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Aqueous extraction and separation of linseed press-cake components enhanced by high voltage electrical discharges

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Industrially oil is produced from seeds after crushing, cooking, solid-liquid expression and eventually a solvent extraction step. The objective of this study is to extract the oil from linseed (40 % oil) with an aqueous process in which organic solvent use would be excluded.

After solid-liquid expression, the press-cake was crushed and mixed with demineralized water in cake/water ratio of 1/10. High voltage electrical discharges (HVED) (40 kV, 10 kA, 3 μ s) were applied to this mixture at a frequency of 0.5 Hertz. The HVED consumed energy was calculated from electrical voltage and current curves. The measurements of electrical conductivity, Brix, pH and viscosity of the liquid phase allowed to characterize the kinetic of cake extraction at different intensities of electric field.

Keywords: oilseed meal, expression, mucilage, protein, oil

Introduction

Flax plant has been grown since the beginnings of civilization; it was already used in old Egypt at the time of the Pharaohs. There are two types of cultivated flax (*Linum usitatissimum*): one is grown for the seed and the other for fiber production.

The whole seed or the oil have been used for edible purposes in Europe and Asia. Nowadays in Europe, the use of linseed oil in food depends of the country: for instance in France, its use is forbidden since 1908 by Order in Council. In North America, whole seeds are used in health foods. Linseed oil is rich in unsaturated fatty acids with single or double bonds. Linseed oil is classified as a drying oil and therefore has a variety of surface coating applications such as paints, stains or linoleum. Nowadays linseed oil is mainly used in industrial applications even if there is a recent renewal of interest for the interesting nutritional properties of oil and seed [1; 2]. Linseed is flat, oval with a pointed end. It has the average dimensions of 2.5 x 5 x 1 mm. The seeds weight range from 3 to 13 mg per seed and have a yellow to brown color [3]. Linseed contains 35 to 45 % of oil and 10.5 to 31 % of protein [4]. Its hull, which accounts for 30 to 39 % of its weight, contains little protein and oil and comprises four layers. The outer layer contains mucilage, an heterogenic polysaccharide mixture [5]. Mucilage accounts for about 8 % of the seed weight [6] but it depends on extraction regimes and can range from 3.5 to 9.4 % [7]. In aqueous solution, mucilage exhibits high solubility, good foam stability and increases the viscosity; it could be a substitute for usual gums used as food industry [8].

In the industry, linseed oil is obtained from the seed by cleaning, crushing into flakes, cooking and either by continuous expression (pressed oil, presscake) with a screw press or by expression followed by solvent extraction (extracted oil, defatted cake). Linseed presscake is mainly used in animal feeding.

Because of present ecological issues, industrials will have to reduce or even abandon solvent use. Therefore non-solvent process is one of our objectives. Aqueous extraction processes have been already studied to produce different oils. It allows to recover oil and protein simultaneously. There are many different ways of mucilage aqueous extraction from the seed. Some authors have tested hot extraction [8-10]. Optimum conditions have been determined with an experimental design: a temperature of 85-90°C, a pH 6.5-7.0 and a 13/1-water/seed ratio [9]. Others have soaked whole seeds in water or sodium bicarbonate solutions (0.05 and 0.10 M for 6 and 12 h) and have used polysaccharide degrading enzymes [11]. There are two steps in the extraction of the mucilage: first an hydration of the seed and mucilage, then a dissolution of the mucilage [12]. Another way to enhance mucilage extraction is the use of high voltage electrical discharges (HVED). HVED in water produce ultraviolet radiations, active species [13] and shock waves [14]. They may have different applications such as degradation of organic compounds contained in water [15], micro-organisms inactivation [16], or extraction of soluble material [17-19]. This treatment seems to have no effect on the quality of proteins extracted with this method [17]. As the functionalities of proteins must be preserved (temperature constraint), linseed press-cake, which is produced with a non-thermal process [19], is treated with electrical discharges to extract the different components. The evolution of the different characteristics of the solid/emulsion system, the emulsion and the solid residue is the aim of the present study.

Materials and methods

Production of the press-cake

Licensed cultivar of flax Barbara produced in 2001 (Lecureur S.A., Artenay, France) was used in the study. Seeds were protected from the light and stored at ambient temperature in paper bags. Seeds were crushed with a three-cylindrical roll crusher (Grenier-Charvet, Saint-Gilles, France). The seed powder was then pressed with a thermo-regulated hydraulic press. As protein functionalities must be preserved, the temperature was fixed at 50°C. The pressure of 12 MPa was used in accordance with the press capacity. Crushing conditions were optimized in a previous work in term of oil yield obtained after a one-hour expression [19]. The composition of the obtained linseed press-cake is given in table 1. The linseed press-cakes were stored at ambient temperature in plastic bags.

	Seeds	Press-cake
Protein	14	20
Oil	40	13
Solid	38	55
Moisture	8	12

Table 1. Composition of seeds and linseed press-cake.

Discharge generator

The HVED generator (fig. 1) (Tomsk Polytechnic University, Russia) can provide 40 kV/10 kA discharges during a few microseconds in a one-liter chamber. This generator can be used to obtain damped oscillations. Voltage sensor (ROSS VD45-8.3-A-K-A, Ross Engineering Corporation, Campbell, CA, USA) and intensity sensor (PEARSON 3972, Pearson Electronics

inc., Palo Alto, CA, USA) are related with a 10^8 Hz sampling system via an oscilloscope (Tektronix TDS1002, Beaverton, Oregon, USA) and an acquisition central (HPVEE 4.01, Hewlett-Packard, Palo Alto, California, USA). Electrical discharges were applied with a 0.5 Hz frequency, which is imposed by the generator. The distance between the electrodes was fixed in this study at 5 mm.

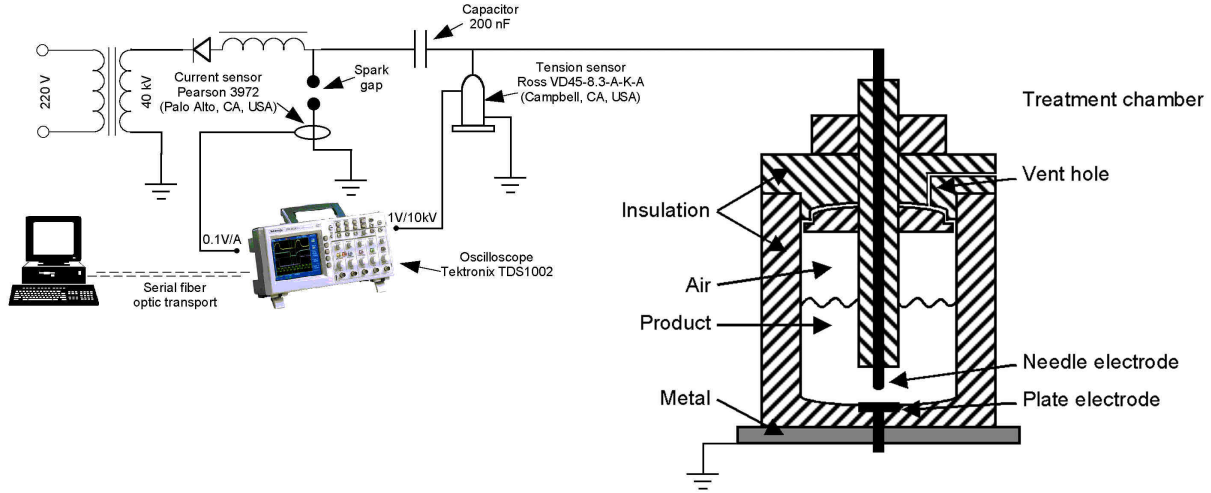


Figure 1. Schema of the generator.

The electrical discharges (fig. 2) are generated by electrical breakdown in water. Energy is stored in a set of low-inductance capacitors, which are charged by a high-voltage power supply. Two stainless steel electrodes are placed inside a cylindrical chamber, which is filled with de-ionized water. In order to obtain breakdown between the two electrodes, the maximum value of the applied electric field E_m should exceed the breakdown electric field E_{br} ($E_m > E_{br}$). One can estimate E_m ($\text{V}\cdot\text{m}^{-1}$) [20] as:

$$E_m = 0.9 \left(\frac{U_0}{d} \right) \frac{r+d}{r} \quad (1)$$

where U_0 is the applied voltage (V), r is the electrode radius (m) and d is the distance between the electrodes (m).

The breakdown electric field E_{br} ($\text{MV}\cdot\text{cm}^{-1}$) can be estimated by the use of Martin's semi-empirical formula [21]:

$$E_{br} = k \cdot S^{-0.1} \cdot t_{eff}^{-1/3} \quad (2)$$

where k is a constant ($\text{MV}\cdot\mu\text{s}^{1/3}\cdot\text{cm}^{-0.8}$), S is the area of the electrode (cm^2) and t_{eff} is the effective time of the applied voltage (μs) (fig. 2).

The application of high voltage across the electrodes leads to formation of streamers propagating from the positive electrode to the negative one. When one of the streamers reaches the negative electrode, fast formation of a discharge channel occurs. This discharge channel, which is then called leader, is characterized by a plasma with high conductivity (due to the large plasma electron temperature and density). Also, the large plasma density and temperature gradients, together with the non-elastic properties of the liquid, lead to the formation of shock waves. In addition to the shock wave formation, intense light emission due to collision radiative processes inside the plasma is produced.

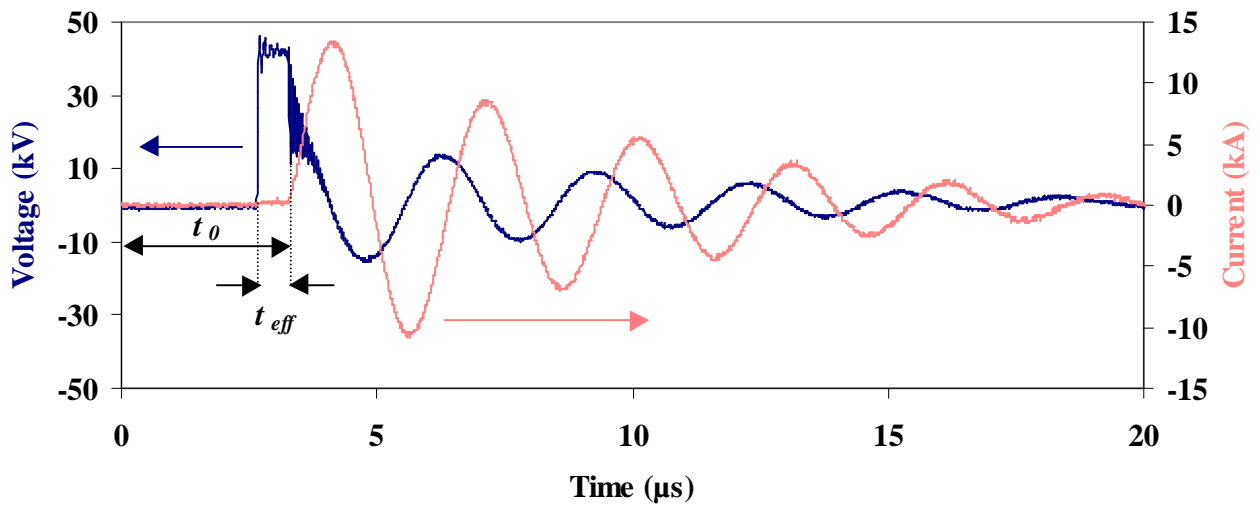


Figure 2. Typical voltage and current curves measured during a high-voltage electrical discharge with a 5-mm inter-electrode distance:
(t_{eff} : effective time of the applied voltage $\approx 0.5 \mu\text{s}$)
(t_0 : duration between the measure beginning and the creation of the discharge channel)

Process (fig. 3)

Linseed press-cake was reduced in powder by grating. The powder (30 g) and demineralized water ($5 \mu\text{S}/\text{cm}$), with a water/press-cake ratio of 10/1, were introduced into the discharge generator chamber.

Electrical treatments with different duration (0, 1, 5, 15, 30, 60, 80, 130, 220, 660, 1 080, 1 640 pulses) were applied.

The mixture composed of water and powder became a three-phase system: in addition to the water and the solid particles, oil, which was initially contained into the powder, has partially transferred in the water to form the emulsion.

This solid/emulsion system is centrifuged at 10 000 g and 20°C during 20 minutes with a fixed angle rotor centrifuge (JA-10, J2-21M/E centrifuge, Beckman Instrument, UK). Two phases are recovered: a solid and an emulsion.

All experiments were performed at room temperature and repeated one time.

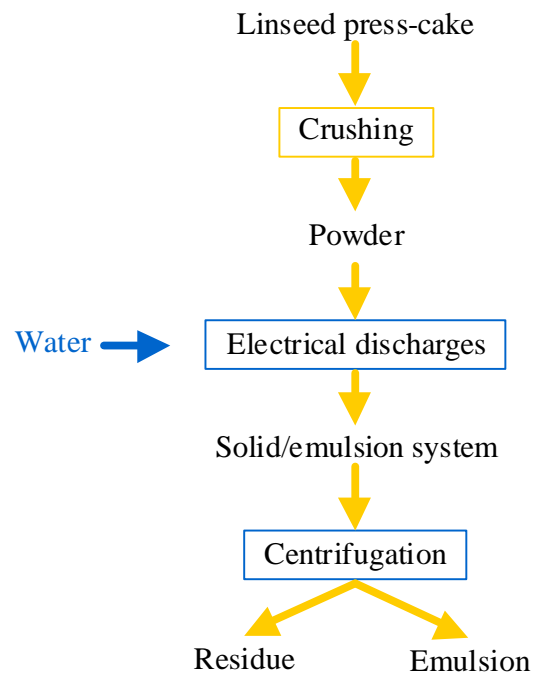


Figure 3. Extraction process.

Modeling of current curves [22]

When a discharge occurs in a RLC series circuit, the Ohm's law is:

$$L \frac{di}{dt} + R \cdot i - \frac{q}{C} = 0 \quad (3)$$

where i is the current (A), q is the electric charge (C) and $dq = -i \cdot dt$, L is the circuit inductance (H), C is the circuit capacitance (F) and R is the circuit resistance (Ω).

Equation 3 can be rewritten as:

$$L \frac{d^2q}{dt^2} + R \frac{dq}{dt} + \frac{q}{C} = 0 \quad (4)$$

The solution of the equation 4 has the following form:

$$q(t) = A \cdot \exp(-\beta \cdot t) \cdot \cos(\omega \cdot t + \varphi_q) \quad (5)$$

where A depends on the initial conditions (C), $\beta = \frac{R}{2L}$ is the decay factor (s^{-1}),

$$\omega = \sqrt{\frac{1}{LC} - \left(\frac{R}{2L}\right)^2}$$
 is the pulsation (s^{-1}) and φ_q is the phase displacement (rad).

By drifting the equation 5, the following equation is obtained for the current:

$$i(t) = -\frac{dq}{dt} = a \cdot \exp(-\beta \cdot t) \cdot \sin(\omega \cdot t + \varphi_i) \quad (6)$$

where a depends of the initial conditions (A) and φ_i is the phase displacement (rad).

The damped oscillations (fig. 3) in the generator begin at t_0 (μs), which corresponds to the duration between the beginning of the measure and the moment when the discharge channel is created. The oscillations have a period $T = \frac{2\pi}{\omega}$ (μs).

Analysis

Two types of analysis were performed in this work. We studied the evolution of the physical properties of the different liquid phases (solid/emulsion system and emulsion) and the composition of the initial and final products (linseed press-cake, solid residue and emulsion).

Physical properties

After the electrical treatment, temperature, electrical conductivity, Brix and pH are measured on the obtained solid/emulsion system. After the centrifuge separation, electrical conductivity, Brix and viscosity are measured on the obtained water-oil emulsion.

- Concentration measurements were based on Brix (g/100 g) values obtained using a digital hand refractometer (ATAGO PR-101, ATAGO Co., Japan).
- pH values were obtained with a pH-meter (MP 220, Mettler Toledo, Switzerland).
- Electrical conductivity was measured with a conductimeter (K610, Consort, Turnhout, Belgium).
- Viscosity was determined using a rotational viscometer (Viscotester 550; Thermo Haake; Karlsruhe, Germany) with SV/DIN at 20°C. The shear rate $\dot{\gamma}$ was increased from 0.1 s^{-1} to

1 030 s⁻¹ during 500 s, then $\dot{\gamma}$ was maintained for 100 s and decreased to 0.1 s⁻¹ in 500 s. Viscosity values are given at the speed of 1 030 s⁻¹.

Composition

- Moisture content of the press-cake, the solid residue and the emulsion was determined by drying samples of each phase overnight using an oven at 105°C [23].
- Oil content was determined on the dried solid residue with hexane according to AFNOR method [24]. Oil extraction yield is defined as percentage of oil extracted on a total oil extractable basis.
- Protein (N x 5.41) was determined by the Kjeldahl method with a Büchi digester (426) and a distillation unit (B-316).
- Sugar content was determined from a 0.05-0.10 ml aliquot of the extract. It was diluted to 1 ml and 10 ml of ice-cold anthrone reagent (0.05 % (w/v) anthrone in 6.5 N H₂SO₄ containing 1 % (w/v) thiourea) was added to it followed by mixing. The reaction mixture was held at 100°C for 10 min and the reaction mixture absorbance was measured at 620 nm. The total sugar content of the mucilage extracts (expressed as milligrams of D-glucose equivalents per milliliter of the sample) was calculated by multiplying the absorbance reading at 620 nm by a factor 3.06. [25]

Results and discussion

First, the evolution of the parameters of the model for the current curves and the relation between energy and t_{eff} are presented. Then the effects of this electrical treatment on the solid/emulsion system, the solid residue and the emulsion are studied.

Electrical diagnostics

A typical waveform of the discharge current obtained after the formation of a discharge channel is presented in figure 2. The amplitude of the discharge current reaches $I_{max} = 13$ kA at an output voltage of $U_0 = 45$ kV.

The obtained current curves were modeled on the oscillating part with the equation 6: the RLC series circuit consisted of the treatment chamber and the charge circuit. The mean values of the different coefficients obtained in a single experience of 2 500 pulses are presented in the table 2.

		Value	Standard error
U_{max} (kV)		54.9	13.1
I_{max} (kA)		12.3	1.5
t_0 (μs)		4.87	1.18
t_{eff} (μs)		2.22	1.17
a	eq. 6	1.41×10^4	0.43×10^4
β (s ⁻¹)	eq. 6	1.87×10^5	0.22×10^5
ω (s ⁻¹)	eq. 6	2.07×10^6	0.13×10^5
φ_i	eq. 6	-0.02	1.89
T (μs)		3.03	1.98×10^{-2}
Q (J)	eq. 11	22.2	13.9

Table 2. Main results obtained with the model (eq. 6) in an experience of 2 500 pulses.

As the total capacitance of the capacitor for the charge circuit, is $C = 0.20 \mu\text{F}$, one can estimate the self-inductance L , the resistance R , the impedance Z of the total electrical circuit and the phase displacement Φ between the current and the voltage signal from the estimated period of the current oscillations $T = 3.03 \mu\text{s}$:

$$L = \frac{1}{C} \left(\frac{1}{\omega^2 + \beta^2} \right) \cong 1.15 \mu\text{H} \quad (7)$$

$$R = 2\beta L \cong 0.43 \Omega \quad (8)$$

$$Z = \sqrt{R^2 + \left(L\omega - \frac{1}{C\omega} \right)^2} \cong 4.82 \Omega \quad (9)$$

$$\Phi = \tan^{-1} \left(\frac{L\omega - \frac{1}{C\omega}}{R} \right) \cong 0.47 \pi \quad (10)$$

The energy dissipated Q (J) into the solid/emulsion system is equal to:

$$Q = Z \cdot \cos(\Phi) \cdot \int_0^{\tau} i^2(t) \cdot dt \quad (11)$$

The evolution of the different parameters of the model was studied during this long experimentation.

The only one, which evolves, was t_{eff} (fig. 4), the time of tension application for the discharge apparition. This evolution is due to the modification of the solid/emulsion system. In the same time the pulse energy decreases (fig. 5). This is due to the decrease of the voltage during the creation of the streamers [13].

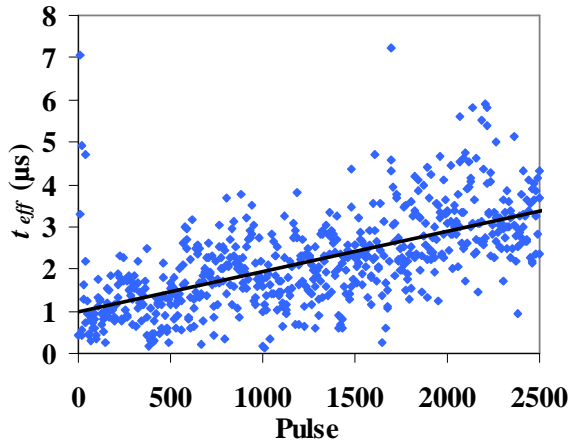


Figure 4. Evolution of t_{eff} during one experience.

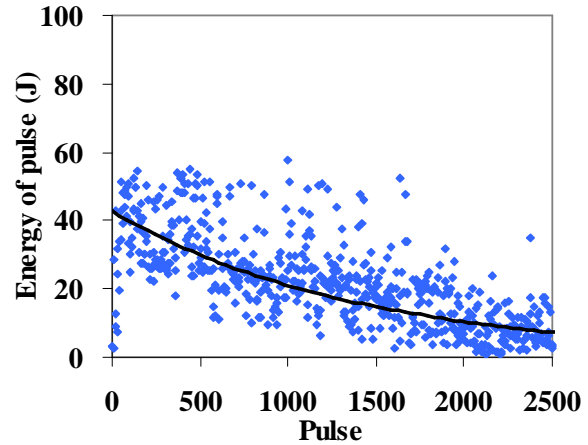


Figure 5. Evolution of Q (eq. 11) the energy of one pulse during one experience.

There is a relation between t_{eff} and the energy: the longer is t_{eff} , the lower is the initial voltage of the damped oscillations and then the lower is the calculated energy (fig. 6).

The sum of the applied energy (fig. 7) increases more and more slowly during experimentation: this is due to the decrease of Q .

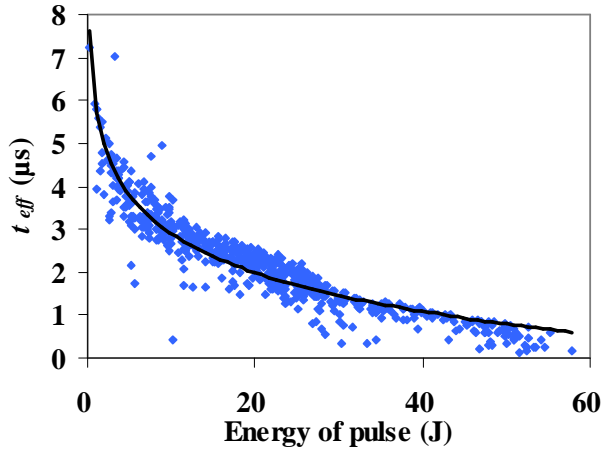


Figure 6. Relation between t_{eff} and Q the energy of the pulse.

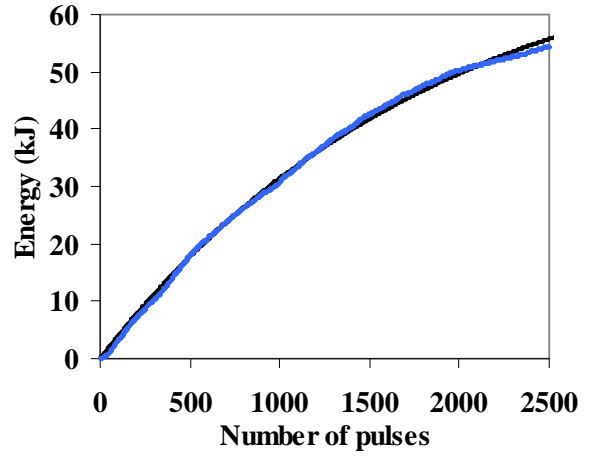


Figure 7. Energy cumulated $Q_{cumulated}$ during one experience.

Based on a linear regression, we obtain the following model for the evolution of t_{eff} (fig. 4):

$$t_{eff} = t_i + p \cdot t_p \quad (12)$$

where t_i is the duration for the creation of the discharge at the experience beginning (1 μ s), p is the number of pulses and t_p (10^{-3} μ s/pulse).

Another model is obtained for the evolution of Q (fig. 5):

$$Q = Q_0 \cdot \exp\left(-\frac{p}{\tau_p}\right) \quad (13)$$

where Q_0 is the energy applied at the experience beginning (40.75 J) and τ_p is the decay factor (1 845 pulses).

This model allows to express $Q_{cumulated}$ (fig. 7) as:

$$Q_{cumulated} = Q_0 \cdot \tau_p \cdot \left(1 - \exp\left(-\frac{p}{\tau_p}\right)\right) \quad (14)$$

Extraction effects

The results presented in this part were obtained by application of different number of pulses on the mixture composed of water and powder.

Evolution of the solid/emulsion system

The pH of the solid/emulsion system is almost constant during the treatment (6.2 ± 0.2). The temperature of the solid/emulsion system increases regularly by 1.5°C for 100 pulses.

The electrical conductivity of the solid/emulsion system is almost constant (1.4 mS/cm) until 220 pulses. Then it increases regularly to $2.3 \text{ mS}\cdot\text{cm}^{-1}$ for 1 640 pulses (fig. 8).

In the same time the concentration of the solution is also almost constant (2.8 g/100 g) until 30 pulses. Then the concentration increases regularly until 600 pulses and stabilizes to almost 5 g/100 g.

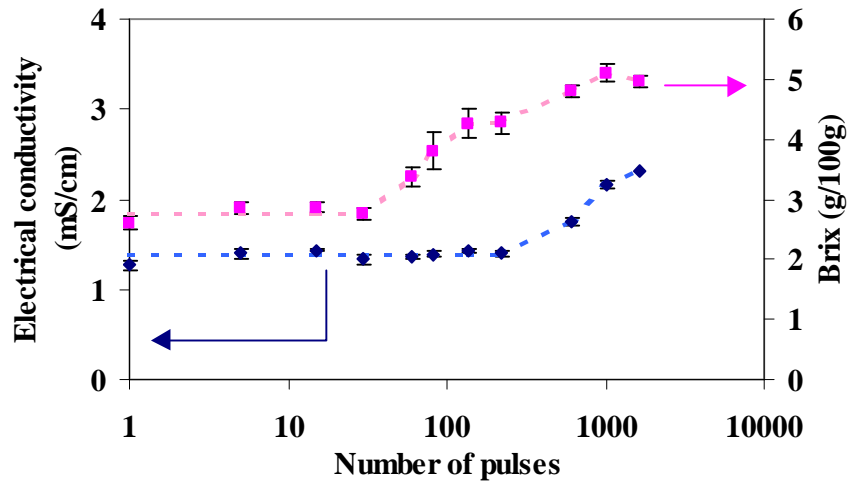


Figure 8. Evolution of electrical conductivity and Brix of the solid/emulsion system after electrical treatment.

Effect of centrifugation

There is an evolution of the repartition of the product at the end of centrifugation. During a first period of time (0-80 pulses) the mass of the residue is constant: the mucilage stands close to the hull particles and absorbs water. After 100 pulses, the mass of solid residue decreases (fig. 9). Such behavior in two parts was already described for the water extraction of mucilage from whole white mustard seed [12].

In our case, under 80 pulses, the electrical discharges are ineffective to extract the mucilage. It stays close to the hull particles and traps a great quantity of water, which is recovered with the solid residue. After 80 pulses, the mucilage extraction begins and the residue contains less water.

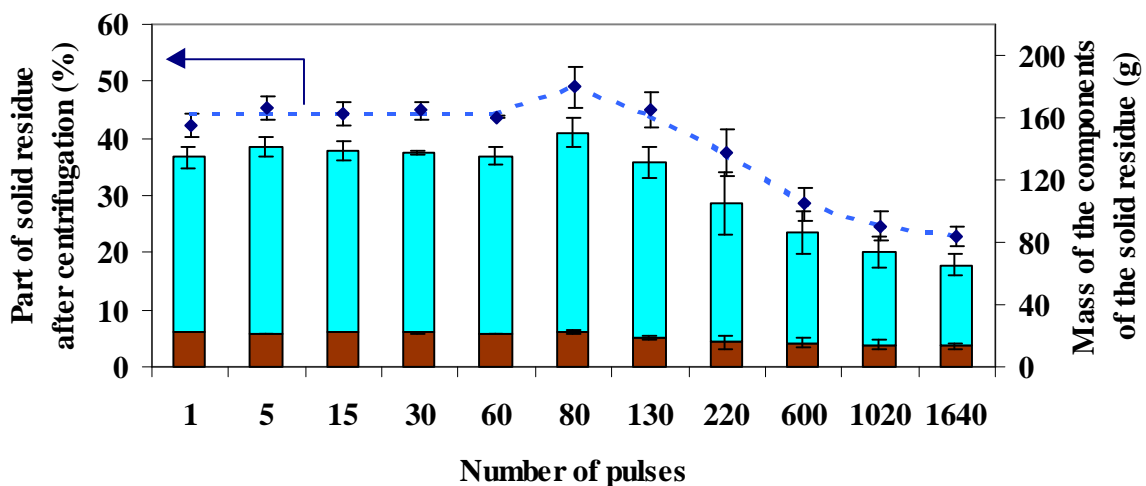


Figure 9. Evolution of the composition of residue (■ : water, ■ : dry matter) and the repartition of the solid/emulsion system after centrifuge separation.

Physical characteristics of the emulsion after centrifugation

The electrical conductivity of the emulsion has almost the same evolution as the electrical conductivity of the solid/emulsion system: it is constant (1.7 mS/cm) before 220 pulses, then it increases regularly to 2.3 mS/cm for 1 640 pulses.

In the same time the concentration of the emulsion is also almost constant (2.9 g/100 g) until 30 pulses. Then the concentration increases regularly until 600 pulses and stabilizes to almost 4.9 g/100 g. This is also the same evolution as the concentration of the solid/emulsion system.

Evolution of the rheological comportment

The following experience was done on the emulsions obtained after centrifugation: the shear rate $\dot{\gamma}$ was increased (0.1 s^{-1} - $1\ 030 \text{ s}^{-1}$, 500 s), then maintained ($1\ 030 \text{ s}^{-1}$, 100 s) and decreased ($1\ 030 \text{ s}^{-1}$ - 0.1 s^{-1} , 500 s). Viscosity values are given for $\dot{\gamma}=1\ 030 \text{ s}^{-1}$ and, for the increase and decrease of $\dot{\gamma}$, a model is proposed for the curves $\tau = f(\dot{\gamma})$.

From 0 to 30 pulses the emulsion viscosity at $1\ 030 \text{ s}^{-1}$ (fig. 10) is constant ($\mu = 17 \text{ mPa.s}$). Then from 30 to 600 pulses, viscosity increases to 45 mPa.s.

There is a linear relationship between the concentration of the emulsion (Brix) and the viscosity (fig. 11): $\mu = a \cdot \text{Brix} + b$, where a ($12.68 \text{ mPa.s.}^\circ\text{Brix}^{-1}$) and b (-18.81 mPa.s) with $r^2 = 0.88$.

The molecules, which increase the concentration, increase the viscosity: this is due to the extraction of mucilage. Either the measure of the concentration in the solid/emulsion system or the viscosity of the emulsion allow to evaluate the mucilage extraction.

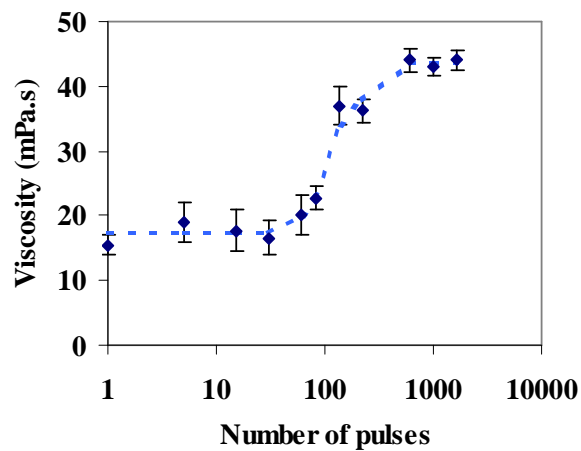


Figure 10. Evolution of viscosity of the emulsion after centrifugation.

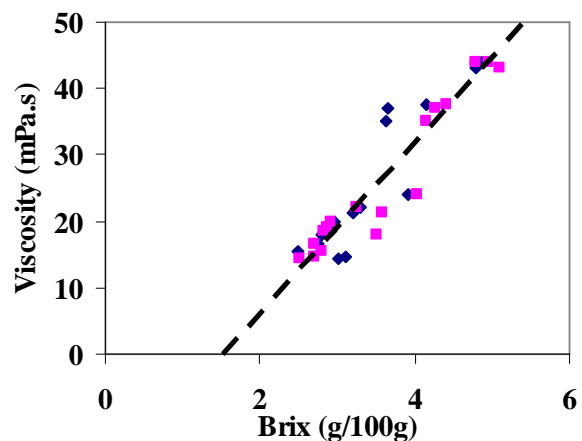


Figure 11. Relation between viscosity and Brix of the solid/emulsion system (■) and the emulsion (◆).

The rheology of emulsions exhibits a pseudo-plastic behavior:

$$\tau = K \cdot \dot{\gamma}^n \quad (15)$$

where K (Pa.s^n) and n are defined as the consistency index and the dimensionless flow behavior index, respectively and τ is the shear stress (Pa).

From 0 to 30 pulses, K and n are constant (fig. 12). The value of n is around 0.9: the emulsion has a quasi-Newtonian comportment. After 30 pulses, the value of n decreases to 0.6: the emulsion exhibits then a rheofluidifiant comportment.

The behavior of the emulsion is modified during the $\dot{\gamma}$ decreases (fig. 13). K and n increase and decrease less than previously: the emulsions have also a thixotropic comportment.

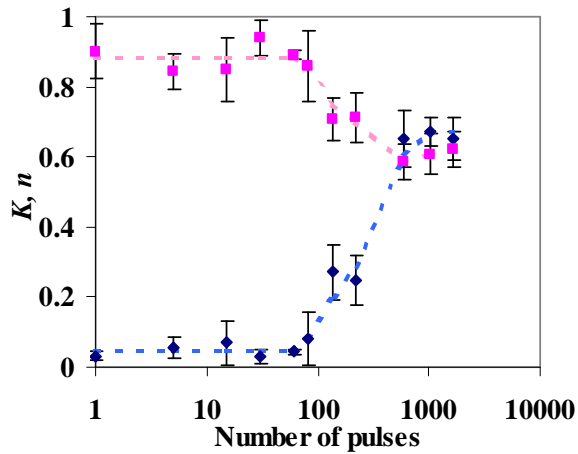


Figure 12. Evolution of the parameters K (\blacklozenge) and n (\blacksquare) of the rheological model (eq. 15) for the increase of $\dot{\gamma}$.

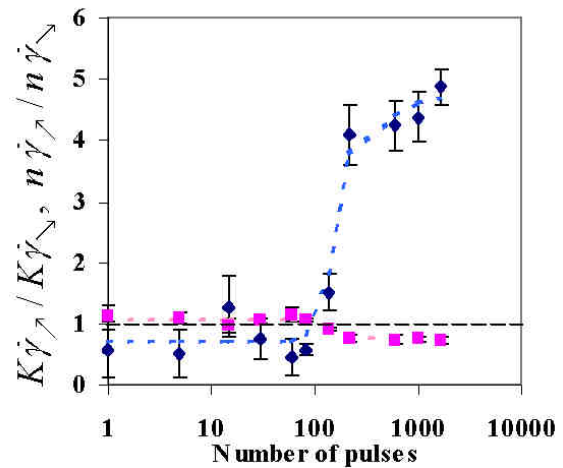


Figure 13. Evolution of $K\dot{\gamma}_{\uparrow} / K\dot{\gamma}_{\downarrow}$ (\blacklozenge) and $n\dot{\gamma}_{\uparrow} / n\dot{\gamma}_{\downarrow}$ (\blacksquare), --- non thixotropic behavior $K\dot{\gamma}_{\uparrow}, n\dot{\gamma}_{\uparrow}$ refer to $K(n)$ for increasing shear rate $K\dot{\gamma}_{\downarrow}, n\dot{\gamma}_{\downarrow}$ refer to $K(n)$ for decreasing shear rate.

Composition of the emulsion and of the residue

As soon as the powder is introduced in the water (fig. 14), almost 17 % (4.8 g) of the dry matter of the product transferred into the liquid phase. No more evolution was noticed before 80 pulses. Then an exponential extraction phase began and stopped between 600 and 1 080 pulses: 42.8 % (11.9 g) of the dry matter was extracted.

For the solid residue extraction a lag phase of 80 pulses was also observed.

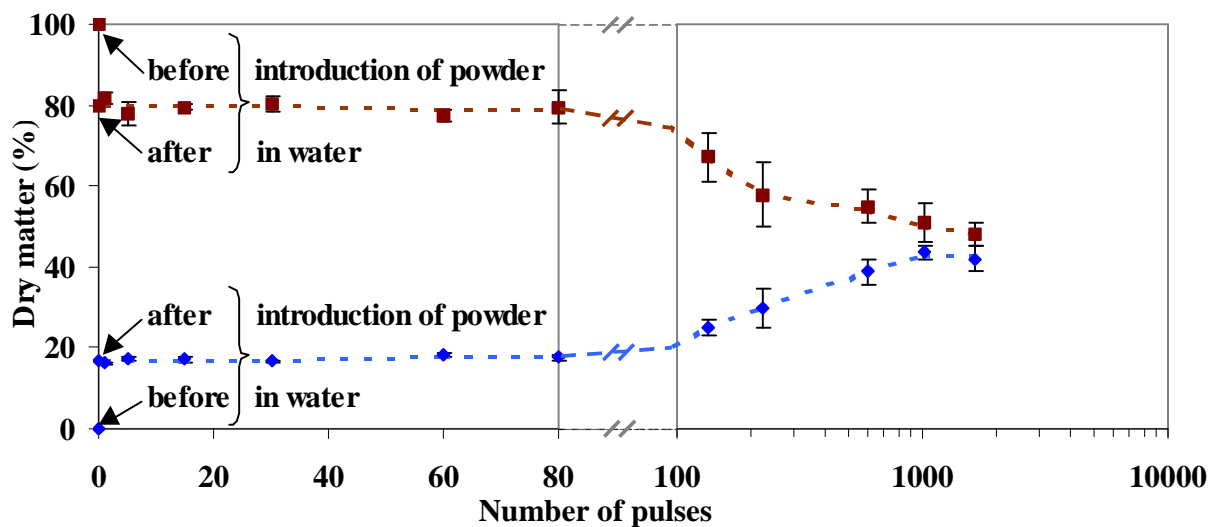


Figure 14. Evolution of the dry matter repartition in liquid phase (\blacklozenge) and solid residue (\blacksquare) during the electrical treatment.

As soon as the powder was introduced to the water (fig. 15), the 26 % (1 g) of the oil transferred into the liquid phase. No additional oil was extracted before 30 pulses. The second extraction of oil occurred between 30 and 220 pulses. Even after 1 640 pulses, 26 % (1 g) of the initial oil retained inside the solid residue.

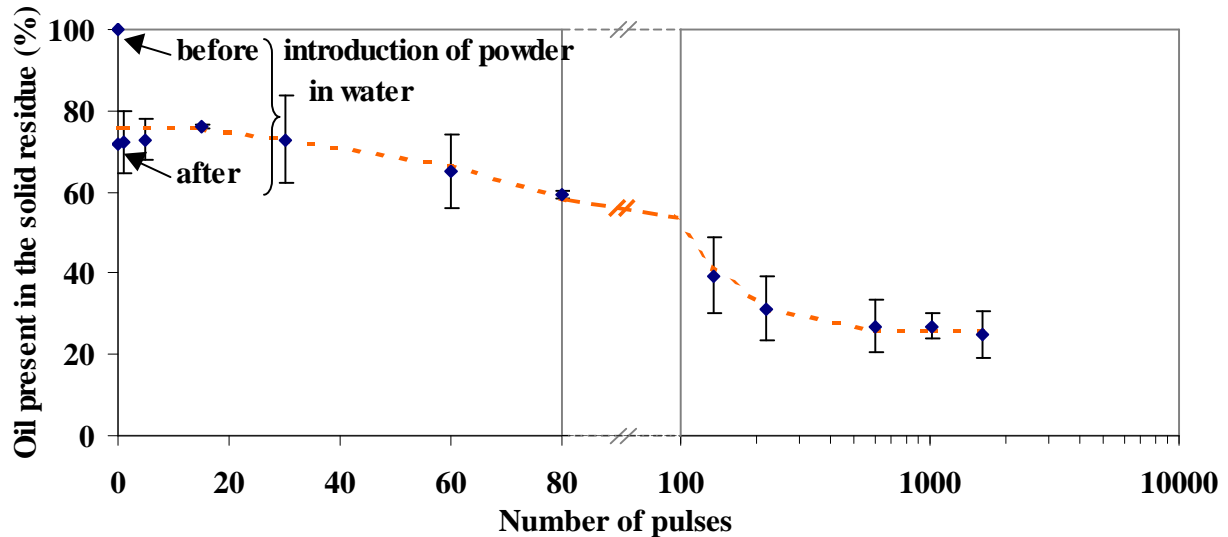


Figure 15. Percentage of the initial oil present in the solid residue after electrical treatment.

The first extraction (0-30 pulses), which is very quick, was due to the action of the water and not to the action of electrical discharges.

As 26 % of the oil is transferred into the emulsion only with the action of water, a short (or no) treatment and a centrifugation could be use to extract this part of oil, which would be easily recovered because the obtained emulsion is not stabilized by the presence of mucilage. Then a further electrical treatment could be applied to extract the mucilage and the remaining oil.

Conclusion

This work has allowed knowing where the different components of linseed press-cake went during this extraction process enhanced by electrical discharges.

The modeling of the current curves has allowed to calculate the different characteristics of the electrical circuit and the applied energy. The relation between the time of apparition of the discharge and the applied energy was expressed.

The mucilage begins to be extracted after 80 pulses. A Brix measure on the solid/emulsion system allows to predict the viscosity value of the emulsion and to assert mucilage is extracted.

A lag phase (0-30 pulses) was determined for the oil extraction: electrical discharges seem to have no effect on the extraction during this period. This could be used to do a first treatment (electrical discharges (less than 30 pulses) and centrifugation) and continue the electrical treatment with the solid residue and fresh water.

The mucilage removal is very efficient with the electrical discharges, and may be valorized due to its interesting proprieties. The residue obtained after electrical treatment contains 26 % of the initial oil and could be treated with others techniques of extraction.

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