Full Length Research Paper

Modeling the kinetic of solute diffusion from sugarbeet particles based on electric conductivity measurements

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The aim of the present work is to validate a method based on electric conductivity measurements to monitor and analyze the kinetics of solute transfer at ambient temperature from agro-food solids (sugar beet particles) into a liquid phase (water). The first step consists of studying the correlation between two simultaneous measurements, that is, concentrations and electric conductivity of particles bed. A linear correlation could thus be revealed and it confirmed the correspondence of these two types of measurements. In fact, a close agreement is obtained between the normalized forms of the concentration and conductivity; the normalized parameters were then used to estimate and compare the kinetic parameters (e.g. k (min^{-1}) or D ($m^2.min^{-1}$)) of solute transfer. Close correspondence could be obtained between the kinetic parameters deduced from the normalized forms of the concentration and conductivity. Thus, using simple electric conductivity measurements may replace concentration ones which simplify the study of the kinetics of soluble matters extraction. Since electric conductivity measurements are quick, precise, and non-intrusive, this leads to gain in time and in number of repetitions when studying the kinetics of solvent solid-liquid extraction.

Key words: Kinetics solute transfer, Fick's law, mass transfer, concentration, electric conductivity, diffusion coefficient.

INTRODUCTION

Solvent solid-liquid extraction is a major agro-food unit operation by which mass transfer is obtained by contacting solid and liquid phases having different concentrations. In practice, it can either be studied on laboratory or pilot scales. In any case, the kinetics of solute or mass transfer is generally studied based on concentration measurements.

A significant number of research works can be found in the literature dealing with the study of mass transfer phenomenon form agro-food particles (Bruniche-Olsen, 1962; Schwartzberg and Chaos, 1982; Roques, 1987; Gekas, 1992; Jemai and Vorobiev, 2003, 2002; El-Belghiti et al., 2005); generally, from these particles one can extract various types of soluble matters using a solvent such water (solid-extraction) or inversely solute

can be added to them (osmotic dehydration) (Roques, 1987; Gekas, 1992). For instance, solid liquid extraction in the sugar industry aims at extracting the maximum of dissolved matter (principally sucrose if not all). This is obtained by diffusion during contact between sugar beet tissue (solute rich) and water (initially without soluble). Successful implementation and/or enhancement of such an operation on industrial scale most often require studying the phenomena on smaller scales (laboratory or pilot) (Jemai and Vorobiev, 2003; El-Belghiti et al., 2005). Usually, the kinetic of solute transfer is monitored by concentration measurements of the solid or liquid phases and necessitates a certain number of runs in order to sufficiently characterize the extraction process. In all cases, either the transfer rate (k in s^{-1}) or diffusion coefficient (D in $m^2.s^{-1}$) are the main parameters by which the diffusion kinetics could be characterized.

The present study concerns a validation of a conductimetric method for studying the kinetic of solid-

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liquid extraction. It is a follow-up study related to a previous published work dealing with the study of pulsed electric field (PEF) on the leaching process from sugar beet particles (Jemai and Vorobiev, 2003). Firstly, a correlation between simultaneously measured values of solute concentration in the solid phase and electrical conductivity of the particles bed is obtained. The normalized parameters based on both types of measurements would then be compared and used to obtain the kinetic parameters.   As in a number of published studies, the electrical conductivity can be effectively used for non destructive testing and measurements in wide range of applications such as in chemistry, biology and soil science (Schmidt, 1972; Jemai and Vorobiev, 2002, 2003; Samouëlian et al., 2005; Bozkurt et al., 2009; Ozcep et al., 2010).

In fact, this is an indirect method for monitoring the kinetic of solute transfer that would be followed continuously without interrupting test runs; by doing so, one could perform a significant number of runs for a shorter period of time compared to discontinuous experimentations. In a previous work (Khezami et al., 2010), a similar method in principle has also been validated for studying the kinetics of drying by continuously monitoring the changes in humidity of the convection drying air as an indirect means to follow the mass loss.

MATERIALS AND METHOD

Raw material

Sugar beet tubers have been generously provided by the sugar factory (Béghin Say, France) and stored in a cool environment of less than 4°C. Control of average properties of the raw material prior to test runs allowed the data in Table 1 to be obtained.

Sugar beet particles (grating type) are obtained using a domestic grating instrument. The dimensions and geometry of the particles are described by Figure 1. During each run, the obtained particles are separated into two lots: (i) One for extraction, and (ii) One for initial composition control.

Experimental setup

The experimental setup is shown in Figure 1, has been described by the current main author in a previous work (Jemai and Vorobiev, 2003). It mainly comprises a solid-liquid bed placed between two electrodes for electrical treatment and continuous conductivity measurements; so that, both concentration and electrical conductivity values could be simultaneously recorded and later correlated. The setup is in fact composed of a polypropylene casing (1) in which two electrodes (2) separated by a distance of 1.60 cm, are connected to a PEF pulse generator (1000 V-20 A) (3); a peristaltic pump (4) continuously delivers distilled water from an overhead container (5) through the extraction chamber. Concentration measurements were based on Brix (g/ 100 g) values obtained using a digital hand refractometer (ATAGO PR-101, ATAGO Co., Japan). A data acquisition protocol via an HPVEE program (HP-VEE, V3.12, Hewlett-Packard Co., USA), ensures generator control for PEF application and electrical conductivity measurements.

Table 1. Properties of sugar beet raw material.

Property	Typical value
Density (g/cm^3)	1.05
%Brix (g/100g of juice)	20
Water (% weight)	77
Soluble solids (% weight)	19.25
Insoluble solids (% weight)	3.75

Experimental procedure

At least 15 repetitions have been performed and each run consisted in placing 20 g of grated sugar beet particles inside the extraction chamber (between two electrodes), thus forming what is termed a fixed particles bed.

The concentrations of the solid phase during time were obtained based on accumulated juices at regular time interval following control of the °Brix (g solubles/100 g juice); °Brix was measured using a digital refractometre (ATAGO PR-101, ATAGO Co., Japan). The electrical conductivity of the bed was based on continuous impedance measurements at low non-destructive voltage (that is, U < 3V).

The electrical conductivity, σ, is related to the electric current, I, the applied voltage, U, and geometry of the sample (cross section,

$$\sigma = \frac{e \cdot I}{A \cdot U} = \kappa \cdot \frac{I}{U}$$

A, and thickness, e, of t he bed) by :   (Jemai and Vorobiev, 2002).

Data analysis method

As mentioned previously, the main parameters used for the purpose of this study are the solid phase solute concentration and the electrical conductivity of the solvent-particles mixture bed. As a first step, electrical conductivity measurements were plotted as a function of the corresponding concentration; Figure 2 reveals a linear dependency between the normalized parameters C^* and σ^*. This linear correlation can be written as:

$$\sigma/\sigma_0 \approx \alpha \cdot C/C_0 + \beta$$

(1)

So, the normalized parameters would be approximately equivalent:

$$\sigma^* = \frac{\sigma(t) - \sigma_\infty}{\sigma_0 - \sigma_\infty} \approx \frac{C - C_\infty}{C_0 - C_\infty} = C^*$$

(2)

Note that the initial and ultimate values of the conductivity and concentration (C_0, C_∞ and σ_0, σ_∞) must be estimated in order to obtain the normalized parameters. For instance, the initial values are measured in the start of the experiments, while the ultimate conductivity of the bed would approach that of the distilled water used (~ 5 to 10 μS/cm). This value can be used to estimate the equilibrium normalized concentration.

The kinetic parameters (that is, the transfer rate, k and the apparent diffusion coefficient, D_{app}) can be obtained as a first estimate by the slopes of the lines of $\ln(C^*)$ and $\ln(\sigma^*)$ versus time curves. So that the slope is in fact equal to (k) and could further be used to estimate the value for (D_{app}) if the characteristic thickness (l) of a given particle is precisely known.

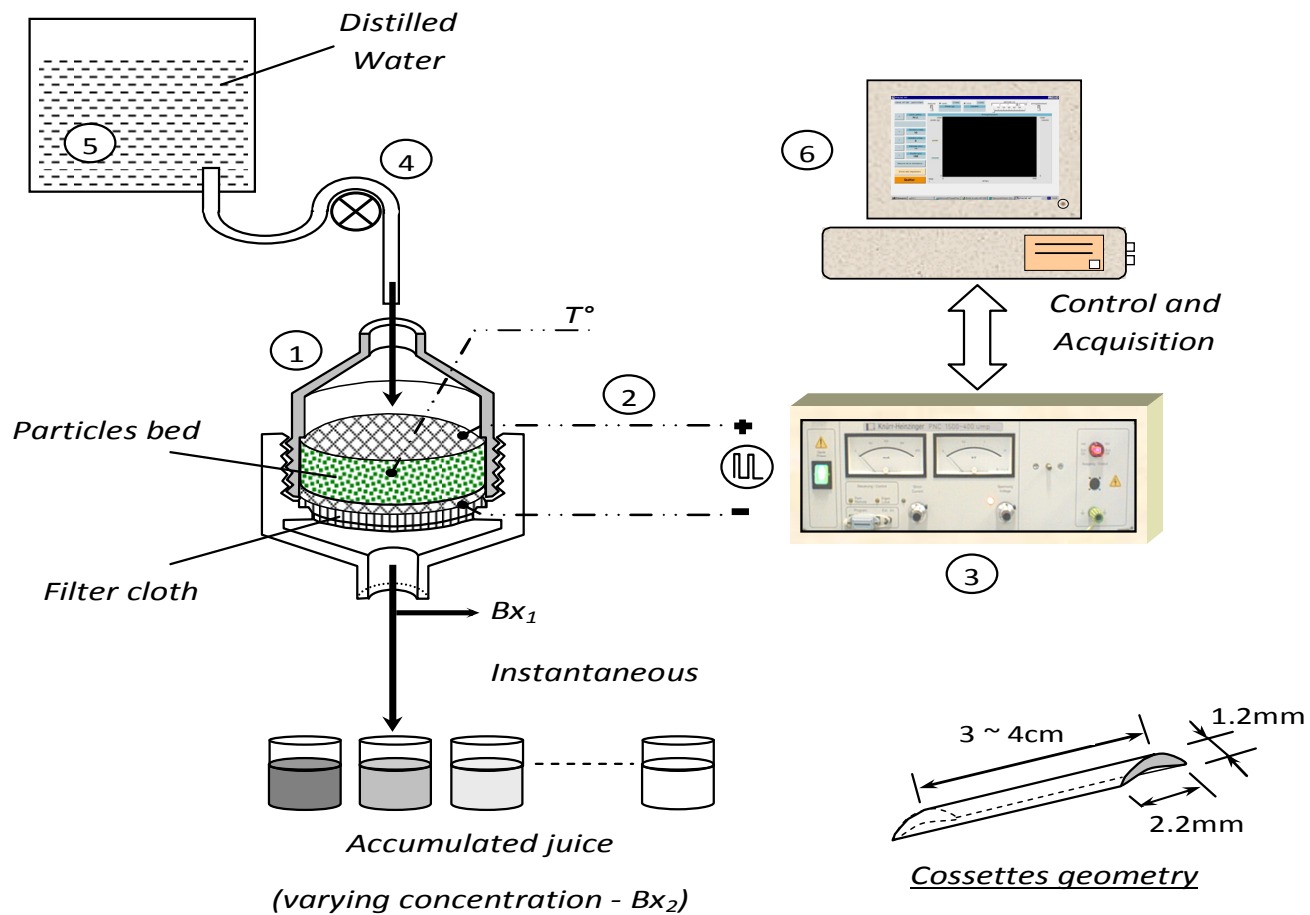In this case, the one-term Fick's law average solution for either

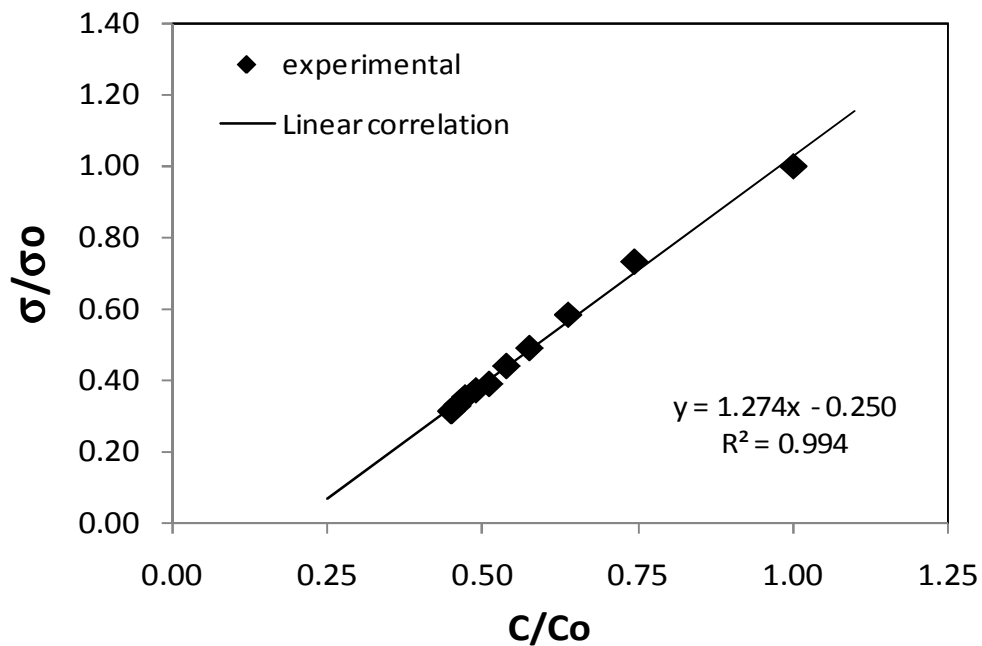**Figure 1.** Experimental setup (Jemai and Vorobiev, 2003).

Figure 2. Correlating between conductivity measurements and concentration ones.
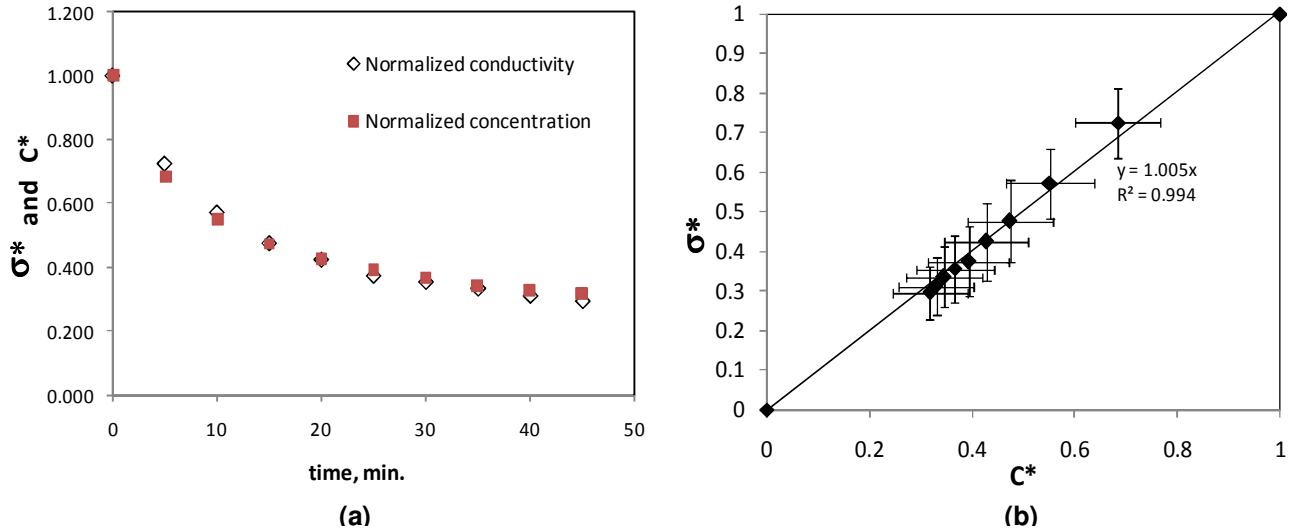
Figure 3. (a) Kinetics of solute transfer during time; (b) correlating between normalized conductivity measurements and concentration ones.

Table 2. Summary curve fitting results using Matlab® (V7.6-R2008a).

Base parameter	a_1	95% confidence bounds	k (10^{-3} min^{-1})	95% confidence bounds	R^2
Concentration	0.566	(0.523, 0.610)	13.75	(11.11, 16.39)	0.973
Electrical conductivity	0.588	(0.533, 0.644)	16.12	(12.83, 19.4)	0.970

($\overline{c}^*$) or ($\overline{\sigma}^*$) would be:

$$\overline{\sigma}^* \approx \overline{c}^* \approx a_1 e^{-\lambda_1^2 \frac{D_{app}^{(1)} \cdot t}{l^2}} \tag{3}$$

And so the slopes (k) of $ln(\overline{\sigma}^*)$ or $ln(\overline{c}^*)$ versus time curves, can be used to estimate the diffusion coefficient by:

$$D_{app}^{(1)} \approx \frac{k \cdot l^2}{\lambda_1^2} \tag{4}$$

Note that, when convection resistance is negligible (that is, sufficient agitation, particles sufficiently spaced so that no blockage occurs, etc...) λ_1 takes on the value of $\pi/2$; from analysis of the solution to Fick's law of diffusion (Crank, 1975; Schwartzberg and Chaos, 1982), λ_1 is in fact the first root of the equation $\lambda tan(\lambda) = Biot$ when the number of *Biot* is sufficiently high (e.g. infinite). As can be depicted from Equation 4, precision on (*D*) estimates will further depend on the precision of the characteristic thickness (*l*) in addition to the previous parameters (*k* and λ_1).

RESULTS AND DISCUSSION

Figure 2 shows a plot of the electrical measurements as a function of concentration ones for at least 15 repetitions (to account for variability amongst raw samples; this is given in terms of ratios for each run and each given raw sample). Clearly, these two parameters are linearly correlated an observation that led to the idea that they can be used interchangeably.

As shown in Figure 3a, the kinetics of solute transfer from both types of measurements are almost the same, which would lead to equivalent kinetic parameters.

Figure 3b gives that plot of (σ^*) as a function of (C^*) showing standard deviation bars based on at least 15 runs.

The kinetic parameters k and D may then be computed (as a first hand estimate) by combining the experimental data and Equation 3. Table 2 gives the estimates for k and D, respectively.

It should be noted that the obtained parameters were based on the kinetics for times starting from 15min; this time lag actually accounts for establishing the flow regime across the particles bed. In a previous study (Jemai and Vorobiev, 2003), it has been found that the established regime was indicated by a linear correlation between the

Fourier number ($Fo = \frac{D_{app} \cdot t}{l^2}$) and time of extraction starting approximately from 15 min; thus, despite some blockage between particles filling the bed, adequate mixing was ensured around any given cossette by the constant renewal of fresh water which would eventually

cause the exhausting of solubles from cossettes after an infinite time of extraction.

From the estimated values of (k), the apparent diffusion coefficient computed by Equation 4 takes on the values 3.34×10^{-11} and $3.92 \times 10^{-11}\, m^2/s$ based on concentration and electrical conductivity measurements, respectively. This order of magnitude concerns in fact, the apparent diffusion coefficient of freshly cut, non-treated samples at ambient temperature. According to Brüniche-Olsen (1962), the average apparent diffusion coefficient for completely thermally denatured sugar beet tissue, having characteristic thickness between 1 to 2 mm, is about 16 to $21 \times 10^{-11}\, m^2/s$; this explains why it takes significantly lesser time for denatured tissue to be exhausted at ambient temperature, while for untreated samples, intact cell walls presents a natural barrier for solute transfer at this same temperature.

Precision of estimates for the kinetic parameters could further be insured via more elaborate solution of the diffusion equation and considering more exponential terms to model the extraction kinetic. Furthermore, convection resistance issues may also be addressed in order to estimate the diffusion coefficient more precisely.

Changing the operating conditions to study the effect of solvent temperature or particles size could also be considered in future works provided that the effect of temperature on the electrical conductivity measurements is accounted for to adequately monitor the kinetics of solute transfer by electrical conductivity. This work deals in fact, with non-thermal extraction of soluble matters and the possibility to obtain such effects by subjecting the samples to PEF treatments during extraction.

Conclusion

By simultaneously measuring the concentration and electrical conductivity of a sugar beets particles bed, it has been found that these two parameters are linearly correlated; so, in normalized forms, they would reflect the same kinetics of soluble transfer during solvent solid-liquid extraction form agro-food particles. This has been confirmed based on a significant number of runs (at least 15 repetitions) and showed that the kinetic parameters are fairly close (within usual agro-food standard variability of <20% deviation).

Since electrical conductivity measurements may be quick, easy, and reliable, they allow adequate study of the kinetic of soluble transfer without loss of precision; in addition, this can be useful when interruption of the measurements is undesired or lead to time consuming runs. Thus, non-thermal extraction of soluble matters form PEF treated samples during extraction may be continuously and uninterruptedly monitored by electrical conductivity measurements.

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