

Diffusion of Ethanol Vapours in Activated Carbon Particles*

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In adsorbers with a bed height equal to the diameter of activated carbon particles the course of stepwise adsorption of ethanol from a stream of air and the course of stepwise desorption of ethanol by a stream of air with a lower content of ethanol compared with adsorption or by a stream of pure air were examined. The stepwise measurements were performed at a concentration of ethanol in air ranging from 0 to 0.35 mol m^{-3} , which corresponds to the concentration of ethanol on activated carbon from 0 to 4180 mol m^{-3} . From experimentally measured data effective diffusion coefficients D_e were calculated for each adsorption and desorption step. The calculated values of D_e are ranging from 1.182×10^{-10} to $6.308 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$. These values are dependent on the adsorbate concentration. From experimental data also values of D_e were calculated for each time of the stepwise adsorption and desorption. These values were used for the determination of the dependence of D_e on the adsorbate concentration:

$$D_e = -4.7424 \times 10^{-21} q^3 + 5.6130 \times 10^{-17} q^2 - 5.9684 \times 10^{-14} q + 1.7647 \times 10^{-10}$$

By comparison of the obtained values of the effective diffusion coefficient with values calculated by models it follows that during the transport of ethanol in particles of activated carbon Supersorbon HS4 both Knudsen diffusion and surface diffusion are decisive.

Activated carbons have been widely used in gas purification and separation processes. To model adsorption processes using activated carbons, information on basic features of adsorption isotherms and pore diffusion coefficients is necessary [1, 2]. The Wicke—Kallenbach technique was applied for the measurement of diffusivity of the systems Ar—N₂ and CO₂—N₂ in a porous carbon at temperatures up to 700°C [3]. By modelling multicomponent adsorption of hydrocarbons from an aqueous solution in a fixed bed of activated carbon effective diffusion coefficients of the order of magnitude of $10^{-10} \text{ m}^2 \text{ s}^{-1}$ were obtained [4]. The mean diameter of particles used was 0.68 mm. In the investigation of adsorption of dyes from aqueous solutions in a mixed vessel a dependence of the effective diffusion coefficient on the input adsorptive concentration was found [5]. Knudsen diffusion in randomly correlated networks of constricted pores is described by Burganos and Payatakes [6]. Do and Do [7] theoretically solved the effect of micropore size distribution on the surface diffusivity in macroporous solids. Chen and Yang [8] formulated a model of surface diffusion of multilayer-adsorbed species. The model is valid for homogeneous surfaces and multilayer surface dif-

fusivity is expressed as a function of surface coverage and gas-solid interaction potential energy. The model, in principle, requires no fitted parameters. It is also capable of predicting the maximum surface diffusivities as the surface concentration is varied. Molecular dynamics simulation method was used to calculate diffusion coefficients in narrow slit-shaped micropores [9]. Borrelli *et al.* [10] modelled diffusion-limited gasification of carbons by branching pore models. Nicholson [11] used computer simulation to study the properties of molecules in pores. The profiles of the dynamic column breakthrough experiments were analyzed to derive mass transfer data as well as heat transfer rate data for the adsorption of methane, ethane, and propane in activated carbon particles [12]. A model for surface diffusion in liquid phase was developed on the basis of the absolute rate theory [13]. The influence of the shape of adsorption isotherms on the diffusion in activated carbon particles was investigated by Linders *et al.* [14]. Simultaneous diffusion and adsorption of hydrocarbons in activated carbon particles was studied in work [15]. Sundaram and Yang [16] used multicomponent diffusion in solving the kinetics of pressure swing adsorption in separating oxygen and

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nitrogen from air by molecular sieve carbon. In the investigation of adsorption of NaI on granulated activated carbon *Drazer et al.* [17] found a concentration dependence of the effective diffusion coefficient. The obtained values were ranging from $1.15 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ to $4.4 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ for 1.0×10^{-3} to 0.1 mol dm^{-3} solutions. A model describing the entrapment of chlorinated hydrocarbons from a stream of air in a granulated active carbon bed exhibiting a high specific surface area is found in paper [18]. In diffusion of chlorinated solvents in a single activated carbon particle surface diffusion is important [18]. According to authors [19] effective diffusion coefficients of benzene and methyl ethyl ketone in the presence of nitrogen are within the range 1.2×10^{-8} – $1.1 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$. With increasing concentration of adsorptive the values of the effective diffusion coefficient increase. Diffusion of hydrogen sulfide and methyl mercaptane from air onto microporous alkaline activated carbon is reported by *Chiang et al.* [20]. In measurements spent activated carbon, regenerated activated carbon, fresh activated carbon, impregnated-regenerated activated carbon, and impregnated-fresh activated carbon were used. The estimated values of effective diffusion coefficients are ranging from 2.1×10^{-12} to $6.5 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$. Binary diffusion of unequally sized molecules in zeolites was described by *Sundaram and Yang* [21]. In binary diffusivities the cross-term from irreversible thermodynamics was used. *Lordgooei et al.* [22] modelled effective diffusion coefficients of volatile organic compounds in activated carbon fibres. *Chen et al.* [23] investigated dyestuffs adsorption on pith. From film-pore diffusion modelling effective diffusivity from $5.27 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ to $8.6 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ was found. The branched pore diffusion model was applied to the single component adsorption of reactive dyes on activated carbon in a batch stirred vessel [24]. The calculated surface diffusivity of dyes is ranging from $1.15 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$ to $4.0 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$ what is an about three orders lower value than the effective diffusivity published in [23]. In spite of numerous works dealing with the transport of component in solid phase pores this problem has not been solved yet. Obviously it is due to a complicated pore structure of the solid phase and the complexity of transport phenomena taking place in the adsorbent pores.

The purpose of this study was to investigate both the mechanism of diffusion and the effective diffusivity of ethanol vapours in activated carbon particles.

THEORETICAL

Let us assume that the porous structure of adsorbent particles is arranged in such a way that the particles are isotropic in terms of mass transfer. This porous material can be then assumed as a quasihomogeneous medium, which differs from a homogeneous medium by the fact that in the solid phase pores ad-

sorption of vapours can take place. It means that besides molecular diffusion and Knudsen diffusion also diffusion in the adsorbed phase can be important. Unsteady simultaneous diffusion and adsorption, which takes place in a quasihomogeneous medium, can be described by the equation of unsteady diffusion

$$\frac{\partial c_A}{\partial \tau} = D_e \Delta c_A \quad (1)$$

in which the effective diffusion coefficient characterizes all processes participating in the mass transfer process. Eqn (1) can be solved analytically or numerically for the given shape of the adsorbent particles and for the given initial and boundary conditions. For cylindrical particles and symmetric diffusion eqn (1) can be rearranged into the following form

$$\frac{\partial c_A}{\partial \tau} = D_e \left[\frac{\partial^2 c_A}{\partial r^2} + \frac{1}{r} \frac{\partial c_A}{\partial r} + \frac{\partial^2 c_A}{\partial z^2} \right] \quad (2)$$

If these particles have a radius of r_0 and a length l , they do not contain adsorbate molecules before the adsorption process proceeds and from a certain time a constant concentration of adsorptive c_{A0} is maintained, initial and boundary conditions are as follows

$$c_A(r, z, 0) = 0; \quad c_A(r_0, z, \tau) = c_{A0}; \quad c_A(r, \pm l/2, \tau) = c_{A0}$$

$$\frac{\partial c_A(0, z, \tau)}{\partial r} = 0; \quad \frac{\partial c_A(r, 0, \tau)}{\partial z} = 0 \quad (3)$$

By solving eqn (2) for conditions (3) and by rearrangement one obtains for the adsorbed amount of component [25]

$$\begin{aligned} \gamma_\tau &= \frac{\Delta m_\tau}{\Delta m_\infty} = \frac{\Delta q_\tau}{\Delta q_\infty} = \\ &= 1 - \frac{32}{\pi^2} \sum_{n=1}^{\infty} \sum_{m=1}^{\infty} \frac{1}{\mu_n^2 (2m-1)^2} \cdot \\ &\cdot \exp \left[- \left(\frac{\mu_n^2}{r_0^2} + \frac{(2m-1)^2 \pi^2}{l^2} \right) D_e \tau \right] \quad (4) \end{aligned}$$

If the cylindrical particles are saturated by the adsorbate to an equilibrium concentration q_{A0} corresponding to the adsorptive concentration c_{A0} and from a certain time a zero concentration of the adsorptive is maintained on the surface, initial and boundary conditions can be expressed in the following way

$$c_A(r, z, 0) = c_{A0}; \quad c_A(r_0, z, \tau) = 0; \quad c_A(r, \pm l/2, \tau) = 0$$

$$\frac{\partial c_A(0, z, \tau)}{\partial r} = 0; \quad \frac{\partial c_A(r, 0, \tau)}{\partial z} = 0 \quad (5)$$

By solving eqn (2) for conditions (5) and after rearrangement one obtains for the adsorbed amount of

Table 1. Results of Calculations

Measurement	Temperature °C	c_A mol m ⁻³	q_A mol m ⁻³	$D_e \cdot 10^{10}$ m ² s ⁻¹	$D_{em} \cdot 10^{10}$ m ² s ⁻¹	$K = \frac{q_A}{c_A}$	$K' = \frac{\Delta q_A}{\Delta c_A}$
1-A1	20.8	0.0123	654	1.816	1.806	53226	53226
1-A2	20.7	0.0798	2191	2.349	2.188	27454	22766
1-A3	21.3	0.1124	2595	2.924	2.675	23098	12417
1-A4	21.6	0.3001	3870	5.705	5.104	12895	6788
1-D1	19.5	0.1124	2667*	3.316	2.770	12895	6344
1-D2	21.9	0.0123	1045*	2.384	1.980	24123	19401
1-D3	21.6	0	470*	1.588	1.322	38764	35349
2-A1	20.4	0.0125	718	1.823	1.870	57448	57448
2-A2	21.0	0.0407	1644	1.874	1.670	40404	32854
2-A3	22.5	0.1903	3390	3.853	3.304	17815	11669
2-A4	22.0	0.3396	4183	7.024	6.308	12318	5313
2-D1	21.8	0.1903	3618*	8.649	6.237	12318	5289
2-D2	21.9	0.0407	1848*	3.230	2.489	15547	10166
2-D3	21.5	0.0125	1135*	2.346	1.962	31515	24817
2-D4	22.3	0	527*	1.470	1.206	37954	34206
4-A1	21.7	0.0288	1148	1.757	1.459	39849	39849
4-A2	22.6	0.0834	2151	3.067	2.846	25796	18381
4-A3	23.2	0.1375	2824	3.617	3.197	20536	12435
4-D1	23.1	0.0834	2253	4.382	3.563	20536	10076
4-D2	22.9	0.0288	1364*	2.894	2.241	27864	20745
4-D3	21.7	0	308*	1.182	0.505	35884	33811

*The values at the end of desorption steps.

component

$$\gamma_\tau = 1 - \frac{\Delta m_\tau}{\Delta m_\infty} = 1 - \frac{\Delta q_\tau}{\Delta q_\infty} = \frac{32}{\pi^2} \sum_{n=1}^{\infty} \sum_{m=1}^{\infty} \frac{1}{\mu_n^2 (2m-1)^2} \cdot \exp \left[- \left(\frac{\mu_n^2}{r_o^2} + \frac{(2m-1)^2 \pi^2}{l^2} \right) D_e \tau \right] \quad (6)$$

EXPERIMENTAL

Ethyl alcohol containing 99.99 mass % ethanol was prepared from technical grade 96 vol. % ethanol by rectification in a packed column and by subsequent drying using CaO and molecular sieve 3A.

Activated carbon Supersorbon HS4 consisted of cylindrical particles with following characteristics: diameter 4.81 mm, length 5.24 mm, specific surface area 917 m² g⁻¹, specific volume of the transport pores determined by mercury porosimetry 0.286 cm³ g⁻¹, particle density 0.709 g cm⁻³.

Both the course of adsorption of ethanol from a stream of air and the course of desorption of ethanol by a stream of mixture of air and ethanol or pure air in an adsorber with a bed height equal to the diameter of a single adsorbent particle were examined in an apparatus described in Ref. [26]. The course of adsorption and desorption was investigated in two adsorbers by weighing, when equal adsorption times elapsed. The required concentration of adsorptive in air during the

adsorption process was obtained by evaporating the adsorptive from a free surface in tempered saturators. This concentration value was estimated from the overall adsorbed amount of ethanol in differential adsorbers and adsorbers incorporated in the stream before the outlet, through which air was released into the atmosphere, and from the overall volume of air passing through the adsorber. The result of these measurements is a set of data $\{q_{\tau i}; \tau_i\}$ for each adsorption step. Both the concentration of ethanol vapours in air during adsorption c_A and the concentration of adsorbate at the end of adsorption q_A for each step are listed in Table 1 and their dependence is depicted in Fig. 1. After the completion of adsorption, desorption of ethanol followed. The desorption was conducted either by an ethanol—air mixture or by pure air, whereby the mass was determined by weighing. Again a set of measurements $\{q_{\tau i}; \tau_i\}$ was obtained for each desorption step.

RESULTS AND DISCUSSION

For each set of adsorption data $\{q_{\tau i}; \tau_i\}$ the value of the effective diffusion coefficient D_e was calculated from eqn (4) and by employing the optimization method. The following relationship was used as the optimization function

$$F = \sum_{i=1}^n \left[(\gamma_{\tau i})_{\text{exp}} - (\gamma_{\tau i})_{\text{calc}} \right]^2 \quad (7)$$

For the calculation of effective diffusion coefficients from desorption data relation (6) was used. The re-

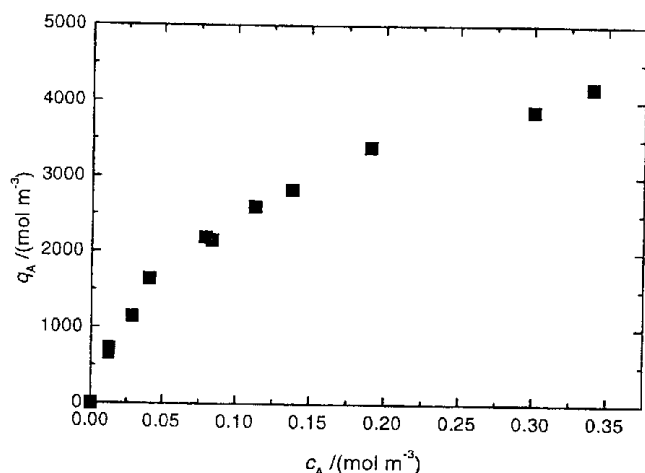


Fig. 1. Equilibrium data of the system ethanol—Supersorbon HS4 at 21.6°C.

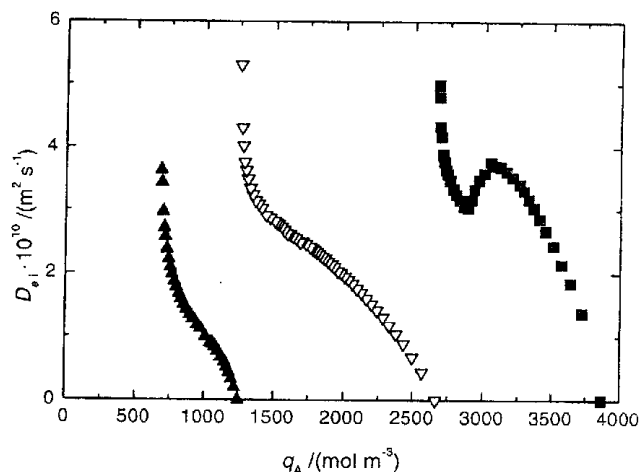


Fig. 3. Effective diffusivity D_{ei} in desorption steps 1-D1 (■), 1-D2 (▽), 1-D3 (▲).

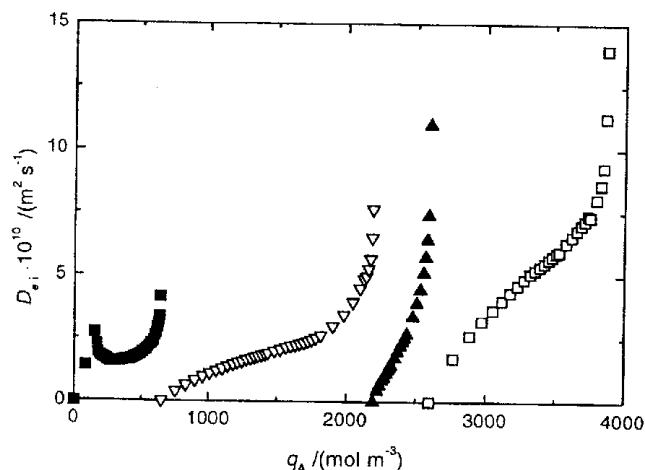


Fig. 2. Effective diffusivity D_{ei} in adsorption steps 1-A1 (■), 1-A2 (▽), 1-A3 (▲), 1-A4 (□).

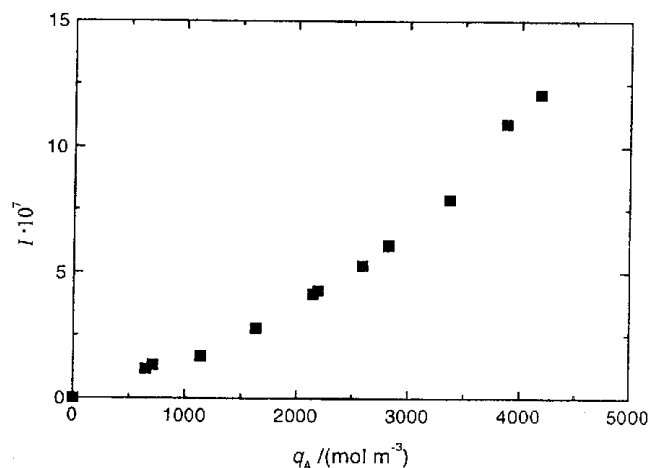


Fig. 4. The dependence of the integral on the left side of eqn (9) $I = \int_0^{q_n} D_e dq$ upon q_A for measurements 1, 2, and 4.

sults of these calculations for adsorption and desorption steps are listed in Table 1.

In addition, effective diffusion coefficients D_{ei} for each point (q_{ri} ; τ_i) were also calculated from experimental set of data $\{q_{ri}$; $\tau_i\}$. Thus a set of values of effective diffusion coefficients for each adsorption and desorption step was obtained. Calculated values of D_{ei} corresponding to a certain adsorbate concentration for measurement 1 are given in Fig. 2 and Fig. 3 for the stepwise adsorption and stepwise desorption, respectively. The mean value of the effective diffusivity D_{em} was calculated for a set of effective diffusion coefficients using the following relation

$$D_{em} = \frac{1}{q_n - q_{n-1}} \int_{q_{n-1}}^{q_n} D_{ei} dq \quad (8)$$

The calculated values of the mean effective diffusion coefficient D_{em} for each adsorption and desorp-

tion step are also given in Table 1. There are not significant differences between D_e and D_{em} and Table 1 indicates a change of D_e and D_{em} with the adsorbate concentration. This observation enabled to derive the following relationship for stepwise adsorption

$$\int_0^{q_n} D_e dq = \int_0^{q_1} D_e dq + \int_{q_1}^{q_2} D_e dq + \dots + \int_{q_{n-1}}^{q_n} D_e dq \quad (9)$$

The contributions of individual integrals can be calculated from the values D_{em} using relation (8). The dependence of the integral on the left side of eqn (9) upon q_A for measurements 1, 2, and 4 is drawn in Fig. 4. This dependence was fitted by a fourth-order polynomial and after derivation with respect to q_A the following relationship was obtained

$$D_e = -4.7424 \times 10^{-21} q^3 + 5.6130 \times 10^{-17} q^2 - 5.9684 \times 10^{-14} q + 1.7647 \times 10^{-10} \quad (10)$$

From the definition of the effective diffusion coefficient [27]

$$D_e = \frac{D''}{\beta + K} \quad (11)$$

it follows that D_e depends on an equilibrium constant which can be obtained either from the relation

$$q_A = K c_A \quad (12)$$

or from the equation

$$q_A = K' c_A + e \quad (13)$$

With regard to the nonlinear course of the equilibrium line drawn in Fig. 1 it is obvious that with increasing concentration of adsorbate the value of K or K' decreases. Hence, in accordance with eqn (11), the value of D_e increases. The transport of an adsorptive in porous media can occur by molecular diffusion, Knudsen diffusion, and surface diffusion, respectively. Supposing that the transport of a component in solid phase pores proceeds *via* molecular diffusion or Knudsen diffusion in combination with surface diffusion the effective diffusion coefficient can be expressed by the relation

$$D_e = \frac{D_{AB} + D_s K}{\beta + K} \frac{\beta}{k^2} \quad (14)$$

for molecular and surface diffusion and by the relation

$$D_e = \frac{D_K + D_s K}{\beta + K} \frac{\beta}{k^2} \quad (15)$$

for Knudsen and surface diffusion.

The coefficient of surface diffusion is dependent on the concentration of adsorbate in the following way

$$D_s = \frac{\theta}{1 - \theta} D_{s0} \quad (16)$$

Data in Fig. 1 were treated according to the Langmuir isotherm

$$\theta = \frac{q}{q_m} = \frac{bc}{1 + bc} \quad (17)$$

The calculated parameters are: $q_m = 5340 \text{ mol m}^{-3}$, $b = 9.025 \text{ m}^3 \text{ mol}^{-1}$. The diffusion coefficient of ethanol in air computed according to Fuller *et al.* [28] is $D_{AB} = 1.216 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$ at 22°C . The coefficient of Knudsen diffusion of ethanol in pores of activated carbon calculated from the relation

$$D_K = \frac{8\beta}{3S_p \rho_p} \sqrt{\frac{2RT}{\pi M_A}} \quad (18)$$

is $2.943 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$.

From the experimental data of D_e and by using eqns (14) and (16) D_{s0} was calculated by an optimization method ($D_{s0} = 4.97 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$). When

Knudsen diffusion and surface diffusion are supposed, $D_{s0} = 1.32 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$.

If values calculated by means of eqn (10) are used instead of values D_e given in Table 1, optimization yields the following values for D_{s0} : $4.55 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ and $1.09 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$ for molecular diffusion and Knudsen diffusion, respectively.

Basing on the assumption that the properties of ethanol in the adsorbed phase approach the properties of liquid ethanol at 22°C , the value of the diffusion coefficient of ethanol in liquid ethanol calculated by semiempirical equations is $1.208 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$ at 22°C .

The comparison of calculated values of the surface diffusivity D_{s0} with the values of the diffusion coefficient of liquid ethanol at 22°C enables to conclude that the transport of ethanol in activated carbon particles proceeds *via* a combination of Knudsen diffusion and surface diffusion.

SYMBOLS

b	parameter of Langmuir isotherm	$\text{m}^3 \text{ mol}^{-1}$
c_A	concentration of component A in the gas phase	mol m^{-3}
c_{A0}	concentration of component A in the inlet gas during adsorption	mol m^{-3}
D'	diffusion coefficient of component in porous medium	$\text{m}^2 \text{ s}^{-1}$
D''	equivalent diffusion coefficient	$\text{m}^2 \text{ s}^{-1}$
D_{AB}	molecular diffusivity of A in B	$\text{m}^2 \text{ s}^{-1}$
D_e	effective diffusion coefficient	$\text{m}^2 \text{ s}^{-1}$
D_{em}	effective diffusion coefficient defined by eqn (8)	$\text{m}^2 \text{ s}^{-1}$
D_K	coefficient of Knudsen diffusion	$\text{m}^2 \text{ s}^{-1}$
D_s	coefficient of surface diffusion	$\text{m}^2 \text{ s}^{-1}$
D_{s0}	coefficient of surface diffusion defined by eqn (16)	$\text{m}^2 \text{ s}^{-1}$
e	parameter in eqn (13)	mol m^{-3}
F	function defined by eqn (7)	
K	equilibrium constant defined by eqn (12)	
K'	equilibrium constant defined by eqn (13)	
l	length of activated carbon cylinders	m
M_A	molar mass of component A	kg kmol^{-1}
Δm_τ	increment of the adsorbed amount at time τ in adsorption or desorption	kg
Δm_∞	increment of the adsorbed amount at the end of adsorption or desorption	kg
q_A	adsorbate concentration	mol m^{-3}
q_m	parameter of Langmuir isotherm	mol m^{-3}
Δq_τ	increment of the adsorbate concentration at time τ	mol m^{-3}
Δq_∞	increment of the adsorbate concentration at equilibrium	mol m^{-3}
R	gas constant	$\text{J mol}^{-1} \text{ K}^{-1}$
r	radial coordinate	m
r_0	particle radius	m

S_p	specific surface	$\text{m}^2 \text{g}^{-1}$
T	temperature	K
z	axial coordinate	m

Greek Letters

β	porosity	
γ_τ	dimensionless adsorbate concentration defined by eqn (4) or eqn (6) for adsorption and desorption, respectively	
μ_n	roots of the Bessel function of the 1st type and zero order	
τ	time	s
θ	degree of surface coverage	
ρ_p	particle density	kg m^{-3}

Subscripts

calc	calculated
exp	experimental
i	number of measurements in adsorption or desorption step
m	index of series in eqns (4) and (6)
n	index of series in eqns (4) and (6)
n	overall number of data in one adsorption or desorption step, eqn (7)

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