Application of High-Voltage Electrical Discharges for the Aqueous Extraction from Oilseeds and Other Plants

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Abstract Aqueous extraction is a traditional operation unit used to recover from food plants various products such as sugar, oil, or proteins. The yields of extraction are generally low. To enhance aqueous extraction different treatments may be applied before and/or during extraction. Application of high-voltage electrical discharges in water leads to original phenomena such as shock waves or active species creation. Steps of creation and required material are presented. High-voltage electrical discharges in water are interesting for different applications especially for extraction.

1 Introduction

The electrotechnology called high-voltage electrical discharges (HVED), which is presented in this chapter, is used in aqueous solutions in order to extract oil and soluble material from plant products. Basically, HVED can be divided into three categories (Fig. 1): (i) arc discharge through the interior of solid material; (ii) underwater arc discharge, which can initiate a strong pressure pulse and produce significant oxidative chemistry in the bulk liquid resulting from UV-photolysis, electrohydraulic cavitation, and supercritical water oxidatior; (iii) electrolytic discharge, which can establish and maintain a strong electric filed in a treatment chamber for a few microseconds but will avoid heating and electrical breakdown.

When electrical treatment is applied in an aqueous solution, different phenomena are observed during HVED, depending on electric field intensity. If the electric field is of low intensity, water carries electricity and behaves like a good electrical conductor. Electrical signals look like simple exponential decreases (Fig. 2) (Sun et al. 1997) such as signals observed in wet solid electrical treatment. When the electric field overshoots the breakdown electric field, a new phenomenon involving this electrical breakdown is observed. Electric current goes through water, which behaves like an insulating material, and produces shock waves, ultraviolet

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Fig. 1 Categories of electric discharges: arc discharge through solid (*left*), liquid (*center*), electrolytic discharge (*right*) (adapted from Bluhm et al. 2001)



Fig. 2 Typical voltage and current curves in absence of electrical breakdown (Gros 2005)

radiations, and active species (Naugol'nykh and Roi 1971; Sun et al. 1998). Then, the electrical signals look like damped oscillations (Sun et al. 1998; Zuckerman et al. 2002; Gros et al. 2004). The application of HVED produces such phenomena. In fact, classical discharge, which mainly depends on electrical conductivity of the aqueous solution and creation of electrical breakdown, can be in competition.

Lightning is the first electrical breakdown observed by humans. The electric origin of this natural phenomenon was discovered by Benjamin Franklin in 1753. The first artificial electrical breakdown was produced by Humphry Davy towards 1800 (Hnatiuc 2002). This is the beginning of intense activity in research and in the application of electrical discharge. In 1905, Swedbery found that a pulsed discharge in water can produce an intense shock wave (Lu et al. 2001). The study of electrical

breakdowns leads to the identification of their main characteristics, namely, their huge complexity, the influence of many factors, and the variety of their forms (electrical breakdown, corona discharge, streamer, lightning, etc.). Even after two centuries of research and about 20 major theories, many aspects of this phenomenon stay unappreciated. Many authors have tried to explain the formation of electrical breakdown in liquids, mainly water (Naugol'nykh and Roi 1971; Katsuki et al. 2002).

Extraction is an essential operation for industries that treat biological materials. Many technologies have been developed for this operation. Extraction may be done by pressure (screw press, hydraulic press (Beckett 1999), belt press, etc.) or by a solvent, for example, hexane (Abu-Arabi et al. 2000), supercritical CO₂ (Bozan and Temelli 2002), or water. The transfer of solute from biological solids to an adjacent liquid is a traditional unit operation in different food applications. It is used to obtain various products such as sugar, coffee, tea, and pectin. To enhance aqueous extraction different treatments (mechanical, biological, thermal, electrical (El-Belghiti et al. 2005)) may be applied before and/or during extraction.

Concerning oilseeds, aqueous extraction may be mainly used to recover oil and/or soluble products such as proteins (Lopes Barbosa et al. 2006), gums, and mucilage (flaxseed, mustard). Aqueous extraction may be the central operation or a part of the process. Indeed, different types of product can be treated: whole or crushed seed, press-cake, and defatted cake.

A seed is considered as oilseed if it is grown largely for oil. The major oilseeds produced in 2005 (FAO 2005; United States Department of Agriculture 2007) are soybean (217.9 Mt), rapeseed (48.6 Mt), cottonseed (42.5 Mt), peanut/groundnut (33.9 Mt), sunflower seed (29.7 Mt), palm kernel (10 Mt), copra/coconut (5.6 Mt), sesame seed (3.2 Mt), flaxseed/linseed (1.8 Mt), and castor seed (1.3 Mt in 2003).

The treatment of whole oilseeds (e.g., flaxseed (Cui et al. 1994) and white mustard (Balke and Diosady 2000)) with aqueous extraction is usually used to extract mucilage that is present in the hull of seeds. Mucilage is a heterogeneous polysaccharide which has interesting properties and may have applications in food industry. There are many different methods of aqueous mucilage extraction. Some authors have tested the hot extraction from flaxseed (Mazza and Biliaderis 1989; Bhatty 1993; Cui et al. 1994). Optimum conditions have been determined by experimental design: temperature of 85–90°C, pH 6.5–7.0, and 13/1 water/seed ratio (Cui et al. 1994). Others have soaked whole seeds in water or sodium bicarbonate solutions (0.05 and 0.10 M for 6 and 12 hours) and have used polysaccharide degrading enzymes (Wanasundara and Shahidi 1997). On white mustard seeds, two-stage extraction process using water with an initial temperature of 45° C at an 8/1 water/seed ratio resulted in over 90% mucilage removal in approximately 3 hours (Balke and Diosady 2000). The authors also show the existence of two steps in mucilage extraction: first hydration of the seed and mucilage, then dissolution of mucilage.

The aqueous extraction of oil is an interesting alternative to solvent extraction due to simultaneous recovery of protein and oil, the better quality of oil, and the absence of volatile organic compounds emission (Rosenthal et al. 1996). But aqueous extraction has also some disadvantages: lower oil yield, high water consumption, and the

addition of demulsification operation. Sometimes this problem of low extraction efficiency of aqueous processes can be overcome by optimization of process parameters (pH, temperature, time, water/seed ratio, agitation, number of treatments, etc.) or by the use of hydrolytic enzymes (Tano-Debrah and Ohta 1997; Sharma et al. 2002).

The aqueous extraction process of oil is generally composed of three main stages:

- Firstly, the crushed product is dispersed in water. The crushing stage is crucial for aqueous extraction. It induces the particles reduction and structure modifications; this facilitates the oil output (Southwell and Harris 1992). However, an excessive crushing leads to stable emulsion formation when the product is dispersed in water. Indeed, smaller oil drops are obtained and demulsification becomes more difficult (Aguilera et al. 1983). Concerning the water/seed ratio, the lowest values seem preferable to obtain less stable emulsions and generate less effluent to treat. But higher water/seed ratios are required to achieve better oil yield. The optimal water/seed ratio depends on the type of treated product.
- Secondly, the aqueous mixture is agitated to enhance oil and proteins extraction. Whereas a simple agitation is needed to obtain high oil yield for groundnut and sunflower, greater energies are needed to have an efficient extraction for other seeds. For each product, an optimal value of pH exists for oil extraction (Kim 1989; Southwell and Harris 1992). This value corresponds also to the optimum of protein extraction: a strong correlation seems to exist between protein and oil extraction. Moreover, weakest yields of extraction for oil and proteins are obtained for the isoelectric pH, which corresponds to the minimum solubility of proteins. Thus, aqueous extraction may be considered as an operation of protein solubilization which involves oil extraction. The temperature of extraction seems to have a complex effect on oil extraction. The optimal duration of extraction depends on treated product. The increase of the duration leads to the production of more stable emulsion.
- Thirdly, this separation stage is performed to recover oil and proteins.

Different physical treatments as ultrasounds, microwave heating, instantaneous pressure drop, etc., can be used to increase extraction yields. This chapter focuses on the application of HVED for the enhancement of aqueous extraction from oilseeds.

2 High-Voltage Electrical Discharges

The steps of creation of HVEDs are explained below. The HVED have already different applications such as degradation of organic compounds contained in water (Sugiarto and Sato 2001), microorganism inactivation (Zuckerman et al. 2002), or extraction of soluble material (Vishkvaztzev et al. 1998; Barskaya et al. 2000).



Fig. 3 Type of electric discharge in aqueous solution. (a) streamer, E = 4.4 kV/cm; (b) streamer and spark, E = 13.3 kV/cm; (c) spark, E = 33.3 kV/cm (Sugiarto and Sato 2001)

2.1 Steps of Creation

The application of high voltage across the electrodes leads to accelerate the electrons that achieve enough energy to excite water molecules. Then an avalanche of electrons is created. Air bubbles, which are initially presented in liquid or formed thanks to local heating, participate and accelerate the phenomenon (Alkimov et al. 1971).

If electric field is intense enough, avalanche of electrons becomes the starting point of streamer propagation from the positive electrode to the negative one (Fig. 3). Lots of active molecules are produced inside of streamers under action of excited electrons (Joshi et al. 1995; Sun et al. 1998).

When one of the streamers reaches negative electrode, electrical breakdown occurs and discharge channel is created. This leads to the increase of arc diameter, current increase, and voltage decrease. This discharge channel, which is also 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 nonelastic properties of the liquid, lead to the formation of shock waves (Vitkovitsky 1997; Zuckerman et al. 2002). In addition to the shock wave formation, ultraviolet radiations and active species are produced (Sun et al. 1998).

All these steps of arc creation may be interesting for comprehension of such a technology, but a question is still there: how can we produce electrical breakdown in water?

2.2 Condition of Apparition

In order to obtain electrical breakdown between two electrodes, the value of applied electric field E (V/m) must exceed the breakdown electric field E_{br} . Different formulas are used to calculate E according to electrodes geometry (Burkes 1978). For needle/plate electrode, electric field is calculated with the following formula:

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$$E = 0.9 \cdot \left(\frac{U}{l}\right) \cdot \frac{r+l}{r} \tag{1}$$

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

The value of E_{br} (V/m) can be estimated from the next semiempirical formula (Martin 1973):

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

where k is a constant (V.s^{1/3}.m^{-0.8}), S is the area of the electrode (m²) and _{eff} is the effective time of the applied voltage (s), that is, the duration between voltage application and leader creation while voltage is almost constant.

2.3 Material and Energy

The production of electrical breakdown in water requires a generator with a huge capacitor. In Figs. 4 and 5 an example of generator and chamber of treatment used to produce HVED for extraction is presented. Some characteristics of electrical circuits of laboratory-scale HVED generators are presented in Table 1.

The energy is initially contained in the capacitor. A huge quantity of energy is needed to create the electrical breakdown in water. This energy is considered as losses of energy. Authors have managed to determine different origins of energy losses for HVED treatment in water (Yushkov 2004). The losses of energy W_L in the pre-breakdown stage of discharge buildup can be represented in the following form:

$$W_L = W_1 + W_2 + W_3 \tag{3}$$



Fig. 4 Schema of a HVED generator (Gros et al. 2004)



Treatment chamber

Capacitor and spark gap

 Table 1 Characteristics of electrical circuits of laboratory scale HVED generators

Energy $W(J)$	Capacitor C (μ F)	Inductance L (µH)	Distance between electrodes l (mm)	Reference
200	0.01	15–200	13–50	Barskaya et al. 2000
160	0.2	1.15	5	Gros et al. 2004

where W_1 is the energy losses with the formation of overheated instability in the zone of potential electrode due to the flow of ion currents from this electrode; W_2 is the energy losses with formation and development of leaders in the working space; W_3 is the energy losses due to spreading of currents from the surface of the bare part of the potential electrode and surface of leaders. Yushkov (2004) has shown that the energy losses are not negligible; they can achieve up to 50% of the energy contained in the capacitor.

With the experimental equipment presented in Fig. 4, Gros et al. (2004) have treated a suspension of linseed press-cake in a long time experiment (2,500 pulses)



Fig. 6 Typical voltage and current curves measured during a HVED with a 5 mm interelectrode 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 (Gros et al. 2004)

Fig. 5 Generator of HVED (Gros 2005)



Fig. 7 Typical voltage and current curves measured at the beginning of HVED with a 5 mm interelectrode distance (photographs: Sun et al. 1998)

and recorded electrical signals (Figs. 6 and 7). They determined the duration of electrical breakdown formation t_{eff} , the electrical characteristics associated to the damped oscillations and the energy consumed during these oscillations. The values of t_{eff} increase during the experiment (Fig. 8a). This is explained by the increase of electrical conductivity of the treated mixture during experiment. This also means that energy losses becomes higher during formation of electrical breakdown: the pulse energy decreases with the increase of t_{eff} (Fig. 8b).

2.4 Interest in HVED for Various Applications

An electricalbreakdown in water has many applications because of various effects that can be produced: ultraviolet radiations, active species (Sun et al. 1998), and shock waves (Vitkovitsky 1997).

These secondary effects can be used, for example, for water ozonation. The application of electrical breakdown leads to bubbles division and improves treatment efficiency. The creation of active substances, involves destruction of polluting substances and microorganisms (Malik et al. 2001). The application of HVED permits the degradation of pollutants such as phenol (Sugiarto and Sato 2001; Chen et al. 2004), the decoloration of dyes (Sugiarto et al. 2003) and inactivation of microorganisms (Zuckerman et al. 2002). Electrical breakdown in water can also be of interest for the effects of associated shock waves. Mikula et al. 1997 have used discharges (U= 40 kV, I_{max} = 45 kA, C = 0.50 μ F, L = 1.4 μ H, l = 7 mm) to treat water suspensions of TiO₂ or wood (sawdust, needles) and water solution of methylhydroxyethylcellulose. Shock waves crush TiO₂ particles: their average diameter decreases and suspension turbidity is modified. In fact, TiO₂ aggregates, which are spontaneously formed in water, are broken thanks to shock waves. In the case of beech sawdust, the rate of acid hydrolysis increases with the number of discharge pulses. These changes have been interpreted as the result of physical destruction of the materials, for example, increasing of specific surface area of the treated materials. The viscosity of the solution of methylhydroxyethylcellulose slowly decreased with application of pulses but the molecular weight of the polymer



Fig. 8 Evolution of the duration of electrical breakdown formation t_{eff} during a 2,500 pulses experiment (**a**). Relation between t_{eff} and the energy consumed after electrical breakdown (**b**) (Gros et al. 2004)

changed only slightly. The treatment of spruce needles improves their wetability and thus the contact with microorganisms (Mikula et al. 1997). Apparatuses were also developed to split up rocks (Wesley and Ayres 1984).

3 Extraction Enhanced by HVED

HVED treatments may also be interesting to enhance aqueous extraction. The effects of classical aqueous extraction and HVED are combined.



Fig. 9 HPLC profiles of soymilk produced with classical extraction and extraction enhanced by HVED (wavelength = 280 nm) (Vishkvaztzev et al. 1998)

3.1 Quality of Extracted Products

As HVED treatment produces active species, authors were interested in the quality of proteins (Vishkvaztzev et al. 1998). They have used HPLCto compare protein profile of soymilk obtain with classical extraction and HVED treatment. Their conclusion is that HVED treatment seems to have no effect on quality of extracted proteins (Fig. 9). Gros (2005) has also tested effect of HVED treatment on quality of proteins. HVED pulses are applied on BSA solution using a 1 l cell chamber related to a generator (U = 40 kV, C = 0.20 µF, l = 5 mm, W = 160 J). Treated and untreated solutions are compared using HPLC (Fig. 10). No difference is observed between the two signals. This result confirms the absence of effect of HVED treatment on proteins.

3.2 Extraction of Soluble Molecules

HVEDs may be used to accelerate soluble molecules extraction from biological products (Fig. 11) (Barskaya et al. 2000). With a generator (U = 50 kV, C = 0.01 μ F, l = 13-50 mm, W = 100-500 J), extraction speed could be multiplied by 40 up to 50 compared to infusion.

HVED treatment is used to enhance a mucilage extraction from whole linseed (Gros et al. 2003). Authors compared aqueous extraction at 34°C under agitation (120 rpm) with different water/seed ratios and HVED treatment. Seeds of 50 g are melted with 500 ml of demineralized water (20°C) and treated for 10 min (i.e., 300 pulses at 0.5 Hz) with a generator (U = 40 kV, $C = 0.20 \mu$ F, l = 5 mm, W = 160 J). A centrifuge separation is then performed (17,700g, 20°C, 10 min) to obtain the solution and residue. The residue is then treated a second and a third time with freshwater in the same conditions. During HVED treatment, linseeds are crushed under action of shock waves. Three 10 min treatments were sufficient to extract mucilage almost entirely (Fig. 12). In the third one, proteins begin to be



Fig. 10 HPLC profiles of untreated (a) and treated (b) solutions of protein (wavelength = 280 nm) (Gros 2005)



Fig. 11 Effect of HVED treatment on extraction of different biological products (Barskaya et al. 2000)



Fig. 12 Amount of matter extracted with different water/seed ratios and with three electrical discharge treatments with generator of electrical discharges (Gros et al. 2003)

extracted. The authors also observed that the increase of first treatment duration has an undesirable effect: mucilage creates a jelly, which traps the seeds, and so centrifuge separation becomes less efficient.

El-Belghiti (2005) has applied HVED for aqueous extraction of solutes from two dried products: tea leaves and *Datura inoxia* (common name: moonflower) roots. The water/gratings ratio was fixed as 10. Aqueous extraction was carried out at room temperature (20°C) under stirring at 250 rpm. HVED (U = 40 kV, 100 pulses) had smashed products to small fragments, and a limited increase of 10°C temperature was observed. The extraction kinetics were obtained by measuring, in the suspension, Brix(tea leaves, Fig. 13) or electric conductivity (*Datura inoxia* roots, Fig. 14).



Fig. 13 Effect of a HVED treatment on aqueous extraction from dried tea leaves (adapted from El-Belghiti 2005)



Fig. 14 Effect of a HVED treatment on aqueous extraction from dried *Datura inoxia* roots (adapted from El-Belghiti 2005)

In both cases, HVED application has led to higher solute concentration and has increased the extraction kinetic.

El-Belghiti et al. (2007) have compared moderate pulsed electric field (MPEF), HVED, and ultrasonic irradiations (UI) as treatments to enhance aqueous extraction from fennel gratings. The objective was to obtain extract used as natural food preservative (antioxidants). The water/gratings ratio was fixed as 2. Aqueous extraction was carried out at room temperature (20° C) under stirring at 250 rpm. The three assisted extractions led to the same final yield of solutes and preserve the



Fig. 15 Kinetics of solute extraction from fennel gratings pre-treated by MPEF, HVED and UI. c^* (%) is the ratio c/c_{∞} , c being the actual solutes concentration in solution and c_{∞} being the equilibrium solutes concentration (El-Belghiti et al. 2007)

antioxidant substances. However, their kinetics were different due to the principles of action and the treatments used, which were different (Fig. 15). HVED and UI offered, respectively, the most rapid and the slowest kinetics. The final yield of 98% was reached in 20 min with HVED, in 40 min with MPEF, and in more than 180 min with UI. The amounts of energy needed for these treatments were also different: the treatment by UI requires a high amount of energy (320 kJ/kg) compared to HVED and MPEF (70 and 40 kJ/kg, respectively). Thus, the MPEF treatment appears to be energetically the most economic one.

3.3 Oil Extraction

Aqueous extraction of oil may also be enhanced by application of HVED. Kinetic of aqueous extraction enhanced by HVED is established for linseed press-cake (Gros 2005). Linseeds are crushed and pressed for 1 hour with an hydraulic press (12 MPa, 50°C) (Gros et al. 2003). Press-cake is reduced in powder (30 g) and melt with demineralized water (300 ml, 5 μ S/cm, 20°C). The mixture is treated with HVED (1–1,640 pulses) and centrifuged (10,000g, 20°C, 20 min). As soon as linseed powder and water are in contact, 26% of the residual oil moves into water (Fig. 16). This first step of extraction is simply due to a washing effect of water. Then from 15 to 220 pulses, oil is extracted under the action of HVED and 26% of oil remains in the residue after 1, 640 pulses.

For dry matter (Fig. 17), the first contact between linseed powder and water provokes an immediate extraction of 15.5% of dry matter (first phase). From 80 to 1,640 pulses dry matter is also extracted (second phase). This extraction in two phases was also described for aqueous extraction of mucilage from mustard seed (Balke and Diosady 2000).

Optimization of such a process is possible using classical lever for aqueous extraction (pH, time, water/seed ratio, agitation, number of treatments, etc.) and



Fig. 16 Effect of number of pulses on oil contain in final residue (Gros et al. 2004)



Fig. 17 Effect of number of pulses on quantity of dry matter in liquid phase (\blacklozenge) and solid residue (\blacksquare) during HVED treatment (Gros et al. 2004)

knowledge on HVED. Thanks to previous results (Gros 2005), a process based on two stages of aqueous extraction enhanced by HVED was proposed to recover oil from linseed press-cake. The extraction process was optimized using an experimental strategy based on a central composite design with star points (Gros et al. 2005). For each experiment, the same number of pulses (280) is applied. Authors have tested the effect of the repartition of pulses on two successive steps (13+267; 60+220; 130+150; 200+80; 247+33) and three process parameters: pH (4.9; 5.2; 5.8; 6.2; 6.5), water/press-cake ratio (6.67/1; 7.5/1; 8.75/1; 10/1; 10.83/1), and temperature (13; 20; 30; 40; 47°C). The authors have examined the main effect of these parameters on oil and soluble matter extraction and on energy consumed after electrical breakdown.

The pH has an effect on discharge efficiency and on aqueous extraction of oil but not on soluble matter (Figs. 18–20). The pH of suspension was controlled by addi-



Fig. 18 Main effects of parameters on oil contained into the final residue (Gros et al. 2005) (*curves*: model; *points*: mean of experimental values)

tion of acid or base: electrical conductivity of suspension was modified and losses of energy during HVED creation were increased. The HVED were less efficient with the addition of ions. pH modifies the solubility of proteins: minimum solubility is observed at pH 3.5–4.0 (isoelectric pH) for linseed proteins (Dev and Quensel 1988). So the increase of pH increases the solubility of proteins and enhances oil extraction.

Water/press-cake ratio had an effect on discharge efficiency and on aqueous extraction of oil (Figs. 18 and 20). The increase of water/press-cake ratio has decreased the extraction efficiency. This effect came from enhanced extraction for concentrated suspensions and from the decrease of discharge efficiency with the increase of water/press-cake ratio.

Duration of first treatment had an effect on extraction of oil and soluble matter. There was an optimal repartition of pulses between the first and the second treatment for oil extraction. This effect results from the decrease of extraction efficiency in the first treatment (Fig. 21) as it was observed during kinetic study.



Fig. 19 Main effects of parameters on dry matter contained into residue (Gros 2005)



Fig. 20 Main effects of parameters on useful energy consumed for a 280 pulses experimentation (Gros et al. 2005)



Fig. 21 Relation between mean duration of electrical breakdown formation t_{eff} and mean energy consumed (Gros et al. 2005)

Temperature had an effect on extraction of oil and dry matter but not on discharge efficiency.

4 Conclusion

HVEDs in water produce different phenomena such as ultraviolet radiation, actives species, and shock waves. All these phenomena may be interesting for different applications (water treatment, pretreatment of biological product, particle crushing). Even if HVED treatment is aggressive, no denaturation of protein is observed on treated samples.

Concerning aqueous extraction, HVED enhances kinetic and quantity of extracted matter (soluble matter, oil). To understand and optimize aqueous extraction enhanced by HVED, knowledge of physical principles of discharges and aqueous extraction are required.

HVEDs in water should not be considered as the unique solution to enhance the aqueous extraction from food plants. However, this type of electrical treatment gives the interesting results for different tested products (soybean, potato, tea, peat, linseed, *Datura* roots, fennel grating, linseed press-cake).

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