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F. W. Petersen[a]; J. S. J. van Deventer[b]
[a] Department of Chemical Engineering, Cape Technikon, Cape Town, South Africa [b] Department of Chemical Engineering, The University of Melbourne, Parkville, Australia

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A PROBALISTIC MODEL FOR THE INTRUSION OF SOLID FINES IN ACTIVATED CARBON

F. W. Petersen[1] and J. S. J. van Deventer[2]

[1]Department of Chemical Engineering,
Cape Technikon, Cape Town, South Africa
[2]Department of Chemical Engineering,
The University of Melbourne, Parkville, Australia

ABSTRACT

The carbon-in-pulp (CIP) process is used extensively in the mining industry to recover dissolved gold from cyanide-leached pulps. The pulp normally contains inorganic material that could penetrate the pores of the carbon. In the formulation of the model it was proposed that the loading of solid species from the bulk suspension onto activated carbon is dependent on different probabilities such as the activity of pores at the outer shell of the carbon, the concentration of fines in suspension, the stirring speed of the suspension, zeta potential of solid fines, and the fraction of fines smaller than the size of a specific pore.

The proposed model was shown to describe the intrusion of solid fines in carbon accurately under a wide variety of conditions. The model was not so sensitive towards zeta potential of the solid particles or the effective stirring speed. The concentration of solid fines in the suspension influenced the collision-kinetics and hence the rate of intrusion. The concentration decay of solid fines in suspension was very sensitive at low concentrations, whereas at high concentrations the model-output concentration remained constant.

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INTRODUCTION

Developments in gold metallurgy have resulted in carbon-in-pulp (CIP) technology being used extensively to recover dissolved gold from cyanide leach pulps. Activated carbon is generally a nonpolar material, and it adsorbs organic compounds in preference to inorganic compounds (1). It has been shown that the organic compounds such as flotation reagents or lubricating oils, which are insoluble in water, have an adverse effect on gold adsorption onto activated carbon (2,3). Fleming and Nicol (4) and Jones and Linge (5) showed that the presence of inert solids in a pulp has a marked effect on the rate of gold extraction. By increasing the concentration of inert solids in the pulp, they found that the rate of gold uptake decreased. However, it was not certain whether this was due to a decrease in the mixing efficiency with increasing concentration, or whether this was due to physical blinding of the carbon surface by slimes in the pulp.

Although numerous authors have confirmed the presence of inorganic species inside the pores of the activated carbon, they have done so by monitoring the concentration decay of gold cyanide. Smith et al. (6) presented micrographs of the carbon particles showing that the organic material on the surface of extruded peat and coconut shell carbons was mainly calcite, with smaller amounts of silica, clays, and sulfide minerals. These authors did not provide any information on the presence of inorganic compounds inside the carbon particle. Jones et al. (7) attempted to provide more insight into the distribution of inorganics on the external and internal surfaces of an Australian activated carbon. Analytical techniques such as optical microscopy and electron microprobe analysis were used to substantiate their findings. Although these authors claimed that the calcite was virtually the only inorganic substance present, no conclusive information on the distribution thereof inside the carbon was provided.

A lack of information exists on the distribution of fine inorganic material inside activated carbon. The main reasons for this are probably the following:

a) The complexity of the internal structure of activated carbon
b) The heterogeneity of the internal surface of activated carbon
c) The lack of available analytical methods to assist in the quantification

A few attempts have been published to model the intrusion of particulate suspensions in porous media (8,9). However, the concept of modeling the intrusion of fine solids into adsorbents such as activated carbon has not been investigated as yet.

EXPERIMENTAL

Carbon

A coconut shell activated carbon, Pica, with an average diameter of 1.4 mm and apparent density of 838 kg·m^{-3} was used. The Langmuir surface area of the carbon was determined with a Micromeritics Flowsorb 2300 apparatus as 1020 $m^2 \cdot g^{-1}$. The carbon revealed a point of zero charge at a pH of 1.8 and a natural pH in distilled water of 5.8. The pore volume of the carbon was determined as 7.24×10^{-4} $m^3 \cdot kg^{-1}$ by measuring the mass loss on oven drying of a sample of carbon saturated with water.

Inorganic Material

Precipitated silica and silica sand ground for different time periods were used to simulate fine material. By carefully monitoring the pH, the chemical conditions were controlled so that these particles did not dissolve during the adsorption reactions. Precipitated silica (average particle size <15 microns) was used in all the experiments, except for experiments where the effect of particle size were investigated, in which case silica sand ground for different time intervals was used.

Analytical Methods

The pH of all solutions was monitored by using a Beckman Chem-Mate pH-meter. A muffle furnace was used for ashing tests to determine the intrusion of fines into activated carbon. Zeta potential measurements were conducted with a Rank Brothers Mark II instrument. A known amount of fine carbon or silica was mixed with 1 liter of distilled water and conditioned for 20 min at 25°C before any measurements were made. The zeta potentials are average values at potential differences of 51.9 and 71.5 mV.

Scanning electron microscopy (SEM) was performed on the activated carbon to detect the presence of fines inside the carbon pores. Secondary ion mass spectrometry (SIMS) was used to determine the concentration gradient of inert species inside the carbon. This technique is based on the fact that charged atomic and molecular species are ejected from the surface of a condensed phase under heavy particle bombardment. The output is in the form of a line scan and depicts the relative concentration of fine silica particles to the distance inside the carbon particle.

Intrusion Experiments

The intrusion of fine material into activated carbon was examined in a batch stirred tank reactor of internal diameter of 11 cm, 15 cm height, and three evenly spaced baffles of 1 cm width. Agitation was provided by a flat blade impeller of 6 cm width and 5 cm height, driven by a variable-speed motor. Typically, 1.0 g of activated carbon was contacted with 1 liter of distilled water to which different quantities of fine particles had been added. After the pretreatment period, the carbon particles were washed thoroughly to remove any silica from the external surface.

MODEL FORMULATION

The following experimental observations had to be considered in formulating the model for the intrusion of solid fines into porous adsorbents (10):

a) The loading of solid fines onto the adsorbents is a function of the size and to a lesser extent the zeta potential of the solids.
b) The equilibrium loading of solid fines is reached in less than 3 h.
c) The concentration of solid fines in suspension does not influence the final loading on the carbon particle. However, it affects the rate of intrusion of the solid fines.
d) The effect of stirring speed on the final solid loading is negligible. However, it slightly influences the rate of intrusion.

Activated carbons are usually poly disperse or have broad pore size distributions. In this paper a fixed pore size distribution was assumed, although the general model accounted for a change in such a distribution. It is further assumed that the particles only penetrated the larger pores (macropores), and intrusion into the smaller pores (micropores) is therefore ignored.

The loading of the solid species *i* from the bulk suspension onto the activated carbon particle is dependent on different *probabilities*. Only species *i* reaches the external surface of the carbon. The intrusion of species *i* into a pore of relevant size can be described via *Fickian transport*.

The following assumptions were made in the development of the model:

- The pores are uniformly distributed throughout the carbon particles, and the pore radius distribution does not change from the outside to the inside of the particles.
- The solid fines will only penetrate the carbon pores of comparable or larger size.
- The maximum loading of fines on the carbon is attained if all the pores are filled with fine solids.
- The radial transport of fine solids in the microprobes can be described by a *Fickian transport* mechanism based on the gradient in the loading of fines.

Pore Mass Balance

A mass balance over the pores of the carbon yields:

$$\frac{\partial Q}{\partial t} = D \frac{\partial^2 Q}{\partial r^2} + \frac{2D}{r} \frac{\partial Q}{\partial r} \tag{1}$$

Changes in Loading of Solid Fines at the Surface of the Adsorbent

It is assumed that the accumulation of fines at the outer shell of the carbon is proportional to the probabilities of availability of pores at the outer shell of the carbon, the concentration of fines in suspension, the stirring speed of suspension, the zeta potential of solid fines, and the fraction of fines smaller than the pore size.

$$\left.\frac{\partial Q}{\partial t}\right|_{\substack{\text{outside}\\\text{surface}}} \alpha\, P\left(\frac{Q_{\max} - Q(t)}{Q_{\max}}\right) \cdot (C) \cdot (N) \cdot (\xi) \cdot (d) \tag{2}$$

which simplifies to:

$$\left.\frac{\partial Q}{\partial t}\right|_{\substack{\text{outside}\\\text{surface}}} = P\left(\frac{Q_{\max} - Q(t)}{Q_{\max}}\right) \cdot (C) \cdot (N) \cdot (\xi) \cdot (d) \tag{3}$$

with P = proportionality constant, and describes the rate of collision between the carbon particle and silica fines.

a) The term $\dfrac{Q_{\max} - Q(t)}{Q_{\max}}$

quantifies the availability of pores for intrusion of solids and is responsible for driving force of transport into the carbon particle.

b) C is the concentration term that gives the amount of solids in suspension per volume of liquid. The concentration is normalized in Eq. (3) with reference to the initial concentration ($C = C_t/C_i$).

c) N represents the stirring speed of the suspension. In view of the fact that this parameter influenced the rate of intrusion slightly, its effect will be incorporated into parameter P.

d) ξ represents the zeta potential of the solid fines. Although zeta potential influenced the final loading of fines on the carbon very slightly, its major effect was on the rate of intrusion. Therefore, this effect will also be incorporated in parameter P.

e) The fraction of fines smaller than pore size d, will affect the value of the maximum loading $Q_{\max}$.

A material balance on the carbon gives:

$$\frac{\partial Q}{\partial t} = \frac{-\partial C_t}{\partial t}\left(\frac{V}{M_c}\right) \tag{4}$$

where C_t is the concentration of fines in suspension at time t, M_c is the mass of carbon, and V is the volume of the liquid.

Determination of Qmax for Different Particle Size Distributions

If the different pore size distribution of the adsorbent is represented by $d_C(x)$ and the different particle size distribution of the fine solids is given by $d_s(x)$, where $x =$ the specific size, then the maximum loading of solids for each x can be calculated by:

$$= \frac{\left[Q_{\max,empirical} M_c \int_x^{\infty} \frac{\frac{\pi}{4}x^2}{A}\, d_c(x)dx\right]}{Mc} \tag{5}$$

In Eq. (5) it is assumed that all the available pores of the adsorbent are filled with solids of a particular size. However, it is necessary to limit the maximum loading if the amount of solids generally available in suspension is inadequate. In such a case the mass of solids M_s in suspension will be the limiting factor. Therefore, Eq. (5) can be extended to:

$$Q_{\max}\ \begin{matrix}\text{different particle}\\ \text{size distributions}\end{matrix} =$$

$$\int_0^{\infty} \mathit{Minimum}\, \frac{Q_{\max,empirical}.M_c \int_x^{\infty} \frac{\frac{\pi}{4}x^2 d_c(x)dx}{\frac{\pi}{4}\int_0^{\infty} x^2 d_c(x)dx};\ M_s}{M_c}\ d_s(x)dx \tag{6}$$

EVALUATION OF MODEL

Intrusion Profile

The solid lines in the figures represent model predictions, whereas the symbols represent experimental data points. Figure 1 illustrates the kinetics of intrusion of fine silica particles into the pores of activated carbon. This profile was obtained by pretreating activated carbon with silica for different time intervals, rinsing them with distilled water, and ashing them in a muffle furnace at 900°C for 10 h, after which the loading of silica was calculated. The results obtained by ashing the carbon particles were relatively reproducible with an error margin of less than 6%.

Although the concentration of silica particles had a positive effect on the rate of loading, the concentration thereof did not influence the final equilibrium loading. When activated carbon was pretreated with silica particles and then suspended in a stirred tank reactor containing distilled water only, the silica loading on activated carbon did not decrease. This result indicated that the intruded particles diffused or penetrated into the pores of carbon and adhered not merely to the external pore openings. The results of Fig. 1 show that an increase in the concentration of silica particles in suspension increased the kinetics of intrusion of fines into the carbon's internal matrix. It is clear that the model gives an acceptable fit for the different concentrations. At a constant value of the transport coefficient ($4.5 \times 10^{-12} \mathrm{m^2 \cdot s^{-1}}$) and a value of 18 $\mathrm{mg \cdot g^{-1}}$ for $Q_{\max}$, a correlation coefficient of 0.97 was obtained, as depicted in Table 1.

Effect of Zeta Potential on Intrusion

Tests were conducted to investigate the intrusion of silica particles into the pores of activated carbon at different pH solutions. The different pHs corresponded

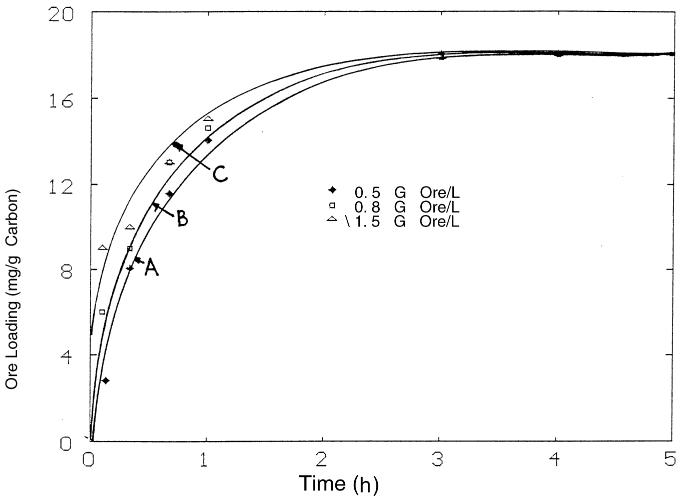

Figure 1. Model fit to the intrusion profiles at different solid concentrations in suspension ($M_c = 1.0$ g; $N = 250$ rpm; $V = 1.0$ L).

Table 1. Sensitivity of Model Parameters

Curve	Conditions	$D \times 10^{12}$ [($m^2 \cdot s^{-1}$)]	Q_{max}[($mg \cdot g^{-1}$)]	P [($mg \cdot g^{-1} \cdot s^{-1}$)]
A	0.5 $g \cdot L^{-1}$	4.5	18	1
B	0.8 $g \cdot L^{-1}$	4.5	18	1
C	1.5 $g \cdot L^{-1}$	4.5	18	1
D	$\varepsilon = -31$ mV	4.5	18	1.3
E	$\varepsilon = -33$ mV	4.5	18	1.5
F	2 h milled	4.3	3.8	1
G	5 h milled	4.1	8.2	1
H	24 h milled	4.5	18	1

to different zeta potentials. Intrusion tests were carried out in a similar manner as mentioned before. Figure 2 indicates an increase in the degree of intrusion of silica particles into activated carbon as the zeta potential becomes more negative. A low negative zeta potential was an indication of the relatively weak repulsion forces between the individual silica particles. The strong repulsion forces, characterized by the high negative zeta potential, caused the silica particles to disperse individually. Ultimately, this would allow a "smaller" particle to exist, and hence promotes the chance of penetrating a pore of the carbon. The model yields different values for the collision parameter P as the zeta potential changes. The transport coefficient, D, and Q_{max} remained constant at $4.5 \times 10^{-12} m^2 \cdot s^{-1}$ and 18 $mg \cdot g^{-1}$ re-

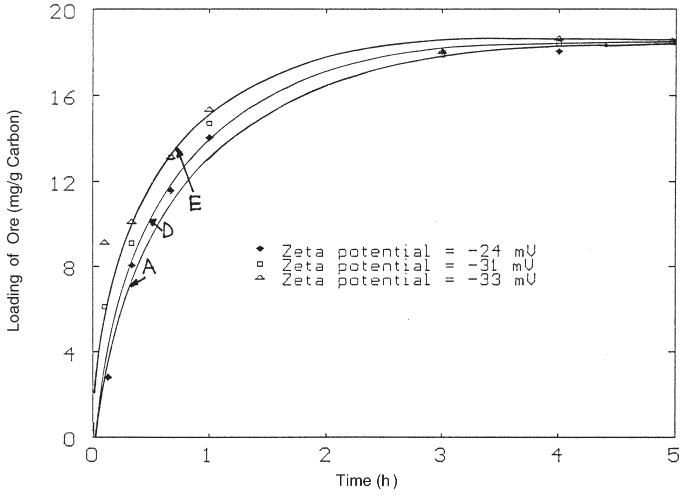

Figure 2. Model fit to the intrusion profiles at different levels of zeta potential (M_c = 1.0 g; N = 250 rpm; V = 1.0 L).

spectively. The variation in the value for P is to be expected, as the zeta potential affects the collision between the surface of the carbon and the solid fines.

Effect of Particle Size of Intrusion

Various tests were performed where activated carbon particles were pretreated with silica fines having different particle size distributions. Figure 3 presents the loading profile of silica fines for activated carbon for different particle size distributions of silica. Although the particle size distributions are not explicitly shown, they have been coupled to the time the silica had spent in an ordinary ball mill. Therefore, it can be assumed that with an increase in the time of milling, a finer particle size will be yielded. Figure 3 indicates clearly that with an increase in the time of milling, hence a finer particle size, the loading of silica fines increased onto activated carbon. This test was performed with a constant silica concentration in suspension, i.e., 2 g silica/L. It is interesting to note that for a coarser particle size (milled for 2 h), equilibrium is reached much faster than for a finer particle size (24 h). This means that coarser particles, which are still smaller than the pore size of the carbon, easily block the pores and do not allow further intrusion. It seems as if there is not an actual diffusion of silica species into the pores of the carbon, but rather a physical penetration that stops as soon as the silica particle comes in contact with the pore wall. It is clear that the size of the solids in-

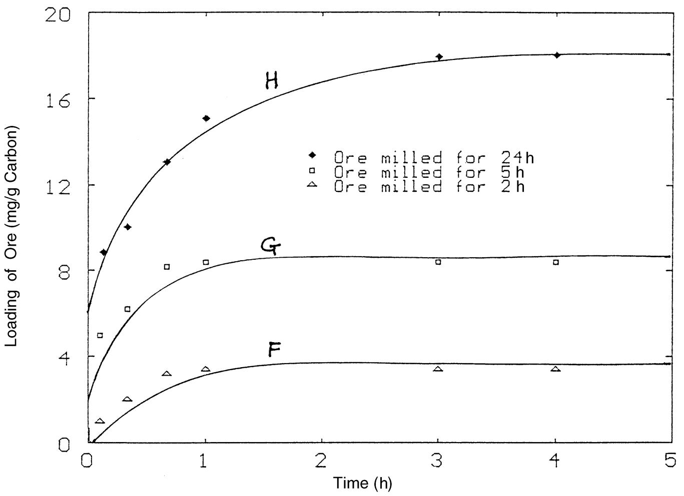

Figure 3. Model fit to the intrusion profiles for different sizes of solids in suspension ($M_c = 1.0$ g; $N = 250$ rpm; $V = 1.0$ L; $C_t = 1$g.L^{-1}).

fluences the value of $Q_{\max}$. In fact, the value of $Q_{\max}$ decreases from 18 $\text{mg}\cdot\text{g}^{-1}$ for 24 h milled silica sand to a value of 3.8 $\text{mg}\cdot\text{g}^{-1}$ for 2 h milled silica sand. It was not possible to obtain an acceptable fit by only changing the value of $Q_{\max}$.

SENSITIVITY ANALYSIS

This section contains simulations with the intrusion model. Unless otherwise specified, the following "standard" set of conditions and parameters was used in the sensitivity analysis of the model:

$Q_{\max} = 16\ \text{mg}\cdot\text{g}^{-1}$ Equilibrium loading
$M_c = 0.001\ \text{kg}$ Mass of carbon
$C = 1$ Dimensionless concentration
$P = 1\ \text{mg}\cdot\text{g}^{-1}\cdot\text{s}^{-1}$ Collision parameter
$V = 0.001\ \text{m}^3$ Volume of suspension
$D = 4.5 \times 10^{-12}\ \text{m}^2\cdot\text{s}^{-1}$ Diffusion coefficient
$C_t = 1\ \text{g}\cdot\text{L}^{-1}$ Concentration of fines in suspension

The sensitivity of the model for the transport coefficient, D, was investigated in Fig. 4. It can be seen that only the rate of intrusion is affected, and the same equilibrium loading is attained. As the transport coefficient decreases, the rate of loading of solid fines into the porous adsorbent decreases. Also, the value of Q_{max} remained the same.

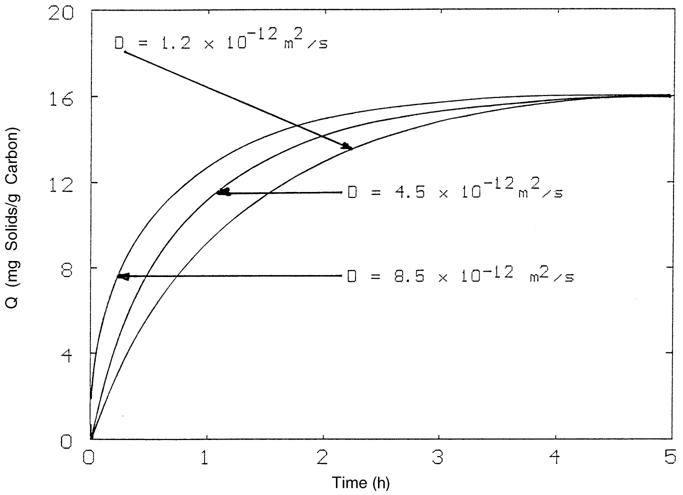

Figure 4. Sensitivity of the model to changes in the transport coefficient, *D*.

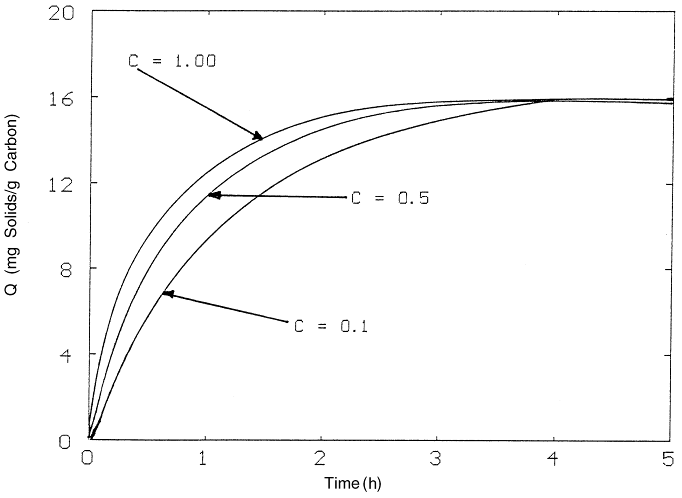

Figure 5. Sensitivity of the model to changes in the dimensionless concentration, *C*.

Figure 5 explores the effect of varying the dimensionless concentration in suspension. As the dimensionless concentration directly controls the collision kinetics, the decrease in the rate of intrusion as the dimensionless concentration drops is to be expected.

It can be seen from Fig. 6 that a change in the collision parameter *P* influences the kinetics of intrusion. This result was to be expected in view of the fact

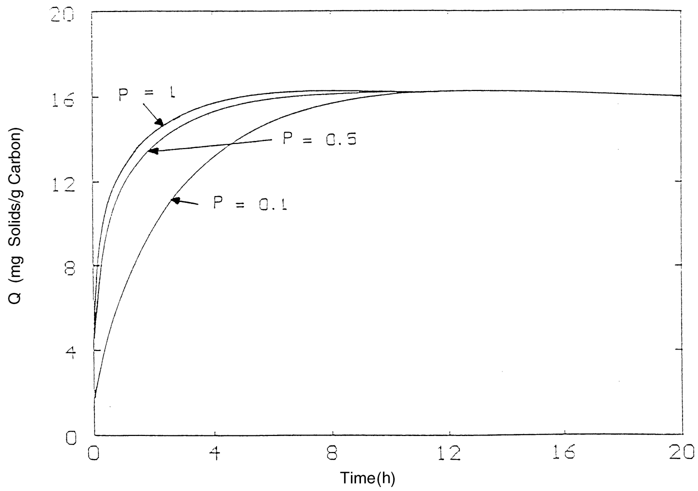

Figure 6. Sensitivity of the model to changes in the collision parameter, *P*.

that this parameter is a function of the stirring speed and the zeta potential and would increase the probability of a single solid particle to reach the surface of the carbon. A normal distribution was selected for both the pore size of the carbon and particle size of the solids. In determining the values for the mean size and standard deviation, it was critical to ensure that the integral of the normal probability equation was close to 1.

CONCLUSIONS

The proposed intrusion model was shown to describe the intrusion of solid fines in carbon accurately under a wide variety of conditions. This model was not so sensitive toward zeta potential of the solid particles of the effective stirring speed. The concentration of solid fines in the suspension influenced the collision kinetics and hence the rate of intrusion. The concentration decay of solid fines in suspension was very sensitive at low concentrations whereas, at high concentrations, the model output for concentration remained constant.

NOMENCLATURE

A	area (m^2)
C	concentration ($mg{\cdot}L^{-1}$)
C_t	concentration at time *t* ($mg{\cdot}L^{-1}$)
D	diffusion or transport coefficient ($m^2{\cdot}s^{-1}$)
d_p	particle size (m)
M	mass of carbon (kg)
N	stirring speed (rpm)
P	proportionality constant ($mg{\cdot}g^{-1}{\cdot}s^{-1}$)
$d_c(x)$	density function of pore size distribution of carbon
$d_s(x)$	density function of solid fines distribution
Q	loading on carbon ($mg{\cdot}g^{-1}$)
Q_{max}	maximum loading of solid fines on the carbon ($mg{\cdot}g^{-1}$)
r	radial distance (m)
t	time variable (s)
V	volume (m^3)
x	distance (m)

Subscripts

c	carbon
i	initial condition
s	liquid-carbon interface

APPENDIX A

Derivation of Equation (5):

If the differential pore size distribution of the adsorbent is represented by $d_c(x)$ and the differential particle size distribution of the fine solids is given by $d_s(x)$, where $x =$ the specific size, then the total area of pores on the external surface of the adsorbent

$$A = \frac{\pi}{4}\int_0^\infty x^2 d_c(x)\,dx \tag{A1}$$

The differential maximum loading for one specific pore size is proportional to the area fraction of size x, i.e.,

$$Q_{\max}(x)\,dx = \frac{\frac{\pi}{4}x^2}{A}\,d_c(x)\,dx \quad Q_{\max,empirical} \tag{A2}$$

Where $Q_{\max,empirical}$ is an empirical constant related to the loading capacity of the carbon for ultrafine solids.

The differential amount of solids that could potentially intrude at pore size x

$$= Q_{\max}(x)\,M_c\,dx \tag{A3}$$

Therefore, the amount of solids that could potentially intrude pores of size larger than x

$$= Q_{\max,empirical}\,M_c \int_x^\infty \frac{\frac{\pi}{4}x^2}{A}\,dc(x)dx \tag{A4}$$

The maximum loading of solids for each x

$$= \frac{\left[Q_{\max,empirical}M_c \int_x^\infty \frac{\frac{\pi}{4}x^2}{A}\,d_c(x)dx\right]}{Mc} \tag{A5}$$

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