Immersion vs. Percolation in the Extraction of Oil from Oleaginous Seeds

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ABSTRACT: The influence of the two contact modes, percolation and immersion, during the extraction of oilseeds by means of a solvent are presented. Experiments were performed in lab-scale equipment with soybean flakes, arranged in beds that reproduce these two contact modes. The extractions were carried out with hexane at constant temperature. To simulate the performance of shallow- and deep-bed extractors, two different bed height/diameter ratios were used. The experimental results are explained in terms of the basic transfer phenomena that occur during extraction. These phenomena are addressed to develop a mathematical model, which is used to simulate extraction under both contact modes. The immersion scheme yielded greater efficiency than the percolation mode to extract soybean flakes for the two bed height/diameter ratios studied. The mathematical model predicts very well the experimental findings. It also predicts the solvent retained by the solid mass after extracting the oil.

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KEY WORDS: Contact mode, mathematical model, oilseeds, solvent extraction, solvent retention.

Today, solvent extraction with commercial hexane is used by most of the crushing industry to produce crude oil from oleaginous seeds. Pretreated oilseeds (solid particles) are put in contact with pure solvent or a solvent/oil mixture (liquid) during a time long enough for the oil to be transferred from the solid matrix to the fluid medium in a process unit known as "the extractor." The way solid particles are contacted with liquid depends to a great extent on the internal arrangements of the extractor itself, and it has a significant influence on extraction efficiency (1).

In most continuous commercial extractors, solid particles are placed on a perforated base forming a bed that moves throughout the unit while the liquid flows through the bed in one of two different modes: immersion or percolation. In the case of immersion, liquid is kept on the top of the bed and each extraction stage is flooded with a slow-flowing bath of miscella. In this extraction mode, it is expected that liquid fills the entire space around solid particles (flakes or collets). In the case of percolation, the liquid is sprayed onto the bed surface at a rate low enough to avoid bed flooding. Some commercial extractors combine both types of contact arrangements (2,3).

The solid–liquid contact scheme used in a given extractor is closely related to bed depth and pretreatment of oilseeds. Immersion mode is frequently used in deep-bed extractors, i.e., those extractors with bed heights greater than 1 m or total extractor length-to-bed height ratio lower than 20. This is the preferred contact scheme when the solid particles are in the form of flakes owing to the low porosity of the resulting bed, which makes it strongly resistant to percolation. When expanded material (¨collets¨) is used, deep-bed extractors can be operated either in the immersion or the percolation mode. On the other hand, the percolation mode is frequently used in shallow-bed extractors, i.e., those extractors with bed heights lower than 1 m or total extractor length-to-bed height ratio greater than 20, when solid particles are either in the form of flakes or collets (4). Inside the extractor itself, the extraction process is carried out in stages arranged in such a way that solid particles and liquid move countercurrently (5).

The theory of this operation has been analyzed by several authors (6,7). Different mathematical models, which usually result from mass balance equations written for each intervening phase, have been considered to represent the extraction process. Because of the complexity of the involved mechanisms, simplified equations are often proposed. In some models, each stage is considered as a discontinuous extraction carried out in a well-mixed tank, where the resistance to mass transfer in fluids outside the particle is dismissed. In other models, each stage is considered to be a fixed bed of particles. The complete performance of the extractor results from the integration of the results obtained in the different stages (8,9).

Although a fixed-bed model (10) has been developed among the specific applications for vegetable oils, it does not account for the resistance to mass transfer inside the particle. Some models recently proposed take into account internal resistance (11), but none of them considers an unsaturated bed working under percolation mode.

The aim of the present study was to analyze the influence of the contact mode on the extraction efficiency. A theoretical model based on phenomena that occur during extraction in both immersion and percolation schemes was developed. The extraction efficiency and solvent retention predicted by the model were compared with measured data.

MATHEMATICAL MODEL

The model developed in this work represents a single extraction stage as a fixed bed. This one-dimensional and unsteady-state model considers that a stage of the extraction process takes

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place in a vertical column, which is filled with a bed of pretreated oleaginous material on top of which the solvent is added. The solvent percolates down through the void interparticle space, dissolves the oil of the solid matter it finds on its way down, and both solvent and oil exit the bed through the bottom of the column. Scheme 1 shows a fixed-bed scheme with an enlarged view of a particle like those that make up the bed.

A part of the solvent that flows through the bed goes into the particles through their pores, dissolves the oil, and returns to the bulk phase from where it drains from the bed (12). An equilibrium is reached between the solvent in the particle pores and the oil in the particles that is a function of the oil concentration in the pores.

The oil diffuses through the internal pores toward the particle surface. Then it enters the bulk phase by a convective mechanism produced by a concentration difference between the pores and the bulk phase. Finally, the oil is carried away by convective movement to the bottom of the bed. In the bulk phase, an oil concentration gradient develops by the increasing concentration of oil that the miscella obtains as it flows down the bed. This concentration gradient induces diffusive mass transfer of solute in the direction from higher to lower solute concentration, producing dispersive flow from the bottom toward the top of the bed which is in the opposite direction to the convective flow of the miscella.

To develop the mathematical model the following assumptions were made: (i) An extraction stage was considered as a solid bed made of spherical, isotropic porous particles. (ii) The extraction was considered as a mass transfer process between the following phases: a solid phase (insoluble solid matrix), an occluded fluid phase (miscella occluded inside particle pores), and a bulk fluid phase (miscella moving through the interparticle space). (iii) Within the particles, oil diffused through the occluded phase toward the bulk fluid phase. Because of this, an oil gradient concentration was established. At each point, the oil concentration in the occluded phase was considered in equilibrium with the oil in solid matrix, through the relationship:

$$x_s = K x_p \qquad [1]$$

where x_s = oil concentration in solid phase at *r* (radial position within particle), *K* = equilibrium constant, and x_p = oil concentration in occluded phase at same position *r*. (iv) The vegetable oil was considered to be a single component substance. (v) The oil concentration in the bulk phase changed only in the axial direction, i.e., along the bed. (vi) Particle porosity, ε_p, and bed porosity, ε_l, were constant and uniform through the bed. (vii) The temperature was constant throughout the process. (viii) The effect of solid moisture content was considered in the equilibrium constant.

The model results from the mass balance for oil in the occluded and bulk phases. The assumptions above allowed us to write the following equation for the oil concentration inside particles:

$$\frac{\partial x_p}{\partial t^*}=\frac{1}{Pe}\frac{H}{R}A\left(\frac{2}{r^*}\frac{\partial x_p}{\partial r^*}+\frac{\partial^2 x_p}{\partial r^{*2}}\right) \qquad [2]$$

where *R* = particle radius, *H* = bed height, $Pe = \nu \cdot R/D_p$ is Peclet number with D_p = molecular diffusion coefficient, and ν = superficial velocity (volume rate of flow through a unit cross-sectional area of bed), $t^* = \nu \cdot t/H$ = dimensionless time, and $r^* = r/R$ = dimensionless radial coordinate. Constant *A* was given by:

$$A=\left[1+K\frac{\rho_s}{\rho_m}\frac{(1-\varepsilon_p)}{\varepsilon_p}\right]^{-1} \qquad [3]$$

where ρ_s and ρ_m were solid and miscella density, respectively. The conditions imposed for the solution of Equation 2 were:

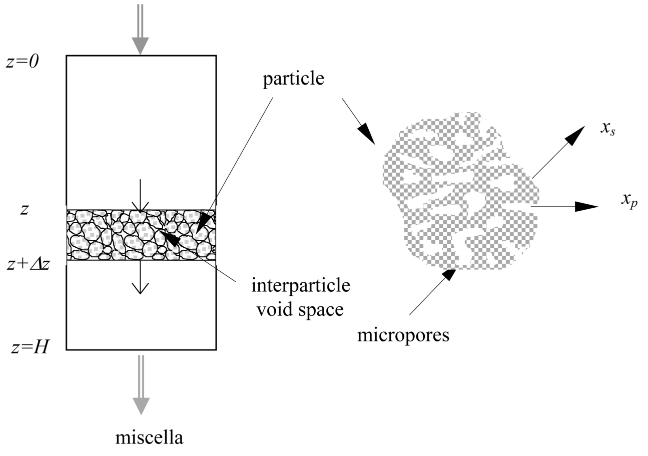
$$\begin{array}{llll} t^*=0 & x_p = x_p^o = \dfrac{x_s^o}{K} & 0 \le r^* \le 1 & \\ r^*=0 & \dfrac{\partial x_p}{\partial r^*}=0 & t^*>0 & [4] \\ r^*=1 & \dfrac{\partial x_p}{\partial r^*}=-Sh\left(x_p\big|_R - x\right) & t^*>0 & \end{array}$$

where *x* = oil concentration in bulk phase, x_s^o = oil concentration in solid phase at initial time, x_p^o = oil concentration in the occluded phase at initial time, and $Sh = k_x \cdot R/D_p$ was Sherwood number with k_x = mass transfer coefficient. After x_p was evaluated from Equation 2, Equation 1 was used to evaluate an average oil content in solid phase as:

$$x_{s,\text{av}}=\int_0^1\left[\int_0^1 3x_s r^{*2}\,dr^*\right]dz^* \qquad [5]$$

To solve Equation 2 under the conditions listed in Equations 4, it was necessary to know the value of *x* that varied along to the axial position and time in accordance to the mass conservation equation for the oil in bulk phase. It was given by:

$$\frac{\partial x}{\partial t^*}+\frac{\partial x}{\partial z^*}=\frac{1}{Pe_z}\frac{\partial^2 x}{\partial z^{*2}}+St\frac{(1-\varepsilon_l)}{\varepsilon_l}(a_{\text{eff}}H)\left(x_p\big|_{r^*=1}-x\right) \qquad [6]$$



SCHEME 1

with the following conditions:

$$\begin{array}{llll} t^* = 0 & x - x^0 & 0 \le z^* \le 1 & \\ z^* = 0 & x\Big|_{z^*=0} = \dfrac{1}{Pe_z}\dfrac{\partial x}{\partial z^*}\Big|_{z^*=0} & t^* > 0 & [7] \\ z^* = 1 & \dfrac{\partial x}{\partial z^*}\Big|_{z^*=1} = 0 & t^* > 0 & \end{array}$$

where $z^*= z/H$ = dimensionless axial coordinate, a_{eff} = effective solid–fluid contact area per unit volume of bed, $St = k_x/\nu$ = Stanton number, and $Pe_z = \nu \cdot H/D_z$ was the Peclet number with D_z = axial dispersion coefficient.

Model for the immersion contact scheme. In this case, the void spaces in the solid material were taken up by solvent or miscella. The model for this scheme results from the application of Equations 1–7, where the effective solid–fluid contact area per unit volume of bed was given by:

$$a_{\text{eff}} = a_p(1 - \varepsilon_l) \qquad [8]$$

and a_p was the specific surface defined as total particle surface per unit volume of particle.

Model for the percolation contact scheme. When the extractor works under the percolation scheme, the void space in the bed is not completely filled with liquid phase and the effective contact area for mass transfer, a_{eff}, is reduced.

The degree of volumetric saturation, Φ, defined as the ratio of liquid volume to total void volume, depends upon the bed drainage capability and the amount of liquid that constantly flows into it. In order to find a relationship between a_{eff} and Φ, the bed was assumed to be a solid mass perforated by cylindrical pores parallel to the acting acceleration. The pore diameters were defined by a continuous spectrum.

Under the condition of no flux coming into the bed and considering the main intervening forces, a_{eff} were evaluated as (13):

$$a_{\text{eff}}(t) = \int_0^{s^*} \pi s H A_T\, g(s)\, ds + \int_{s^*}^{\infty} \pi s\, y(t) A_T\, g(s)\, ds \qquad [9]$$

where s represented the pore diameter, A_T is bed cross section, $s^* = (4\sigma \cos(\alpha)/\rho\ g\ H)$ was the critical pore diameter, σ being the surface tension and α the solid–liquid contact angle. The term s^* represented the larger pore that was flooded with liquid phase; those pores with diameter $s > s^*$ were partially filled, and the liquid height inside them, $y(t)$, was given by

$$y(t) = \left(1 - \frac{4\sigma\cos(\alpha)}{\rho g b}\right) e^{-\left(b^2 \rho g / 8 \mu H\right)t} + \frac{4\sigma\cos(\alpha)}{\rho g b} \qquad [10]$$

Here, ρ and μ represented the density and viscosity of liquid phase respectively. With time, $y(t)$ decreased and so did a_{eff}.

In Equation 9, $g(s)$ represented the pore-size distribution function. For the bed model described above and using a Boltzmann-type distribution function, $g(s)$ was given in terms of bed permeability, k, and porosity, ε, as (13):

$$g(s) = \frac{s\varepsilon^3}{32\pi k^2} e^{-(\varepsilon/16k)s^2} \qquad [11]$$

Following this model, the volume of liquid retained inside the bed was evaluated as:

$$V(t) = \int_0^{s^*} g(s)\pi \frac{s^2}{4} H A_T\, ds + \int_{s^*}^{\infty} g(s) \frac{\pi s^2}{4} A_T\, y(t) ds \qquad [12]$$

Starting from a saturated bed, liquid drained until an equilibrium was reached. At this point, the volumetric saturation was evaluated as:

$$\Phi(t) = \frac{V(t)}{V(t=0)} \qquad [13]$$

If liquid is fed to the bed at a volumetric rate of Q, then a new equilibrium state will be reached where the liquid flux leaving the bed equals the liquid flux coming into it. So, using the plot of $V(t)$, a point can be found where the slope equals the Q value; i.e,

$$\frac{dV(t)}{dt}\bigg|_{t=t^{eq}} = Q \qquad [14]$$

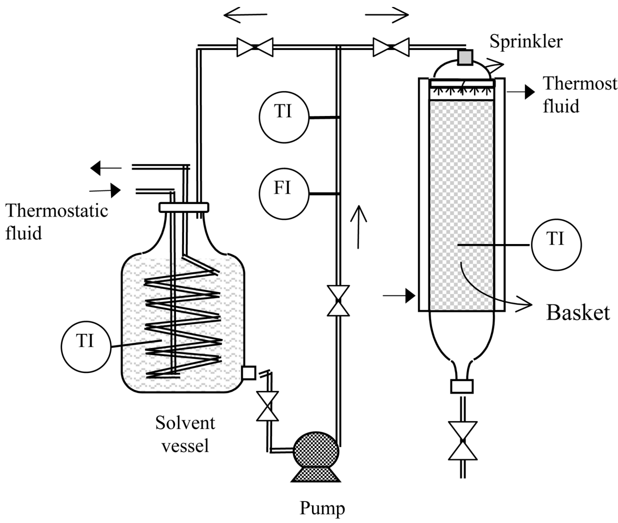
This point represented the equilibrium state under this flow condition. The a_{eff} evaluated at $t = t^{eq}$ was the solid–liquid contact area available for mass transfer and it was used in Equation 6.

Resolution procedure. Equations 2–4, 6, and 7 were solved numerically using the implicit finite difference method with MatLab 5.3 (14). The integration of Equations 5, 9, and 12 were done using Gauss–Legendre quadrature (14). The physical and transport properties involved in the model (ρ, μ, σ, k, D_z, D_p, and k_x) were evaluated using correlations available in the literature (13,15–17).

EXPERIMENTAL PROCEDURES

Extraction equipment. The experimental apparatus was designed to provide a controlled, measured flow rate of solvent to the extractor unit. Scheme 2 shows a schematic drawing of the experimental setup (IT, temperature indicator; FI, flow indicator). The extractor itself was a cylindrical container made of glass (i.d. = 95 mm; height = 600 mm) which allows the insertion of a wire basket to hold the sample. It had an external jacket for circulating hot water to keep the operation isothermal. The temperature of the solid sample was measured by using a thermocouple located inside the basket. The solvent was heated to the extraction temperature by means of a coil heater in the conditioning tank and then was uniformly supplied to the extractor through a spray placed at the top of the basket. Flow rate of solvent, delivered by a centrifugal pump, was adjusted at the set value by using a control valve and a flow indicator.

Upon leaving the conditioning tank, the solvent entered the extraction unit or flowed through a bypass when starting the apparatus.



SCHEME 2

Extracted solid and solvent. The extracted bed was made of soybean flakes with an average thickness of 0.37 mm and moisture content as reported in Table 1. They were provided by a local oilseed crushing industry. It was expected that the mathematical model developed under the assumption of spherical particles will represent the physics of the extraction process even when particles used in experiments were slab-shaped. Expressions similar to Equations 2–4 may be written and solved for any regular particle shape, parallel-sided slab among them (7). The resulting expressions were less convenient than the simple form for spherical particles. Because of this, it is a common practice to use spherical particle expressions with an equivalent particle radius defined as the radius of the sphere having the same surface to volume ratio. This approach was used. The validity of this approximation has been explored and shown to be satisfactory to describe the adsorption process in single particles (18) within the time limits used in this work. The solvent used was technical-grade hexane.

Experiments. The contact schemes analyzed were (i) immersion and (ii) percolation. In both cases, the solvent flow through the bed was kept continuous during the test. In each trial, the following procedure was followed: (i) Solvent (in a storage tank), a given amount of solid material (in the extraction basket), and the extraction unit were simultaneously heated to the extraction temperature, T_{ex}. (ii) Once at T_{ex}, the extraction unit was filled with solvent to a level that initially flooded the solid sample. (iii) At this point (considered as $t = 0$), the basket was introduced into the extraction unit to contact the solvent with solid material. At the same time, the solvent sprayed on the top and the miscella exited from the bottom of the equipment. (iv) In case (i), the fluid level was regulated by the discharge valve so as to maintain a constant level during the trial. In case (ii), the discharge valve was totally open and solvent flow similar to that of the case (i) was sprayed on top of the bed. This procedure ensured the same initial conditions in cases (i) and (ii), rendering both experiments comparable. (v) The extraction was run for a time period, t_{ex}. During this time, samples of miscella exiting from the extraction unit were taken and their oil concentrations determined. (vi) When the extraction time was over, the solid sample was allowed to drain for a predetermined time period, t_{dr}, taken out of the basket, weighed, and air-desolventized.

Oil content in raw material, m_b, and in solid sample after extraction, m_a, were measured and used to compute extraction efficiency, %EE, as

$$\%\mathrm{EE} = 100\frac{(m_b - m_a)}{m_b} \qquad [15]$$

On the other hand, %EE was computed from predicted $x_{s,\mathrm{av}}$ as

$$\%\mathrm{EE} = 100\left[\frac{x_{s,\mathrm{av}}(t=0) - x_{s,\mathrm{av}}(t=t_{\mathrm{ex}})}{x_{s,\mathrm{av}}(t=o)}\right] \qquad [16]$$

The solvent retained by the solids after extraction was evaluated by defining the retention percentage, %RS, as

$$\%\mathrm{RS} = 100\frac{(m_e - m_d)}{m_d} \qquad [17]$$

where m_e = weight of solids after extraction and m_d = weight of solids after desolventizing. The solvent retained was related to the volumetric saturation, Φ, through the following relationship:

$$\%\mathrm{RS} = 100\frac{\varepsilon}{1-\varepsilon}\frac{\rho_h}{\rho_s}\Phi(t_{\mathrm{dr}}) \qquad [18]$$

where ρ_h = hexane density and ε = total bed porosity, given by

$$\varepsilon = \varepsilon_l + (1-\varepsilon_l)\varepsilon_p \qquad [19]$$

TABLE 1
Comparison Between Experimental and Theoretical Results (solvent flow: 0.7 L/min)

					Experimental results		Model outputs	
Bed height	Contact mode	%RS	Moisture content[a]	Initial oil content[b]	Residual oil[b]	%EE	Residual oil[b]	%EE
Deep bed	Immersion	27.06	8.48	22.96	3.69	83.93	3.64	84.15
Deep bed	Percolation	26.61	8.48	22.96	5.65	75.40	6.41	72.08
Shallow bed	Immersion	21.62	8.98	19.88	4.57	77.01	2.57	87.07
Shallow bed	Percolation	21.69	8.98	19.88	5.86	70.52	5.32	73.24

[a] Expressed as 100 × kg water/kg total.
[b] Expressed as 100 × kg oil/kg total. %RS, retention percentage of solvent retained by solids; %EE, extraction efficiency.

Analytical determinations. The oil content of the solids was determined according to AOCS method Ac 3-44 and the moisture content according to AOCS method Ac 2-41 (19). The miscella concentration was determined by evaporating the solvent to constant weight. The total bed porosity was determined according to the method proposed by Lozano *et al*. (20).

RESULTS AND DISCUSSION

Experiments were done by putting different amounts of solid material in the extraction basket to simulate the behavior of shallow- and deep-bed extractors. In the first case, about 0.2 kg of solids was used, producing a bed 0.10 ± 0.02 m high. To represent a deep-bed extractor, 0.8 kg of solids (0.34 ± 0.02 m bed height) was extracted. In all cases, solvent flow was kept between 0.5 and 1.0 L/min, drainage time was t_{dr} = 10 min, and extraction temperature and time were T_{ex} = 50°C and t_{ex} = 10 min, respectively.

Figure 1 shows experimental values and model predictions of the oil concentration in the miscella leaving the bed as a function of time for both immersion and percolation contact schemes in a deep-bed arrangement. Parameter values used in the simulations are shown in the Figure caption. From these results it is concluded that the immersion mode produced higher miscella concentrations all along the extraction period. This trend can be explained by the higher contact area in immersion as compared to percolation contact.

Model predictions of oil concentration in bulk fluid phase, x, as a function of position in the bed at different times during the extraction period are shown in Figure 2 for a deep bed in immersion mode. The oil concentration increased as the miscella moved from the inlet down to the outlet of the bed. The x values at $z^* = 1$ were used as the model output in Figure 1. Figure 3 depicts the average oil concentration in solid particles, $x_{s,\mathrm{av}}$, (see Eq. 5) during the extraction period, under the same conditions as in Figure 2. Here again, as in Figure 1, immersion mode produced better results than percolation mode.

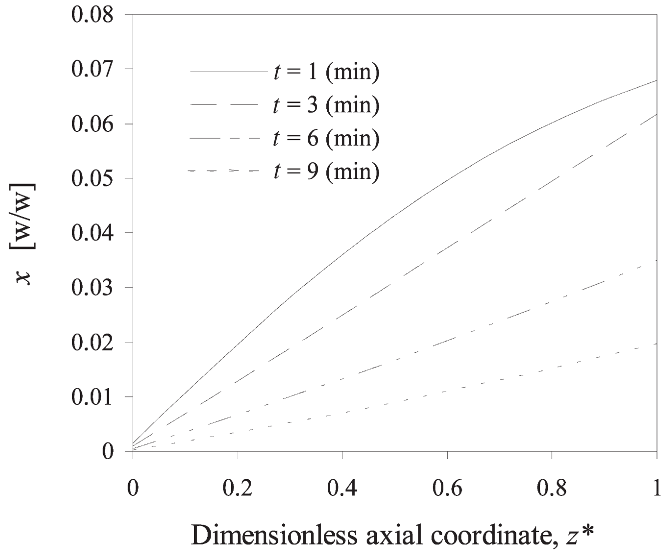


FIG. 2. Oil concentration in bulk fluid phase as a function of dimensionless bed height at different times during the extraction period. Deep-bed arrangement and immersion mode were employed. The solvent flow and model parameters were the same as in Figure 1.

Data for %RS, measured at different drainage times, were transformed using Equation 16 and compared to predicted Φ values in Figure 4. Predicted and measured data were in good agreement. After a quick decrease, Φ leveled off to an asymptotic value. This behavior was used to set the drainage time in t_{dr} = 10 min since there is not a noticeable change in solvent retention after that time. The experimental %RS values shown in Figure 4 were measured after the extraction of a deep bed under percolation mode.

Table 1 gives the results for one group of experiments. The table shows values of solvent retention, residual oil, and

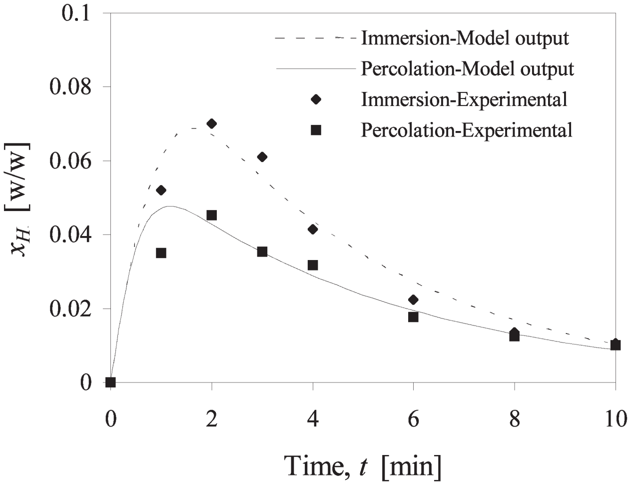


FIG. 1. Oil concentration at bed outlet in a deep-bed arrangement. Solvent flow: 0.59 L/min; model parameters: ε_p = particle porosity = 0.3; ε_l bed porosity = 0.5; D_p = molecular diffusion coefficient = $9.33 \cdot 10^{-10}$ m^2/s; D_z = axial dispersion coefficient = $9.86 \cdot 10^{-6}$ m^2/s; k_x = mass transfer coefficient = $1.42 \cdot 10^{-5}$ m/s; K = equilibrium constant = 0.6; x_H = oil concentration in the miscella leaving the bed.

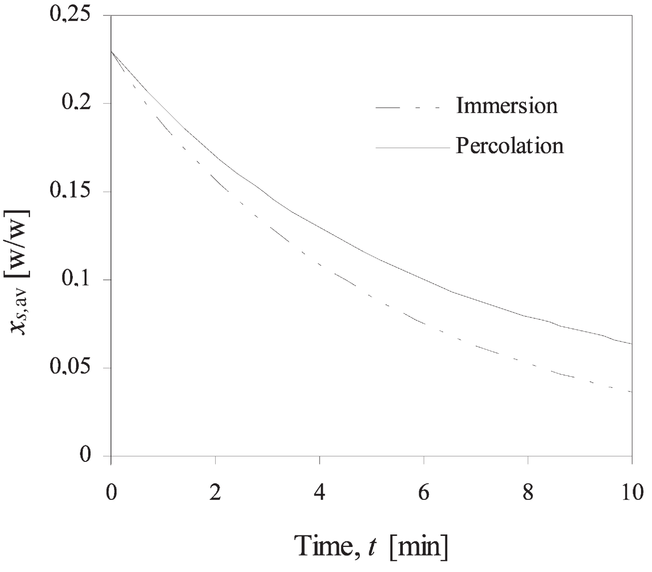


FIG. 3. Average oil concentration in the solid phase ($X_{s,\mathrm{av}}$) as a function of time. Solvent flow and model parameters are the same as in Figure 1.

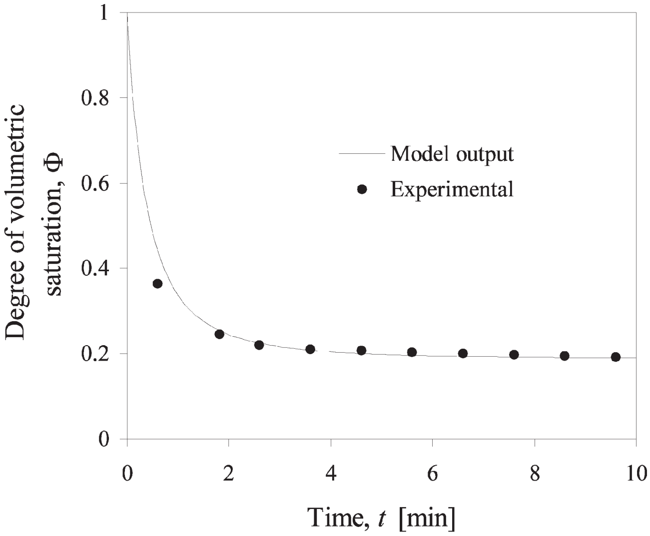


FIG. 4. Volumetric saturation Φ as a function of drainage time. Bed height: 0.20 m. Model parameters: ε = porosity = 0.65; σ = surface tension = $20.9 \cdot 10^{-3}$ N/m; k = bed permeability = $1.3 \cdot 10^{-6}$ m^2; cos(α) = 0.7, where α = solid–liquid contact angle.

extraction efficiency on both immersion and percolation modes for shallow and deep beds. Values of these parameters obtained from experimental measurements and model computations were compared. Differences in extraction efficiency between immersion and percolation mode for two replicates of each experiment have been analyzed statistically with Student's *t*-test using a paired design (21). This analysis is shown in the Table 2. The results indicated that the immersion scheme was better (at 5% significance level) than percolation under the conditions of deep bed, but there was not a significant difference (at 5%) in the case of shallow bed. Since the model outputs showed a good agreement with the experimental values, the mathematical model developed in this study closely represented the complex phenomena taking place during the extraction process. If the right parameters are used, it is expected that this model can be used to analyze the extraction of solid particles other than flakes (for example, collets) under conditions different from the ones used in this study.

TABLE 2
ANOVA on the Experimental Results

Experiments	Differences in %EE (immersion – percolation)	Significance testing[a] at 5% level
Deep bed—Test 1	8.96	Significant
Deep bed—Test 2	8.53	
Shallow bed—Test 1	6.49	Not significant
Shallow bed—Test 2	0.07	

[a] Paired comparison design using *t*-distribution.

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