MASS TRANSFER IN A GRANULAR COLUMN IN THE PRESENCE OF NONEQUILIBRIUM SORPTION AT THE PHASE INTERFACE

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The direct and inverse problems of nonequilibrium sorption dynamics are considered for the case of a variable filtration velocity; the direct problem is solved by means of orthogonal polynomials and the inverse problem with the help of statistical moments.

When a flow of fluid material passes through a column filled with spherical sorbent granules, the filtration velocity of the flow will increase. In general the pressure gradient in a granular column (allowing for both inertial and viscous forces) depends nonlinearly on the filtration velocity [1, 2]. For fairly low velocities only the viscous forces have to be taken into account. In this case the pressure gradient [3]

$$|\operatorname{grad} p| = 600\frac{(1-\sigma)^2 u\eta}{a^2\sigma^3}. \tag{1}$$

Regarding the quantity of gas involved as constant and considering Eq. (1) for the case of weak turbulence, we may find the change taking place in the filtration velocity along the column

$$u = \frac{u_0}{\sqrt{1 - z/L_0}}, \tag{2}$$

where $L_0 = p_0 a^2\sigma^3/1200u_0(1-\sigma)^2\eta$.

In the presence of sorption at the phase interface, the same transfer in the granular column is described by the material-balance equation and the equation of sorption kinetics. The kinetics of the acts of sorption for small quantities of the sorbate in the solution (or weak adsorption on a uniform surface) are described by a linear differential equation [4]

$$\frac{\partial q^0}{\partial t} = k_1 c^0 - k_2 q^0. \tag{3}$$

At equilibrium the linear differential equation (3) passes into the linear sorption isotherm. The linear equation of sorption kinetics (3), despite its limitations, is of particular interest in view of the fact that it enables us to analyze the sorption of each component of a mixture independently of the presence of the other components. Using the solution of the sorption-dynamics problem for one component, we may thus analyze the effect of the rate of the acts of sorption and the mass-transfer velocity on the form of the dynamic output curves of various sorbates, which is important in choosing the optimum conditions for a number of processes in chemical technology. We describe the change in the concentration of sorbate inside the spherical sorbent grains by the equation of material balance in the grain:

$$\frac{\partial c^0}{\partial t} = D_i\frac{1}{r^2}\cdot\frac{\partial}{\partial r}\left(r^2\frac{\partial c^0}{\partial r}\right) - \frac{\partial q^0}{\partial t}. \tag{4}$$

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In order to gain a correct solution of Eqs. (4) and (3), we set out the following initial (zero) and boundary conditions:

a) continuity of the external and internal fluxes at the boundary of a spherical grain

$$\beta(c - c^0|_{r=a}) = D_i \frac{\partial c^0}{\partial r}\Big|_{r=a}; \tag{5}$$

b) symmetry in the center of the grain

$$\frac{\partial c^0}{\partial r}\Big|_{r=0} = 0, \tag{6}$$

where $\beta = \beta_0 (u/u_0)^{1/2}$ [5].

Using the integral Laplace transformation, we now find a solution to the system of equations (3), (4) with boundary conditions (5), (6). After certain transformations, we write the following for the Laplace transforms:

$$\tilde{c}^0 = \left(\frac{a}{r}\right)\left(\frac{\operatorname{sh}\lambda r}{\operatorname{sh}\lambda a}\right)\frac{\beta\tilde{c}}{\left[\beta + \frac{D_i}{a}(\lambda a \operatorname{cth}\lambda a - 1)\right]}, \tag{7}$$

where

$$\lambda = \left[\frac{1}{D_i}\left(p + \frac{pk_1}{p + k_2}\right)\right]^{1/2}.$$

For a cylindrical column, the equation of material balance, allowing for longitudinal mixing, takes the form

$$\frac{\partial c}{\partial t} + \frac{\partial (uc)}{\partial z} + \delta\gamma(c - c^0|_{r=a}) = \frac{\partial}{\partial z}\left[D(u)\frac{\partial c}{\partial z}\right], \tag{8}$$

where

$$\gamma = \frac{3\beta}{a};\quad D(u) = D_g + D_1 u + D_2 u^2;\quad \delta = \frac{1-\sigma}{\sigma}.$$

In view of the fact that transient field of concentrations exists in the column, the effective longitudinal mixing may be described by a dispersion coefficient. In general, the coefficient of dispersion D(u) is made up of the molecular diffusion D_g in the narrow channels of the sorbent grain [6], the convective mixing D_1u in the granular layer which takes place on passing around the grains [6], the turbulent mixing due to turbulent pulsations [7], the mixing due to velocity fluctuations in the granular layer, the mixing D_2u^2 due to the existence of a velocity profile in the column cross section (Taylor diffusion) [8], and the mixing in the stagnant zones between the grains (relaxation dispersion) [9]. For a liquid flow, the coefficient of relaxation dispersion is greater than for a gas flow, since hardly any stagnant zones are formed in the latter.

In order to present a correct formulation for the problem of sorption dynamics in a granular column, we write down the zero initial and boundary conditions

$$c(z, t)|_{z=0} = c_0 f(t), \tag{9}$$

in which for frontal sorption dynamics $f_1(t) = 1$, for developing sorption dynamics and short columns $f_2'(t) = \delta(t)$, and for long columns $f_2''(t) = 1 - \eta(t - t_0)$. The time t_0 for admitting the test substance into the column should be considerable in the latter case owing to the substantial liquefaction of the test substance (sample) at the exit from the long column.

Using the integral Laplace transformation, we rewrite the system of equations (7), (8) for the transforms in the following manner:

$$\frac{d}{dz}\left[D(u)\frac{d\tilde{c}(p, z)}{dz}\right] - \frac{d(u\tilde{c}(p, z))}{dz} - \varphi\tilde{c}(p, z) = 0 \tag{10}$$

with boundary conditions

$$\tilde{f}(p) = \begin{cases} \tilde{f}_1(p) = \dfrac{1}{p}, & (11a) \\ \tilde{f}'_2(p) = 1, & (11b) \\ \tilde{f}''_2(p) = \dfrac{1}{p}\left[1 - \exp(-pt_0)\right], & (11c) \end{cases}$$

where

$$\varphi(p) = p + \frac{\dfrac{3D_i}{a^2}\,\delta(\lambda a \operatorname{cth} \lambda a - 1)}{\left[\gamma + \dfrac{3D_i}{a^2}(\lambda a \operatorname{cth} \lambda a - 1)\right]} = \sum_{n=1} w_n(-1)^n p^n. \tag{12}$$

If longitudinal mixing has a considerable influence on the mass transfer in the granular layer ($D \neq 0$), and the length of the column is such that $z \sim L_0$, we must solve Eq. (10) rigorously.

In view of the fact that, in order to construct the solutions, we require expressions for various statistical moments, we shall seek the solution to Eq. (10) in the form of a series in p:

$$\tilde{c}(z, p) = \sum_{m=0} g_m p^m + \sum_{m=0} h_m p^m = Ag(z) + Bh(z). \tag{13}$$

Substituting (13) into (10) we obtain the system

$$\begin{gathered} \frac{d}{dz}\left[D(u)\frac{dg_0}{dz}\right] - \frac{dug_0}{dz} = 0, \quad \frac{d}{dz}\left[D(u)\frac{dh_0}{dz}\right] - \frac{duh_0}{dz} = 0, \\ \frac{d}{dz}\left[D(u)\frac{dg_m}{dz}\right] - \frac{dug_m}{dz} = \sum_{i=0} g_i(-1)^{m-i+1} w_{m-i} \quad (m - i \geqslant 1), \\ \frac{d}{dz}\left[D(u)\frac{dh_m}{dz}\right] - \frac{duh_m}{dz} = \sum_{i=0} h_i(-1)^{m-i+1} w_{m-i} \quad (m - i \geqslant 1). \end{gathered} \tag{14}$$

Solving the system (14) successively we find

$$\begin{gathered} g_0 = \exp\left(\int \frac{u}{D}\,dz\right)\int \exp\left(-\int \frac{u}{D}\,dz\right)\frac{dz}{D}, \\ g_m = \exp\left(\int \frac{u}{D}\,dz\right)\int \left\{\exp\left(-\int \frac{u}{D}\,dz\right)\frac{1}{D} \right. \\ \left. \times \left[\sum_{i=0} \int (-1)^{m-i+1} w_{m-i} g_i\,dz\right]\right\} dz, \\ h_0 = \exp\left(\int \frac{u}{D}\,dz\right), \\ h_m = \exp\left(\int \frac{u}{D}\,dz\right)\int \left\{\exp\left(-\int \frac{u}{D}\,dz\right)\frac{1}{D} \right. \\ \left. \times \left[\sum_{i=0} \int (-1)^{m-i+1} w_{m-i} h_i\,dz\right]\right\} dz. \end{gathered} \tag{15}$$

We find the constants A, B from the boundary condition (11) at $z = 0$ and the boundary condition at $z = L$; these conditions specify the continuity of the concentrations $\tilde{c}(z, p)$ and concentration flux $j = D[d\tilde{c}(p, z)/dz] - u\tilde{c}(p, z)$ in Eq. (13) and in the equation obtained from the solution of Eq. (10) with $u = u_0(1 - L/L_0)^{-1/2} = \text{const}$. After certain transformations, allowing for the boundary condition (11b), we may write

$$\tilde{c}(z, p)\big|_{z=L} = \frac{g(L)\left.\dfrac{dh}{dz}\right|_{z=L} - h(L)\left.\dfrac{dg}{dz}\right|_{z=L}}{g(0)\left.\dfrac{dh}{dz}\right|_{z=L} - h(0)\left.\dfrac{dg}{dz}\right|_{z=L}} = \sum_{n=0} v_n p^n. \tag{16}$$

We find the expressions for the initial moments from [10]

$$\alpha_n = \lim_{p\to 0}\left[\frac{(-1)^n}{\tilde{c}(z,\ p)}\cdot\frac{d^{(n)}\tilde{c}(z,\ p)}{dp^n}\right] = (-1)^n n!\,\frac{v_n}{v_0}. \tag{17}$$

The initial and central moments are related by

$$\mu_n = \sum_{k=0}^{n} C_n^k(-\alpha_1)^k\alpha_{n-k}. \tag{18}$$

We see from Eqs. (15) and (16) that finding the moments reduces to simple quadratures. If we substitute (2) into (15), after integration we obtain some extremely cumbersome expressions for the moments, very difficult to use. Let us therefore find the simplest expressions for the moments with D = 0 (i.e., when the longitudinal mixing plays a minor part). In this case, substituting $\tilde{c} = c_0\,(u_0/u)\sum_{n=0} f_n p^n$ into Eq. (10) with due allowance for Eq. (12) and the boundary condition (11b), we obtain

$$f_m = -\sum_{i=0}\int \frac{w_{m-i}}{u}(-1)^{m-i+1}f_i\,dz \qquad (m-i\geqslant 1), \tag{19}$$

where $f_0 = 1$; $f_m|_{z=0} = 0$ $(m \geq 1)$.

Using Eq. (19) and the relations (17) and (18), we may write down the first initial and three central moments

$$\alpha_1 = \frac{2[1+(1+k)\,\delta]\,L_0}{3u_0}\left[1-(1-L/L_0)^{3/2}\right], \tag{20}$$

$$\mu_2 = 2\alpha_1\left\{\frac{1}{k_2}+\tau_i+\frac{6}{7\gamma_0^*}\left[1-(1-L/L_0)^{7/4}\right]\left[1-(1-L/L_0)^{3/2}\right]^{-1}\right\}, \tag{21}$$

$$\begin{aligned}\mu_3 = 6\alpha_1\Bigg\{&\frac{1}{k_2^2}+\frac{2\tau_i}{k_2}+\frac{10}{7}\tau_i^2+\frac{12}{7\gamma_0^*}\left(\frac{1}{k_2}+\tau_i\right)\left[1-(1-L/L_0)^{7/4}\right]\\ &\times\left[1-(1-L/L_0)^{3/2}\right]^{-1}+\frac{3}{4\gamma_0^{*2}}\left[1-(1-L/L_0)^{2}\right]\left[1-(1-L/L_0)^{3/2}\right]^{-1}\Bigg\},\end{aligned} \tag{22}$$

$$\begin{aligned}\mu_4 = 3\mu_2^2+24\alpha_1\Bigg\{&\frac{1}{k_2^3}+\frac{3\tau_i}{k_2^2}+\frac{15}{7}\tau_i^3+\frac{6}{7\gamma_0^*}\Bigg(\frac{3}{k_2^2}\\ &+\frac{6\tau_i}{k_2}+\frac{27}{7}\tau_i^2\Bigg)\left[1-(1-L/L_0)^{7/4}\right]\left[1-(1-L/L_0)^{3/2}\right]^{-1}\\ &+\frac{9}{4\gamma_0^{*2}}\left(\frac{1}{k_2}+\tau_i\right)\left[1-(1-L/L_0)^{2}\right]\left[1-(1-L/L_0)^{3/2}\right]^{-1}\\ &+\frac{2}{3\gamma_0^{*3}}\left[1-(1-L/L_0)^{9/4}\right]\cdot\left[1-(1-L/L_0)^{3/2}\right]^{-1}\Bigg\},\end{aligned} \tag{23}$$

where

$$k = \frac{k_1}{k_2};\quad \tau_i = \frac{a^2(k+1)}{15D_i};\quad \gamma_0^* = \frac{\gamma_0}{k+1} = \frac{3\beta_0}{a(k+1)}. \tag{24}$$

The form of the dynamic output curve (which is almost Gaussian) is mainly determined by the specification of these four moments. The first initial moment (α_1) characterizes the position of the "center" of the curve, the second (μ_2) the degree of "scatter" of the curve, the third (μ_3) gives a measure of the asymmetry of the "bias" (or "skewness"), where the asymmetry coefficient $Sk = \mu_3/\mu_2^{3/2}$ and for $Sk > 0$ the leading edge is steeper than the trailing edge, while for $Sk < 0$ the reverse is the case. For a symmetrical curve all the odd central moments are equal to zero. The fourth moment (μ_4) characterizes the "sharpness," the coefficient of sharpness or the "excess" being $E_x = \mu_4/\mu_2^2 - 3$ (for a Gaussian curve $E_x = 3$). For a curve sharper than a Gaussian, $E_x > 3$, while for a flatter curve $E_x < 3$. Using the expressions for the moments (20)-(23), we may analyze the effect of a variable filtration velocity u on the form of the dynamic curve. For a flow velocity variation of $u_0 \le u < \infty$ $(0 \le z \le L_0)$ the limiting value of the first initial moment $\alpha_{1p} = (2k\delta L_0/3u_0) = (2/3)\alpha_{01}$ (α_{01} is the initial moment for $u = u_0 = \text{const.}$). In view of the fact that $1 \le \alpha_1/\alpha_{01} \le 2/3$, it is reasonable to introduce an "effective velocity," in such a manner that the initial moment calculated by Eq. (20) for an effective velocity u* constant along the column

$$\alpha_1 = \frac{2[1+(1+k)\delta]L_0}{3u_0}\left[1-(1-L/L_0)^{3/2}\right] = \frac{[1+(1+k)\delta]L}{u^*}.$$

Hence the effective flow velocity

$$u^* = \frac{3u_0}{2}\left[1-(1-L/L_0)^{3/2}\right]^{-1} = \lambda u_0, \tag{25}$$

where $1 \le \lambda \le 3/2$. The limiting value of the last term in (21) calculated rigorously for $L \to L_0$ equals $6/7\gamma_0^* = 0.855\,(1/\gamma_0^*)$. Using the expression for the limiting velocity $u_p^* = (3/2)\,u_0$, we may find the approximate limiting value of the last term in (21), which equals $(\gamma_0^*\sqrt{u^*/u_0})^{-1} = 0.815\,(1/\gamma_0^*)$ (the difference from the rigorous value is 5%). Analogous calculations for the last term of (22) gives the exact limiting value $0.75\,(1/(\gamma_0^*)^2)$; the approximate value using (25) is $((1/\gamma_0^{*2})\,(u_0/u^*))^{-1} \simeq 0.66\,(1/\gamma_0^{*2})$ (difference 12%). These calculations confirm the reasonability of introducing the effective flow velocity given by (25).

On analyzing the moments (20)-(23), we see that for a variation of $u_0 \le u < \infty$ $(0 \le z \le L_0)$ the output curve becomes flatter and more asymmetrical. The limiting changes in the moments are as follows. The first initial moment α_1 falls by 34%, the second central moment μ_2 falls more than 34%, the asymmetry coefficient Sk increases by 17%, the excess E_X diminishes by 34%.

For an effective flow velocity $u^* = \text{const}$, we shall seek the solution to Eq. (10), allowing for conditions (11b) and Eq. (12), in the form

$$\tilde{c}(z,\ p) = c_0 \sum_{n=0} s_n p^n. \tag{26}$$

Substituting (26) into (10), we obtain

$$s_n = \frac{1}{D(u^*)}\exp\left(\frac{u^* z}{D(u^*)}\right)\int\left\{\exp\left(-\frac{u^* z}{D(u^*)}\right)\right. \times \left.\sum_{i=0}\left[(-1)^{n-i+1} w_{n-i}\int s_i dz\right]\right\}dz, \tag{27}$$

where $s_0 = 1$, $s_n|_{z=0} = 0$ $(n \ge 1)$.

Using (27), (17), (18), and (25), we may write down the expressions for the first initial and three central moments thus:

$$\alpha_1 = \frac{[1+(1+k)\delta]L}{\lambda u_0} \tag{28}$$

$$\mu_2 = 2\alpha_1\left(\frac{1}{k_2} + \tau_i + \frac{1}{\sqrt{\lambda}\,\gamma_0^*} + \tau_l\right), \tag{29}$$

$$\mu_3 = 6\alpha_1\left[\frac{1}{k_2^2} + \frac{2}{k_2}\left(\tau_i + \frac{1}{\sqrt{\lambda}\,\gamma_0^*}\right) + \frac{10}{7}\tau_i^2 + \frac{2\tau_i}{\sqrt{\lambda}\,\gamma_0^*} + \frac{1}{\lambda\gamma_0^{*2}}\right] + 6\mu_2\tau_l, \tag{30}$$

$$\mu_4 = 3\mu_2^2 + 24\alpha_1\left\{\left(\frac{1}{k_2} + \tau_i + \frac{1}{\sqrt{\lambda}\,\gamma_0^*} + \tau_l\right)^3 + \tau_l^3 + \left(3\tau_l^2 + \frac{6}{7}\tau_i^2\right)\left(\frac{1}{k_2} + \tau_i + \frac{1}{\sqrt{\lambda}\,\gamma_0^*} + \tau_l\right) + \frac{3}{7}\cdot\frac{\tau_i^2}{k_2} + \frac{2}{7}\tau_i^3\right\}, \tag{31}$$

where

$$\tau_l = \frac{[1+(1+k)\delta]D(u^*)}{(u^*)^2} = [1+(1+k)\delta]\left(\frac{D_g}{\lambda^2 u_0^2} + \frac{D_1}{\lambda u_0} + D_2\right). \tag{32}$$

We showed in the foregoing discussion that the "spreading" of the dynamic output curve was determined by the second central moment. We see from (29) that the total rate of mass transfer is determined by the rate of the acts of sorption at the phase interface, the velocity of internal and external mass transfer, and the longitudinal mixing. The physical meaning of the terms in round brackets in (29) is as follows: $1/k_2$ is the delay time (retardation) due to the kinetics of the acts of sorption; τ_i is the delay time due to the finite rate of mass transfer within the sorbent grains; $1/\sqrt{\lambda\gamma_0^*}$ is the delay time due to the finite rate of mass transfer at the outer boundary of the sorbent grains; τ_l is the delay time due to the existence of longitudinal mixing.

We see from Eq. (26) that the original functions may only be found on the basis of various asymptotic approximations $t \to \infty$ (or $p \to 0$). For a fixed column length L the dynamic output curves (direct problem) must be sought in terms of orthogonal Hermite polynomials [11]

$$c(L, t) = \sum_{n=0} A_n H_n\left(\frac{t-\alpha_1}{\sqrt{2\mu_2}}\right) \exp\left[-\left(\frac{t-\alpha_1}{\sqrt{2\mu_2}}\right)^2\right]. \tag{33}$$

Using the expression for the Hermite polynomials $H_n(x)$ given in [12] and making use of their orthogonal properties, we derive expressions for the coefficients

$$A_n = \sum_{k=0}^{\left[\frac{n}{2}\right]} \frac{(-1)^k 2^{-\frac{n}{2}-k} \mu_{n-2k}}{\sqrt{2\pi}\,(n-2k)!\,\mu_2^{\left(\frac{n}{2}-k+\frac{1}{2}\right)}}.$$

Using the experimental dynamic curves and the analytical expressions for the moments (28)-(31), we may solve the algebraical system of equations and find the parameters γ_0^*, τ_i, $1/k_2$, D (inverse problem). The solution to the algebraical equation of the third order may be found by the Cardan method and that of the fourth-order equation by Euler's method [13].

We note that the mass transfer in a granular column here considered involves the concept of "effective retardation," the delay time due to different forms of mass transfer being given by Eqs. (24) and (32). Chromatography makes extensive use of the concept of the "effective theoretical plate" considered by Van Deemter [14].

In order to describe the dynamic output curve, Van Deemter [14] used a symmetrical Gauss curve and the equilibrium diffusion equation of kinetics, which only approximately describes the mass transfer in the sorbent grains [15]. The real dynamic output curves are always asymmetrical (μ_3 in Eq. (30) is always nonzero), so that the Van Deemter theory only approximately describes the shape of the dynamic output curves (direct problem). In order to determine the kinetic and dynamic parameters (inverse problem) from the experimental dynamic output characteristics for a variable filtration flow velocity, it is quite impossible to use the Van Deemter theory, and the moment equations (28)-(31) must be employed.

The solution of the equations of nonequilibrium sorption dynamics was considered in [16, 17] on the basis of statistical moments with a constant filtration velocity for a specified initial concentration distribution in an unbounded, infinite column (unbounded problem).

The dependence of the flow filtration velocity on the length of the column was considered for the case of equilibrium sorption dynamics in [18, 19]. In these papers the authors used the diffusion kinetic equation [20] (which only approximately describes mass transfer in the sorbent grains), not using the exact expression for the coefficient of longitudinal diffusion ($D = D_1 u$). The method of obtaining the effective theoretical plate equation was analogous to [14]; hence the calculations of [18, 19] may only be used to describe symmetrical dynamic curves.

An experimental confirmation of the effect of variable filtration velocity on the dynamic output curves was obtained in [21, 22]. However, in view of the fact that the dynamic curves were recorded for a short column length (the filtration velocity varied very little) and some of the parameters characterizing the column not presented, these experimental results can hardly be compared with the theoretical results of the present investigation.

NOTATION

σ	proportion of free space in the column filled with spherical sorbent grains;
η	dynamic viscosity;
a	radius of the spherical sorbent grains;
p_0	gas pressure at inlet;
u_0	linear flow velocity at inlet;
c^0	concentration of sorbent within the free space of the sorbent grains;
q^0	concentration of absorbed material;
k_1, k_2	sorption and desorption constants;
D_i	coefficient of (internal) diffusion in the channels of the sorbent grains;
c	concentration of sorbate in the flow;
β	coefficient of (external) mass transfer;
γ	kinetic coefficient allowing for external mass transfer;
D	dispersion coefficient allowing for effective longitudinal mixing;
α_n	initial moments;
μ_n	central moments.

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