<td>gas pressure</td>
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<td>(p_i)</td>
<td>partial pressure of a component in gas phase</td>
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</tr>
<tr>
<td>(p_i')</td>
<td>partial pressure of a component in particle</td>
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<tr>
<td>(\Delta P_{\text{loss}})</td>
<td>pressure loss</td>
<td>Pa</td>
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<td>(\dot{q}_w)</td>
<td>heat flux through the internal adsorber wall</td>
<td>W m(^{-2})</td>
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<td>(\dot{q}_{\text{dis}})</td>
<td>loss of heat flux</td>
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<td>(R)</td>
<td>gas constant</td>
<td>J K(^{-1}) mol(^{-1})</td>
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<td>(r)</td>
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<td>K</td>
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<tr>
<td>(T_p)</td>
<td>particle temperature</td>
<td>K</td>
</tr>
<tr>
<td>(T_w)</td>
<td>adsorber wall temperature</td>
<td>K</td>
</tr>
<tr>
<td>(u_1)</td>
<td>overall heat-transfer coefficient</td>
<td>W m(^{-2}) K(^{-1})</td>
</tr>
<tr>
<td>(w)</td>
<td>superficial gas velocity</td>
<td>m s(^{-1})</td>
</tr>
<tr>
<td>(\dot{V})</td>
<td>gas flow rate</td>
<td>m(^3^) s(^{-1})</td>
</tr>
<tr>
<td>(x_i)</td>
<td>adsorbate concentration</td>
<td>kmol kg(^{-1})</td>
</tr>
</tbody>
</table>
\( z \) \hspace{1cm} \text{axial coordinate} \\
\( \rho \) \hspace{1cm} \text{fluid phase density} \hspace{1cm} \text{kg m}^{-3} \\
\( \rho_b \) \hspace{1cm} \text{adsorbent bulk density} \hspace{1cm} \text{kg m}^{-3} \\
\( \rho_p \) \hspace{1cm} \text{adsorbent particle density} \hspace{1cm} \text{kg m}^{-3} \\
\( \rho_o \) \hspace{1cm} \text{adsorber jacket density} \hspace{1cm} \text{kg m}^{-3} \\
\( \varepsilon \) \hspace{1cm} \text{void fraction} \\
\( \tau \) \hspace{1cm} \text{time} \hspace{1cm} \text{s} \\

Subscripts \\
i \hspace{1cm} \text{adsorptive component (acetone)} \\
j \hspace{1cm} \text{adsorptive component (cumene)} \\
1 \hspace{1cm} \text{internal adsorber surface} \\
2 \hspace{1cm} \text{external adsorber surface} \\

REFERENCES 