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Pretreatments for the efficient extraction of bioactive compounds from plant based biomaterials

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Abstract

The extraction of medicinal or functional compounds from herbal plants is an important unit operation in food and bio-industries. The target compounds generally present inter- or intra-cellularly in an intricate microstructure formed by cells, intercellular spaces, capillaries, and pores. The major resistance of molecular diffusion in plant origin materials always comes from the intact cell walls and adhering membranes. Therefore, increasing the permeability of cell walls and membranes plays a very important role to increase extraction yield and/or extraction rate.

Important pretreatment methods to modify the cellular structures and increase the permeability of cell walls or membranes were discussed in this paper. They include physical treatment, biological treatment, and chemical treatment. In physical methods, mechanical disruption, high pressure (HP) process, pulsed electric field (PEF) application, ultrasonic treatment, and freeze-thaw, etc. were applied. In biological methods, different cell wall-degrading enzymes were applied to breakdown cell walls or membranes and to diminish the overall internal resistance for transporting bioactive compounds from internal matrix to the external solution. In chemical methods, various chemicals for increasing the inner- or outer-membrane permeabilization were introduced. The principles of the technologies, examples of

improvements, and advantages and disadvantages of the pretreatment methods were critically reviewed in this paper.

Keywords: Pretreatment methods, physical treatment, pulsed electric field, ultrasound, freeze-thaw, enzymatic treatment, chemical treatment.

1. Introduction

Nutra-pharmaceuticals, and bioactive or functional compounds from natural products have been used as medicinal agents by human beings for thousands years. There is no doubt that health-related product/process development has tremendous importance on the application of biofunctional materials in food industry. Although some bioactive compounds can be chemically synthesized (Thuong & Asseline, 1985; Ortholand & Ganesan, 2004; Nikolaev et al., 2007), the extraction of compound-rich biomaterials from natural resources still play a very important role in current separation technologies because of the tremendous diversity in molecular structure, complexity of synthetic methodologies, or high cost of chemical synthesis technologies (Giri & Lakshmi, 2004; Guo, 2008; Larghi et al., 2009). Furthermore, the extraction of pharmaceutical compounds from natural materials, especially plant or animal origin, has attracted the attention of researchers and industries due to the increasing market demands. Thus, extraction technology with high quality product, less energy consumption, and more environmental friendly operation is more spotlighted by pharmaceutical/functional food and cosmetic industries.

Continuous and active attempts were made to improve the mass transfer during the crude extraction process. In the industry, most popular extraction method is solid-liquid extraction,

which is an unsteady state mass transfer of multi-components from a solid matrix to a solvent. Some novel extraction technologies to improve the extraction process have been developed continuously. Those are supercritical fluid extraction (Mishra et al., 1993; Papamichail et al., 2000), microwave assisted extraction (Pan et al., 2000; Camel, 2000), accelerated solvent extraction (Richter, 2000; Breithaupt, 2004), and ultrasound assisted extraction (Vinatoru, 2001; Zhao et al., 2007), etc. During the extraction process of solid-liquid system, extraction occurs at a rate expressed in terms of change in solute concentration in the solid matrix within a unit of time. Generally, the transporting speed of solute through the solid matrix into the external solvent is a rate-controlling step. In order to maximize the yield of the novel extraction, a variety of pretreatments were used to increase the cell wall permeability. Currently, no comprehensive reviews on the pretreatment for effective extraction of bioactive components are available. In this review we dealt with various pretreatment methods with principles of the technologies, examples of extraction improvement, advantages and disadvantages of the technologies. The aforementioned pretreatment methods were divided into physical, biological, and chemical means to introduce respectively. At the beginning, physics related to solid-liquid extraction was also introduced to understand the extraction process better with pretreatment.

2. Fundamental physics behind extraction

Generally, the biomaterials from plant origin contain an intricate microstructure including cells, intra-cellular spaces, capillaries, and pores as shown in Figure 1. The desired solute (i.e.: bioactive compound) in the tissue may be present inter- or intra-cellularly. The molecular may pass through the pore between two neighbor cells and the solute transport from solid matrix to

the exterior solvent phase. Or the solute may move through the capillaries in the solid matrix into the bulk external solvent. During extraction, turbulence unlikely occurs in small capillaries and pores, leaving molecular diffusion as the main transport mechanism within the solid matrix. This so called molecular diffusion is the process by which molecules are transported by random movements from a high concentrated region to a lower one according to the concentration gradient (Equation 1, Simeonov et al., 1999).

$$\frac{\partial C_a(x,t)}{\partial t} = D_{eff}\,\frac{1}{x^{\alpha}}\frac{\partial}{\partial x}\left(x^{\alpha}\,\frac{\partial C_a(x,t)}{\partial x}\right) \qquad (1)$$

where α can obtain 0, 1 and 2 associated with plate, cylindrical and spherical geometries, respectively, and D_{eff} is the effective diffusivity in m^2s^{-1}.

After that, bioactive compounds inside the particles will move to the particle surface by diffusion along with concentration gradient inside the particles, the bioactive compounds will be convected away to the extraction solvent. There should be a mass balance at the boundary as below (Izadifar & Baik, 2008):

$$-\rho_p D_{eff}\left.\frac{\partial C_{a\alpha}(t)}{\partial x}\right|_{surface} = \rho_{sol} h_m\left(C^* - C(t)\right) = \rho_{sol}\, h_m\left(\frac{C_{a\alpha}(t)\big|_{surface}}{k} - C(t)\right) \qquad (2)$$

where C^* represents equilibrium solute concentration at the interface just in the fluid phase, in kg kg^{-1}, h_m is the mass transfer coefficient in ms^{-1} and k is partition coefficient of target solute between solid phase and fluid phase at the interface.

The most important physical parameter that governs solid-liquid extraction speed is diffusivity of biocompound, D_{eff}. Thus, the most significant resistance barrier for the diffusion process is the cell walls and membranes of plant materials. Unlike animal cells, the plant cells

have rigid and thick cell walls, where cellulose is the major component forming most of the supporting tissues and certain types of conducting cells. In cellulose, glucose monosaccharide units are combined by beta linkage forming the polymer straight and fibrous. They form microfibrils by hydrogen bonds and further fibrils. Moreover, cell walls are lignified to a different extent, further reducing the passage of molecules. For efficient extraction process, it is important to make the cell wall permeable. By doing that, the effective diffusivity of biocompound increases dramatically. A lot of research on increasing the permeability of cell wall had been done and in this paper we reviewed the methods under the categories of physical, biological, and chemical methods.

3. Physical methods

3.1 Mechanical disruption

Theoretically, the extraction time varies inversely with the square of the characteristic dimension of particles. The conventional method to break the cell walls of plant materials is reducing the particle size (characteristic dimension) into small ones. The cell wall can be disrupted with mechanical applications, such as cutting or grinding. Within a fixed unit mass of materials, the ruptured outer cells will also have a larger surface to volume ratio as particle size decreases. Thus, extraction rate also increases with surface area for mass convection.

Ma et al. (2009) ground Liuwei Dihuang, a Chinese herb, to the particle size of 161.9 nm using a high speed centrifuge sheering pulverizer. They found out the content of paeonol in extracts increased from 0.047 mg/g in micro particles to 0.058 mg/g in nano particles, which

indicated the extraction yield of paeonol jumped by 23.5% due to the breakdown of the plant cell walls.

However, size reduction is not always favored in the applications of extraction. When the particles are too small, it will bring a series of problems such as pressure drop in the extractor, slow drainage rates, contamination of fines in the extract, flow instability and entrainment. Therefore, the various novel treatment methods of cell wall breaking were introduced and discussed in the following content.

3.2 High pressure assisted process

3.2.1 Principles and mechanisms

Recently, the application of high pressure technology, generally high hydrostatic pressure (HHP), to inactivate pathogens in preserving food has raised great attention in food industry. It is generally regarded as one of promising emerging technologies to be adopted in the inactivation of vegetative microorganisms in the preservation of fruit juices, milk, seafood, and prepared meals without sacrificing organoleptic and functional properties of treated products (Ashie & Simpson, 1996; Dogan & Erkmen, 2004; Jung & Mahfuz, 2009). Plant cell walls can be also ruptured by HHP treatment for efficient extraction.

Generally, the high pressure pretreatment process includes three steps: compression, holding at the desired pressure, and pressure release. All of the sample, pressure medium, and a vessel are fixed at an initial temperature at the beginning of the experiment. During the compression, pressure increases up to the desired pressure with compressing fluid. The temperature normally increases by 2-3°C per 100 MPa as result of the physical compression depending on the composition of medium or food (Cheftel, 1995). There is no additional energy required during

the holding phase, where the sample is kept under the same pressure for extended periods of time. When the pressure is not applied, the compression induced temperature goes down to the initial value, resulting in volume expansion. Through these compression and expansion process, the application of HHP may bring the change in the pressure of cell membrane inside and outside, which pierces the holes on membranes or cell walls.

3.2.2 Practical issues for high pressure treatment

It is known that sublethal injury of the membrane for microorganisms or cells are influenced by the application of pressure. Much work has been done on the inactivation of microorganism, while limited reports are discussed and observed on the plant or animal cells widely used in food industry. With larger cell sizes than the microrganism cells, the plant or animal cells can be broken down at lower levels of the applied pressure. Their effects and experimental conditions are listed in Table 1.

Corrales et al. (2009) reported an optimization of anthocyanin extraction from red grape skins assisted by high hydrostatic pressure (HHP). The various levels at 200, 400, and 600 MPa of pressure were used in the experiment for finding the optimal anthocyanin extraction yield and strongest antioxidant activity. The highest antioxidant activity of the extracts at the optimum condition of 600 MPa and 30 min holding time were three-fold greater than that with the control extractions. And the extraction yield achieved was 23% higher than control.

Compared with conventional solvent extraction, ultrasonic extraction, and heat reflux extraction methods, the application of high hydrostatic pressure most efficiently promoted the extraction yield of caffeine from green tea leaves among (Xi, 2009). The extraction yield with

conventional method for 20 hours at room temperature was obtained by HPP at 500 MPa for only 1 min. The holding time for the formation of equilibrium between intra- and extra-cellular solutions to achieve complete contact between bioactive components and the solvent was tested from 1 to 10 min. The results showed that there was no significant increase in extraction yield with holding time, which indicated 1 min was enough to achieve the equilibrium, which made HHP methods as an energy efficient method in the extraction.

High-pressure was also applied to recover antioxidant and antityrosinase compounds from longan fruit pericarp (Prasad et al., 2010). The dried longan fruit pericarp powder (10 g) was mixed with 500 mL of 50% ethanol, and then pressurized for 30 min at 200, 300, 400 or 500 MPa, with dioctyl sebacate acting as pressure transmitting media. The highest total phenolic content obtained was 20.8 ± 1.9 mg/g at 500 MPa compared to 11.9 ± 1.2 mg/g for control experiment. It indicated that high pressure increased phenolic compound recovery approximately twice the control. In the meanwhile, the highest total antioxidant activity of the extract was obtained at 500 MPa.

Zhang et al. (2006) developed a new method of ultrahigh pressure extraction (UPE) to extract ginsenosides from *Panax quinquefolium L.* (American ginseng) root at room temperature. Different solvents, including water, ethanol, methanol, and n-butanol were used in the high pressure extraction, and ethanol showed to be the most efficient solvent. The yield of ginsenosides increased linearly with pressure in the range of 100 –500 MPa. There was slight decrease on the extraction yield when the pressure was higher than 500 MPa. Under the pressure of 200 MPa, no obvious increase in the yield of ginsenosides was observed within the period of 1-5 min. The extraction yield of 0.861% ginsenoside-Rc in 2 min at 25°C as achieved by the

UPE, which is much higher than that of 0.661% by heat reflux extraction at 70℃ for 6 hours. Therefore, UPE was proved to be the most efficient extraction methods compared with the other methods.

Trejo-Araya et al. (2007) reported the effects of high pressure processing on textural changes of fresh carrots integrating microstructural and biochemical responses. Analysis of microscopic images provided insight into the mechanisms of textural changes, which included cell deformation related factors such as shape factor and elongation. Pectin solubilization and the release of low molecular weight uronides from cell walls can relate to the softening of plant tissue. At 20°C, hardness losses of 5, 25 and 50% were found for treatments at 100, 200 and 300 MPa, respectively. Textural changes of fresh carrot tissue were mainly associated with turgidity loss, a direct result of the applied hydraulic pressure. Therefore, the application of high pressure can increase the cell wall permeability, which facilitates the extraction process.

High pressure can also be used in the cell destruction to create desirable functional properties. When endosperm cells present in rice grains were pressurized at 100–400 MPa, the partial destruction of endosperm cells enhanced their permeability to facilitate the preferential release of the major rice allergens (Aertsen et al., 2009).

3.2.3 Advantages and disadvantages of high pressure treatment

Compared with conventional treatment methods, HPP can be utilized as a promising tool either in fundamental research or in the development of new biotechnological applications. Within a very short process time, the high pressure application can achieve a higher extraction yield on a commercial basis. The minimal thermal process conveyed by high pressure shows great potential for different industrial fields, from food to pharmaceutical applications. The HHP can

be applied at room temperature successfully, and be regarded as a substitute technology for heat processing in microorganism inhibition. Furthermore, the high pressure processing presents the advantage of its uniform and instantaneous application, which is independent to the size and shape of the treated products (Knorr, 1993). While as being regarded as an environmentally-friendly, industrially-tested technology, high pressure also has its drawbacks: limited application as a batch or semi-continuous process and high cost of pressure vessels. The pressure adoption may turn a raw product (such as fruit) to a paste rather than remain its original shape because of the operation procedure: the first collapses by high compression and then suddenly expansion when withdrawing the pressure. Therefore, the fruit products are pressurized as "prepared product" rather than in natural presentation (Guerrero-Beltrán et al., 2005).

3.3 Pulsed electric field application

3.3.1 Principles and mechanisms

Through a phenomenon called irreversible electroporation, PEF is a promising method with a high capability of increasing cell membrane permeability, which is possible to increase the extraction yield during the compression of plant tissues or solvent extraction. The application of a moderate electric fields (between 0.1 kV/cm and 40 kV/cm for micro-seconds caused pores of limited size in cell membranes/walls due to increase in the transmembrane potential. The cell pores generated by PEF treatment can be reversible or irreversible depending on the intensity of PEF treatment (Serpersu et al., 1985). For the reversible pores on the membrane, the viability of the cell is still maintained, pores generated by electrical strength will be resealed, and the permeability of membranes decreases to the original level when the electrical field withdrew.

PEF receives a lot of attention and a variety of work has been done on the microbial inactivation for the pumpable liquid foods and beverages such as milk, pea soup, liquid egg yolk, green tea extracts, and red wine (Amiali et al., 2007; Zhao et al., 2008; Jaeger et al., 2009; Puértolas et al., 2010), and all kinds of juice of apple, orange, tomato, grape, mango, cranberry, etc (Jia et al., 1999; Liang et al., 2006; Marsellés-Fontanet & Martin-Belloso, 2007; Nguyen & Mittal, 2007; Evrendilek et al., 2008). Less work has been done on the use of electrical fields (PEF) for the modifications of the microstructure and texture of cells for the solid foods.

Figure 2 showed the basic diagram of pulsed electric field apparatus (Heinz et al., 2001). The main components of the PEF system are high voltage generator, charging capacitor, discharging switch and application (or treatment) chamber. The generator provides high voltage with the help of capacitor. The electrical energy is stored in the capacitor and the energy is released as a form of high voltage current to the samples between two electrodes for microseconds. For the thermo-labile bioactive compounds, a cooling device needs to be considered for application design due to potential heat generation during PEF treatment.

3.3.2 Practical issues for PEF treatment

The applied voltages are normally between 0.1 kV/cm and 40 kV /cm depending on the purpose of the treatment. The critical transmembrane potential to induce membrane permeabilisation is dependent on the size and geometry of cells exposed in the electric field. The smaller cells, the higher field strength is required. Generally, the size of vegetable or animal cells in the range of 40- 200 μm for food industry is greater than that of micro-organisms in the range of 1-10 μm. Thus, the critical electric field strength for cell membrane plasmolysis for plant cells

in around 1-2 kV/cm, which is much smaller than that of 12-20 kV/cm for microorganisms (Heinz et al., 2001).

Most of food materials will not tolerate the low intensity electric fields between 1-10 kV/cm and their texture or microstructure will be changed under this range (Gudmundsson & Hafsteinsson, 2005). Their effects and experimental conditions are listed in Table 2.

Dörnenburg & Knorr (1993) investigated the application of PEF at the strength from 0 to 1.6 kV/cm on cultured plant tissues of *Morinda citrifolia* cells and examined its effect on cell permeabilization. No distinct pigment release at 0.5 kV/cm showed the cell permeability was not improved under this level of electric field for the system. In the extraction of pigment from beetroots (Fincan et al., 2004), low intensity PEF treatment of 1 kV/cm with 270 rectangular pulses of 10 μs was applied. The extractability of pigments was shown to be approximately linearly proportional to the release of ionic species, so the researchers assumed relatively low levels of the cell damage occurred.

In Guderjan et al.'s work (2005), the effect of PEF treatment of dry milled maize on the extraction of phytosterol was observed in terms of cell disintegration index with different field strengths. The disintegration index steeply increased from 0 to 2 kV/cm, after then the index change with field strength was not significant. It is interesting that the disintegration of cells was more severe (29.7%) at field strength of 7.3 kV/cm, and the oil yield from maize germ did not increased accordingly. This indicated that the induction of stress reactions by a low PEF might stimulate metabolic activity and accumulate secondary metabolites.

When the electric field strength and/or treatment duration (such as number of pulses and pulse width) increases, the large pore will be generated and persisted even the PEF treatment is terminated. Then reversible permeabilisation will turn into irreversible breakdown.

According to Abidor et al.'s report (1979), the electrical breakdown for cells membranes at a low electric strength (<0.2 kV/cm) and short duration (10^{-5}–10^{-6} s) was reversible and the pores on the cell membranes could be recovered after the cease of the treatment. When the strength of electric field was higher and treatment duration prolonged (0.5-2 kV/cm, 10^{-4}–10^{-5} s), the pores persisted on the cell membranes and integrity of cells decreased.

Zimmermann et al. (1974) reported that reversible structural change of cell membranes occurred at an electric field strength of 1–10 kV/cm the with the short pulse duration of 20 ns–10 ms, while the irreversible rupture occurred when pulse duration prolonged to 10-15 ms (Zimmermann et al., 1976).

The impact of PEF at field strength of 0.6–1.3 kV/cm and pulse duration of 280 μs was studied to examine its effect on the extraction of oils from olives, maize, and soybeans (Guderjan et al., 2005). Irreversible disrupted cells cause a better and easier extraction/separation of food and biological ingredients. The maize germ oil yield increased by 2.9% compared with untreated control. For extraction of phytosterol in the maize germ oil, the yield rose up to 14.7% higher with the PEF treatment.

Whether reversible or irreversible, the pores generated by PEF will promote the mass transfer of bio-compounds from the solid samples. Lebovka et al. (2000) established a simplified model to simulate the kinetics of dielectric breakage of cells from thin apple slices under PEF treatment at field strengths of 0.2-2.2 kV/cm and pulse duration of 10-100 μs. The proposed

model included a “jamming” behavior, which was consistent with the experimental observations. The effect of electric treatment was more efficient at higher levels of field strength because of the associated higher cell destruction degree.

The incidence low pulsed electric field (0.1-1 kV/cm) on the diffusion characteristics of soluble substances from apple slices was investigated (Jemai et al., 2002). The diffusion coefficient of soluble substances was reported to increase after the electrical field application. The enhancement of diffusion coefficient was detected with the intensities of 0.1-0.15 kV/cm at 20ºC and 75ºC At a level of electrical intensity at 0.5 kV/cm and 100 μs duration, the diffusion coefficient was 3.9×10^{-10} m^2/s at 20°C for PEF treated sample, which was higher than the value of 2.5×10^{-10} m^2/s for untreated samples. And the diffusion coefficient was 13.4×10^{-10} m^2/s with PEF treatment, compared with 10.2×10^{-10} m^2 /s for thermally denatured samples at 75°C. The result indicated that PEF treatment increased the permeability through the apple tissue and improved the mass transfer.

In Kulshrestha & Sastry’s report (2003), the optimum mass transfer increase of beet dye from beet roots was obtained at the field strength of 10–20 V/cm. Reflecting the cell membrane damage, the concentration of betanin was 3.04 ppm with PEF, higher than 2.87 ppm at conventional extraction. When the electric strength was under 5 V/cm, no distinct enhancement was detected.

Lopez et al. (2009) tested the effect of PEF for the betanine extraction from red beetroot. Thin disc samples were treated with 5 pulses and 1 to 9 kV/cm (corresponding specific energies of 0.02 to 0.70 kJ/kg). With 5 pulses at 7 kV/cm, they found about 90% of total betanine was

released after 300 min extraction and the extraction rate was five times higher than that of non-treated samples.

With PEF pretreatment of carrots, apples and coconuts, there were significant increase by 10-40% in the yield of their juices and coconut milks (Gudmundsson & Hafsteinsson, 2005). For carrot processing for the coarse particles (~3.0 mm), the maximum juice yield of the control was 30%, while the yield increased to 70.3% with PEF treatment applied. For the fine particles (~1.5 mm), the PEF treatment raised the extraction yield from 51.3% to 76.1% (Knorr et al., 1994).

3.3.3 Advantages and disadvantages of PEF treatment

The low energy consumption, short process time, and moderate temperature make PEF treatment as a potential means to improve the extractability of valuable bioactive compounds from different food matrices at an industrial scale (Praporscic et al., 2007). In addition, the application of low-intensity PEF may be noteworthy to separate valuable compounds from metabolically active tissues at higher energy efficiency. The high-intensity PEF treatment can be a promising alternative to produce more fresh-like and safe pasteurized liquid foods than the conventional heat treatments.

However, the disadvantages of PEF technology such as non-uniform distribution field strength which is intrinsic characteristics of technology and limited selection pool of solvent with PEF might slightly reduce the feasibility of this method in some industrial implementation. Further investigation on the effect of PEF operating parameters on extraction of bioactive compounds needs to be extensively carried out.

3.4 Ultrasound irradiation

3.4.1 Principles and mechanisms

Ultrasound has a frequency higher than 20 kHz (Mason, 1990). It is another tool which can be effectively used to make the cell walls of plant based biomaterials more permeable to the solute. Unlike electromagnetic waves, sound waves must travel in matters since sound waves are mechanical vibrations. As passing through matters, sound waves impose expansion and compression cycles to the medium. In liquid, if the ultrasound frequency is high enough, the expansion cycle causes localized negative pressure. When the local negative pressure exceeds the tensile strength, bubbles or cavities are formed in the liquid. In a liquid-solid (biomaterial) system, adjacent to the solid boundary, the shape of cavity during collapse is asymmetric and high-speed jets of liquid are produced when bubbles are collapsed. The whole process from cavity formation to cavity collapse takes place within a very short time period, which may generate a powerful liquid jet when the cavity is closed to a solid surface. This asymmetric implosion induced liquid jet move through the bubbles to the surface at the speed of 400 km/h (Stephanis et al., 1997). The influence of the jets on the solid surface is so strong that serious damage to the solid surface is made. It is shown that ultrasonically induced cavitation will increase the permeability of the plant tissues. The rupture of cell walls identified by scanning electron microscopy in soybean (Li et al., 2004) and Radix Bupleuri (Zhao et al., 2007) proved the mechanical breakage by ultrasound and explained the extraction yield increase as result of more release of desired content from the solid matrices.

The apparatus of ultrasound treatment can be designed as bath or probe type. The process can be also designed as online or batch. The important operating parameters during ultrasound treatment are operating temperature, sonication duration and power, probe shape, vessel geometry, and process type (continuous or batch). To obtain the best extraction yield, the

ultrasonic wave distribution inside an extractor is also a key parameter. The maximum sonication power is observed in the vicinity of the radiating surface of the ultrasonic horn. As the distance from a radiating point increases, the ultrasonic intensity decreases abruptly. The presence of solid particles also attenuates the ultrasound intensity (Romdhane et al., 1995). The effect of ultrasound on extraction yield and kinetics also varies with the various nature of the plant material to be extracted.

3.4.2 Practical issues for ultrasound treatment

Plenty of ultrasound assisted extraction has been widely reported, while a limited number of ultrasonic pretreatment has been summarized and published. Table 3 outlined more details in the application of ultrasound as an effective pretreatment method prior to the extraction process.

Jiménez et al. (2007) investigated the effect of high-power ultrasound pretreatment for virgin olive oil extraction from olive paste. There were two treatments of ultrasound in the experiment: the direct sonication by a probe horn at the intensity of 105 W/cm^2 and frequency of 24 kHz, and indirect sonication in an ultrasound-cleaning bath at 150 W/cm^2 and 25 kHz, respectively. Direct sonication achieved a better oil extractability at high moisture content (>50%) of olives paste, whereas indirect sonication resulted in greater extractability at low moisture (<50%) olive fruits. According to the experimental result, the better product quality of extracted oils such as lower bitterness and higher content of tocopherols, chlorophylls and carotenoids, was obtained from sonicated pastes.

Shah et al. (2005) applied ultrasound as a pretreatment method before aqueous oil extraction from the seeds of *Jatropha curcas* L. The ultrasound assisted extraction provided 67% (w/w) of

oil yield after 10 min sonication in alkaline medium. And the extraction yield of 74% was obtained by ultrasound irradiation for 5 min with aqueous enzymatic oil extraction afterwards. The process period was shortened from 18 to 6 hours with ultrasound pretreatment. The positive effect of pretreatment by ultrasound in the aqueous enzymatic oil extraction from almond and apricot seed was also confirmed (Sharma & Gupta, 2006). Compared with the 75% (w/w) oil yield at 40°C for 18 hours by aqueous enzymatic oil extraction from almond seeds, the application of 2 min and 70 W ultrasound increased the extraction yield to 95% in only 6 hours. The scanning electron micrographs (SEM) image visually proved the disruption of cell walls by ultrasound application. Similar result was obtained in the oil extraction from apricot seeds.

High power ultrasound was also adopted in the treatment of corn slurry to increase liquefaction and saccharification for ethanol production (Khanal et al., 2007). The corn slurry samples were pretreated by ultrasound for 20 and 40 s at amplitudes of vibration ranging from 180 to 299 μm_{pp} (peak to peak amplitude in μm), and then followed by enzymatic reaction converting cornstarch into glucose. Compared with the untreated sample, the corn particle size decreased about 20-fold after the ultrasonic treatment, and the cell walls of the corn were ruptured according to scanning electron micrographs. The glucose release rate speeded up as much as three times for sonicated samples than that for the control group.

For the defatted soy flakes, the ultrasonic pretreatment prior to the extraction facilitates the protein and sugar release from the solid matrix (Karki et al., 2010). The ultrasound at high amplitude for 120 s could provide the highest increase yield of 50% for total sugar and 46% for protein. The irradiation of ultrasound only broke the cell walls identified by SEM images, also reduced particle size around 10 fold in comparison of untreated sample. The new generating

surface explosion to the medium solvent also increase the contact for protein and sugar to the solvent, which speed up the extraction rate and increase the extraction yield. The result showed that the ultrasound application also can reduce the overall cost of producing soy protein from flakes.

In the recent years, the application of high power ultrasound to increase the biodegradability of the sludge as an emerging pretreatment method has raised more attention (Pilli et al., 2010). Ultrasonication is proved to be a very effective mechanical method to enhance the sludge digestibility by disrupting the properties of the sludge, which is very meaningful to treat sewage sludge in all wastewater treatment plants. The sonication parameters and sludge characteristics determine the degree of disintegration of sludge. The optimum parameters for the best effect vary with sludge characteristics and the ultrasonication reactor system.

3.4.3 Advantages and disadvantages of Ultrasound treatment

High power ultrasound has been widely used in food industry for its low cost on apparatus, simple operation, and effectiveness in increasing yield. The application of ultrasound promotes the extraction yield and speeds up the extraction rate in solid-liquid extraction. It can also reduce the temperature and time of operation, which is specially preferred for the extraction of the thermo-liable compounds. Compared with other techniques such as high pressure application, the ultrasound apparatus is much cheaper and easier to operate. The energy consumption was less concerned in the domain of ultrasound pretreatment. Through Zhang et al.'s (2011) report, ultrasound seems to be considered as an energy-efficient technique in silica gel regeneration at low temperature. Under experimental conditions of 35°C and moisture ratio at range of 0.15-0.3 at dry basis, the total specific energy consumption was 38-40 MJ/kg and18-20 MJ/kg for

conventional thermal regeneration and ultrasonic treatment at 60 W, respectively. The lowest total specific energy consumption was achieved at 55°C and 60 W. Furthermore, unlike PEF, there is no restriction of the solvent type in the extraction, which provides its application to a wide variety of natural compounds.

However, the effectiveness of ultrasound associates closely with the nature of products and the extraction system. The ultrasound wave may be attenuated by the shape of the extractor or presence of solid particles. The distribution of ultrasound wave is non-uniform and restricted to the area in the vicinity of ultrasonic probe, which brings the challenge in the application of a very large industrial scale.

3.5 Freeze-thaw process

3.5.1 Principles and mechanisms

Takamatsu and Kumagae (2002) demonstrated that biological cells may be injured not only by chemical damages but also by mechanical damages which are caused by ice crystal compression. Freezing rate can be adjusted so that relatively large ice crystals grow in the biological material. Griffiths et al. (1979) investigated Chinese hamster ovary (CHO) cell size during the cooling, warming and post-thawing periods of the freeze-thaw cycle. The cells were cooled at 1 °C/min or 200 °C/min and subsequently thawed, and its structure change was studied with a cryomicroscope. The cells shrank significantly and no intracellular ice appeared at the cooling rate of 1 °C/min, while partial intracellular ice formation occurred and cells containing most ice shrank least at 200 °C/min cooling rate. In thawing process, the cells swelled and their

size was larger than that of non-frozen controls at slow cooling rate, cells contained intracellular ice swelled to a greater extent at fast cooling rate.

Where the freezing rate is not fast, the number of ice crystals will be small but large in size. These large crystals bring the alteration of plasma membrane due to mechanical compression, and the cells may not tolerate subsequent swelling leading to cell damage during thawing process. It is believed that the elevated concentration of extracellular solute mainly causes the cellular injury. However, the investigation of membrane alteration and damage at microscale level still needs to be further conducted (Takamatsu & Zawlodzka, 2006). Drying evacuates the water from such damaged biomaterials and can induce more fine pores within the product. The desired solute will be promoted to release from the solid particles because of the cell rupture by freeze-thawing process.

Therefore, freeze-thaw process can be considered as an approach to damage biological cell walls by mechanical compression.

3.5.2 Practical issues for freeze-thawing treatment

The fresh fruits and vegetables contain more water and their cell walls are less elastic than cell membranes of animal cells (Mohsenin, 1986), which is more ease to form large ice crystal and cause inevitable cell damage in freezing process.

Modise (2008) investigated the effects of various freezing and thawing treatments on the volatile profile of strawberries. The freezing was carried out at −20°C or −80°C or rapidly frozen in liquid nitrogen (−196°C). After one night or a week, berries were later left to thaw at room temperature for natural thawing, and some were forced-thawed in a 30°C water bath. In the extraction process, the concentration of most esters such as hexyl acetate, ethyl methyl

hexanoate, methyl acetate were increased significantly by week-long freeze/thaw treatment in comparison of fresh berries. It was found that more acetaldehyde compounds exist in forced-thawed berries than naturally thawed samples.

Oszmiański et al. (2009) studied the stability of phenolic compounds of strawberry cultivars at various freezing and thawing conditions. 4.5–33.6% of polyphenols were lost after prefreezing treatment. In thawing process, the forced-thawed strawberries in a microwave oven had a further positive effect on retention of (+)-catechin and ellagic acid, in comparison of natural thawing process at 20°C for 20 hours.

3.5.3 Advantages and disadvantages of freeze-drying treatment

The freeze-thaw process provides a clean alternative to break the cell walls to improve the extraction yield in food extraction processes. After the pretreatment, there is a minimal change on color or flavor, and it keeps most of the nutrients. With the increasing concern about food quality, this process could be considered as a valuable alternative to pretreat the food for a better quality in the extraction of valuable compounds from food materials.

Currently, this freeze-thaw process is not widely used for the pretreatment in the food industry due to its high operating costs and long processing time.

4 Biological methods

4.1 Principles and mechanisms

Most of plant cell walls contain about 10% protein and the other large portion is made of polysaccharides including cellulose, hemicellulose, and pectin (McNeil et al., 1984). The

component composition of polysaccharides varies with the variety of the raw plant materials. For example, the proportion of polysaccharides is 22% for cellulose, 29% for semicellulose, and 39% for pectin in rapeseeds (Domínguez et al., 1994). However, there is approximately 30% cellulose, 30% hemicellulose and 35% pectin in grasses (Carpita, 1996; Cosgrove, 2007). The enzymes which hydrolyze these components can be used to improve extraction of bio-component inside the plant materials as a pretreatment. By disintegrating the cell wall with enzymatic reaction, the increased permeability of cell wall will enhance the extractability of desired solute from the broken cells.

The important parameters which should be considered for the enzyme treatment are reaction time, temperature, pH, particle size of raw material, type and concentration of enzyme. The optimum temperature for the treatment related to thermal kinetics of molecular movement and protein denaturation. Normally, the optimum temperature lies between 35°C and 65°C in the enzymatic pretreatment for extraction of oils from fruits and oilseeds (Domínguez et al., 1994).

4.2 Practical issues for enzymatic treatment

One of the important applications of an enzymatic pretreatment is to increase the yield of edible oil from the fruit and oilseeds. Domínguez et al. (1994) summarized the enzymatic pretreatment for oil extraction from olive, avocado, coconut, sunflower, soybean, rapeseed, and palm, etc. The effects of reaction time, temperature, pH, particle size, dilute ratio, centrifuge speed, and enzyme concentration have been compared. It was shown that the effect of enzymatic pretreatment was significant on the oil extraction yield. High quality oil was extracted and not influenced by the type of enzymatic reaction. But the enzymatic reaction depended on the characteristic of seeds and the shape of cultivar. The extraction yield of oil from grape seeds

increased 106% after the enzymatic treatment compared to non-treated samples (Passos et al., 2009). With the prolonged reaction time with enzyme from 24h to 120 h, the extraction yield increased from 13.7% to 17.5%, which presented 163% increment to the samples without enzymatic pretreatment.

The enzymatic reaction can be also applied to break cells in the fruits and promote the juice production. An enzyme cocktail of pectinases and cellulases has been reported to give a juice yield up to 100% (Alkorta et al., 1998). The function of various types of pectinases in the fruit juice production was discussed (Kashyap et al., 2001). The cloudiness and bitterness of fruit juices can be reduced by adding the acidic pectinases coming from fungal sources, especially *Aspergillus niger*. The alkaline pectinases, mainly from *Bacillus* spp., was generally used in the pretreatment of pectic wastewater from fruit juice industries, and in the textile industry for the retting and degumming of fiber crops.

4.3 Advantages and disadvantages of enzymatic treatment

Enzymatic pretreatment can be done effectively at moderate temperature (Domínguez et al., 1994). The method needs no specific requirement on apparatus and can be used in continuous production at industrial scale. Enzymatic reaction can occur in mild condition, which is preferred from the view point of energy saving. After the enzymatic reaction finishes, part of cell walls are disintegrated, leaving primary frame without considerable chemical contamination. The quality of extracts, such as oil, will not be affected by adding enzyme in the system, making the enzymatic pretreatment as a clean, pollution control, and safe method.

The main disadvantage of the enzymatic pretreatment is the sensitive properties of enzymes. The enzymatic reaction will be affected by various conditions, not only by pH and temperature, but also the characteristic of raw materials, solvent, and environment, etc. Some enzymes are expensive or unstable to the environment. All these need to be further developed or improved in practical application of enzymatic treatment.

5 Chemical methods

5.1 Principles and mechanisms

As previously mentioned, the major components in the cell walls are polysaccharides, which are also targeted as sugar sources for biofuel production. Through various chemical pretreatments, such as acid and alkaline hydrolysis, the molecular structure of cell walls may be degraded. The effects of chemical treatments for cellulose microfibrils isolated from banana rachis were evaluated by means of SEM, ion chromatography, ATR-FTIR, TEM, and electron and X-ray diffraction (Zuluaga et al., 2009). The morphology and structure of the SEM images of treated samples showed that the constituents like pectins and hemicelluloses were hydrolyzed by the action of alkaline solutions, while the removal of lignin needed additional steps of sodium chloride or hydrogen peroxide treatment. Acid was applied as a catalyst to break down heterocyclic ether bonds between sugar monomers in the polymeric chains, which are formed by hemicellulose and cellulose (Laopaiboon et al., 2010). By changing molecular structure of the lignin with chemicals, cellulose and/or hemicellulose will be more accessible for further processing. Ozone and peroxide can also be used in oxidation and reaction with organic

compounds by attacking the cell walls and outer membrane, especially for bacteria, which may bring the damage on cells.

5.2 Practical issues for chemical treatment

Chemical pretreatment, utilizing acids, alkali, or organic solvents, has been widely used to cleanse cellulose of lignin and hemicellulose in the natural fiber or for textile treatment (Thomsen et al., 2006). The effects of chemical pretreatment for the plant tissues are listed in Table 4.

Sun et al. (1995) investigated the effects of alkaline and oxidizing agents as pretreatments of wheat straw at various temperatures and exposure times. 60% and 80% of lignin and hemicellulose were released through delignification and dissolution with the pretreatment of 1.5% sodium hydroxide (NaOH) for 144 hours at 20°C.

Yamashita et al. (2010) also used sodium hydroxide as a pretreatment to enhance the digestibility of the holocellulose component in bamboo. Holocellulose components are covered with the rigid lignin in bamboo cells. Because of the poor accessibility of enzyme and digestibility of these holocellulose components, the pretreatment to degrade or remove the rigid lignin is necessary to promote the production of sugars by enzymatic saccharification. Through the pretreatment of 20 atm steam explosion and 10 wt.% sodium hydroxide treatment at 121°C, maximum value of glucose production 456 mg/(g initial dry sample) of glucose and 460 mg/(g initial dry sample) of reducing sugar was obtained. However, the pretreatment of combining 1% (v/v) hydrogen peroxide and 1 wt.% sodium hydroxide at 90°C for 60 min also introduced as much as 399 mg/(g initial dry sample) glucose and 568 mg/(g initial dry sample) reducing sugar production without severe conditions of high pressure and temperature for steam explosion. It

was shown that alkaline peroxide pretreatment was an effective and environmentally friendly method for the enzyme saccharification of bamboo.

The acid hydrolysis of sugarcane bagasse for lactic acid production was studied by Laopaiboon et al. (2010). In their work, the lignin was firstly removed by NH_4OH, then the remaining solid was hydrolysed by 0.5%, 2%, 3%, and 5% (v/v) of HCl or H_2SO_4 at a range of reaction time (1–5 hours) and incubation temperature (90–120°C). The optimal catalytic efficiency could be obtained with pretreatment of 0.5% of HCl at 100°C for 5 hours, on 89% xylose as the main fermentable sugar in the hydrolysate.

Silverstein et al. (2007) compared the effectiveness of various chemical pretreatments on the saccharification of cotton stalks to ethanol. The pretreatment methods include sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and ozone (O_3) treatment. There was a positive linear relationship between xylan solubilization and pretreatment severity. The pretreatment with sulfuric acid at 2% at 121 °C/15 psi for 90 min, resulted in substantial solubility of xylan up to 95.23% in cotton stalks, but the cellulose to glucose conversion was lowest as 23.85%. Delignification was the most significant effect of sodium hydroxide pretreatment. The highest level of delignification of 65.63% was achieved with 2% NaOH treatment at 121 °C/15 psi for 90 min. Sodium hydroxide pretreatment also showed higher cellulose conversion at 60.8%. However, hydrogen peroxide pretreatment resulted in lower lignin and xylan solubilization than expected. 29.51% delignification and 49.8% cellulose conversion were obtained with 2% hydrogen peroxide pretreatment at 121°C/15 psi for 30 min. The decomposition of hydrogen peroxide to water at high temperatures may reduce the effect of pretreatment. Not as expected, ozone did not cause any significant changes in lignin, xylan, or

glucan contents over time. Possible explanations include insufficient time, low ozone concentration, or uneven distribution of ozone throughout the sample. In other works (Tanaka et al., 1997; Sakai et al., 1997), the barely degradable compounds can be transferred into more easily degradable ones in the digestion of waste activated sludge using ozone, acids or alkali as the chemical pretreatment.

5.3 Advantages and disadvantages of chemical treatment

The merit of easy operation, cheap and abundant source of chemicals, effectiveness in cellulose or lignin degradation has made the chemical pretreatment as a convention method to make the cell walls more accessible for further degradation. However, chemical pretreatments also have serious disadvantages: specialized corrosion-resistant equipment is required; the chemical wastes will exist in the system and cause contamination; additional washing and separation to remove the chemicals needs to be accompanied and makes the process more complicated.

6 Other possible methods

The steam explosion treatment is a well-known way of disrupting various lignocellulosic plant materials into cellulose, hemicellulose, and lignin (Saddler et al., 1993). At a high pressure and temperature, steam was used to hydrolyze the cell walls components in plant materials. Then a sudden reduction of the pressure for the hydrolyzed products led to generate the low-molecular weight substances from highly lignocellulosic biomass (Vignon et al., 1995). Chemical effects and mechanical forces are combined in this pretreatment. The formation of acetic acid from acetyl groups present in hemicellulose was promoted by the hydrolysis (autohydrolysis) at high

temperatures. The explosive decompression of pressure causes the mechanical effects on fibers and facilitates the production of small molecular compounds. The reports showed steam explosion can be recognized as one of the most efficient pretreatments for hardwood and agricultural residues (Excoffier et al., 1991; Moniruzzaman, 1995), while less effective for softwoods (Clark & Mackie, 1987). Similar to the steam explosion pretreatment, ammonia/CO_2 explosion is the other type of physico-chemical pretreatment for lignocellulosic materials, such as recycled paper mix, sugarcane bagasse, barley straw, corn stover, and rice straw (Vlasenko et al., 1997; Zheng et al., 1998). The application of high pressure and temperature in these methods increases the requirement of specific equipment and cost of operation.

Microwave (MW) dielectric heating is widely applied in the food industry because of its high energy efficiency and short processing time. Different from the conventional heating method, microwave heats the whole sample simultaneously by absorbing and converting MW energy directly into heat. Weak hydrogen bonds can be disrupted through the promotion of the rotation of molecular dipoles such as water with an alternating (2450 million times/s) electric field. Microwave can heat wet biomaterials quickly, resulting in the occurrence of steam explosion when the stream pressure development by microwave irradiation is faster than pressure release to outside the cells. The movements of dissolved ions also enlarge solvent penetration into the matrix and thus facilitate the desired solute dissolved into the solvent. Ooshima et al. (1984) demonstrated that the microwave treatment played a positive role in biomass digestion and largely increased the accessibility of the cellulose materials. Ma et al. (2009) also reported that silicified waxy surface of rice straw was disrupted by microwave pretreatment through the chemical composition analysis. The application of microwave resulted in the breakage of lignin–

hemicellulose complex and partially removal of silicon and lignin. Microwave has been widely applied in the extraction of bioactive compounds from the natural materials, with its advantages in terms of less required solvent, shorter process time, better products, and lower costs.

Radio Frequency (RF) heating is also an emerging heating technology which provides uniform volumetric internal heat generation within particles placed between its two electrodes. Different from microwave (MW), RF has larger penetration depth, up to several meters, into the solid samples, which makes it suitable for industrial applications where bulk materials are treated. The amount of electromagnetic energy, which is transferred into biomaterial between the electrodes in the form of heat, relies on dielectric properties of the biomaterials, the clearance between electrodes, the RF supplied voltage and frequency. Despite low frequency, if biomaterials are soaked by an appropriate ionic solution in order to reach a suitable dielectric loss factor and small clearance between electrodes with a sufficiently high voltage is applied to the biomaterials, within a short period of time the temperature of the whole biomaterial can reach high enough to produce steam within cells. A preliminarily test which was performed for rhizome particles of *Podophyllum peltatum* containing podophyllotoxin (Izadifar & Baik, 2008) indicated that a packed bed of the particles soaked with ethanol could reach 70°C in 4 seconds. For some poorly or slowly germinating seeds, the application of RF can increase the seed-coat impermeability, but avoid the damage on seed by mechanical abrasive process (Nelson, 1985).

7 Hybrid processes

Toepfl et al. (2005) compared the effects of the various pretreatments, including PEF, mechanically pressurized, enzymatic, and freeze-thawing method, on the total permeabilisation of apple and potato tissues. PEF treatment required the lowest energy input in the range of 1-5

kJ/kg. In the contrast, the energy consumption of 20-40 kJ/kg were associated with mechanical, 60-100 kJ/kg for enzymatic, ~250 kJ/kg for thermal, and ~280 kJ/kg for freezing-thawing method, respectively. From the view point of energy saving, PEF may be a good choice for the pretreatment of solid materials for the extraction. However, non-uniform distribution field strength of PEF may reduce the popularity of the technique.

There seems no perfect method that can satisfy all the requirements yet. Thus, hybrid applications of each method might be alternative solutions. For example, the hardwood or agricultural stems can be processed by steam explosion to break down supporting frameworks in the cell walls. Then chemical pretreatment, especially alkaline method can be applied to destroy the structure of the lignin in cells, or at least make them more accessible for the further hydrolysis. There have been more and more reports on emerging hybrid processes to achieve both effectiveness and efficiency. Combinations of ultrasonication and enzymatic hydrolysis (Bermejo et al., 2004) promote cell disruption efficiency significantly. Sun & Chen (2008) reported the improvement of the enzymatic hydrolysis of lignocellulosic biomass by aqueous glycerol pretreatment. The enzymatic hydrolysis yield was obtained up to 90% in 48 hour for the pretreated fiber of wheat straw. The fibrolytic enzyme activity of carboxymethyl cellulase (CMCase) and avicelase activity on the rice straw was tested (Chen et al., 2008). The activities of both CMCase and avicelase are promoted by adding sodium hydroxide (SH), which means the enzymes favor more in alkaline environment in their study. With alkaline/oxidative (A/O) pretreatment and the enzymatic hydrolysis of aquatic plants sugar production was achieved three times more than that of untreated samples (Mishima et al., 2006). The application of ultrasound, microwave, or steam explosion, can also facilitate the enzymatic hydrolysis of lignocellulosic

biomass to get a higher yield in a shorter time period (Alvira et al., 2010). In food freezing–thawing cycles, the application of high-pressure in freezing can provide uniform and rapid ice nucleation through the whole sample, while microwave and ultrasound can also offer a quick thawing process to improve the overall product quality of vegetables and fruits (Li & Sun, 2002).

8 Summary and concluding remarks

Active researches have been conducted to study the effects of different pretreatments on cell disruption. Each method reviewed in this paper has its own technical advantages and disadvantages.

From the viewpoint of industrial application, the energy consumption, processing time, process mode (batch or continuous), and cost of operation are very important to make the technology commercially feasible. Except freeze-thawing process, the physical treatments such as HP, PEF, and ultra-sonication seem valuable technologies to fulfill the current demand for energy saving.

Furthermore, the methods including HP, PEF, ultrasound, and freeze-thaw can be operated at lower temperature. The processing time for these methods in the magnitude of second to min is also very competitive in comparison of the process duration in hours for enzymatic or chemical treatment. These physical pretreatment methods appear to be effective alternatives because they will not produce any harmful residues by adding extra materials into the system, which may bring pollution issues. During HP, PEF, freeze-thaw pretreatments, most of bioactive compounds are intact and causing minimal degradation of color or flavor, which shows promising potential as better food quality processing techniques.

Nevertheless, the physical methods have their own intrinsic disadvantages that have prevented their popularity. The disadvantages include high initial capital costs for high pressure applicators in HP application, limited choice of solvent type in PEF application, high energy consumption in freeze-thawing application, or non-uniform energy distribution in ultrasonication. The effectiveness of these techniques also depends on the characteristic of raw materials. Most of reports about HP or PEF pretreatment are focused on the physical changes of soft tissues of vegetables, fruits or meat. There is little publication concerning HP, PEF, or freeze-thaw treatments of rigid plant tissues. Although chemical methods may draw attention to environmental issues, their high effectiveness of cell wall disruption on plant fibers still makes it available as a valuable pretreatment. For those highly lignified tissues, the enzymatic or chemical pretreatments are more adequate methods to apply. A good understanding of each technique is essential to select proper methods according to specific purposes for extraction in the food or bioprocess.

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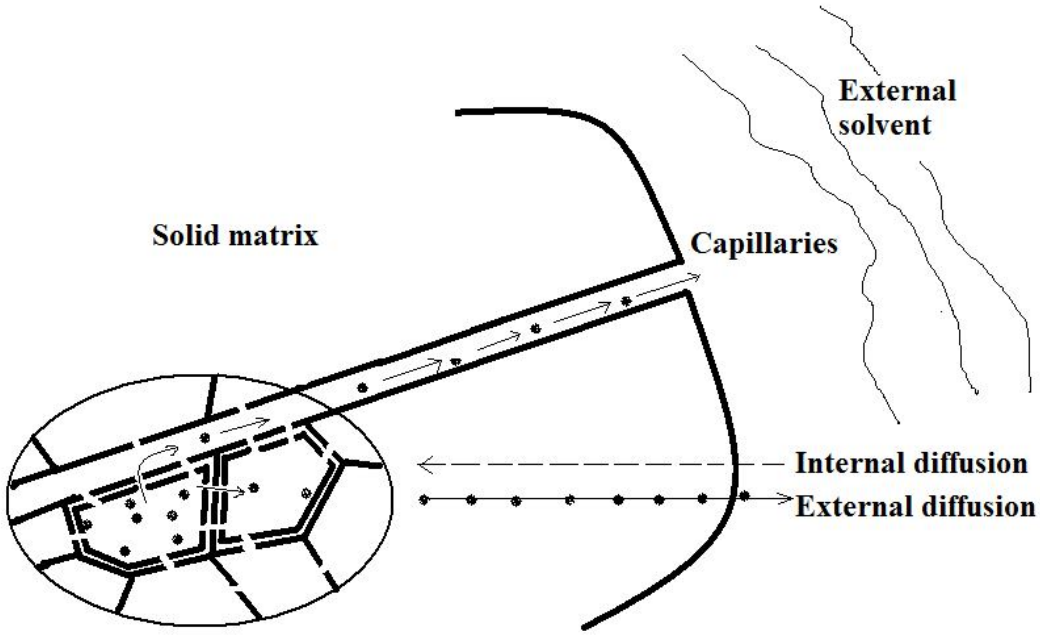

Figure 1. Scheme of the mass transfer in solvent extraction of solid food materials.

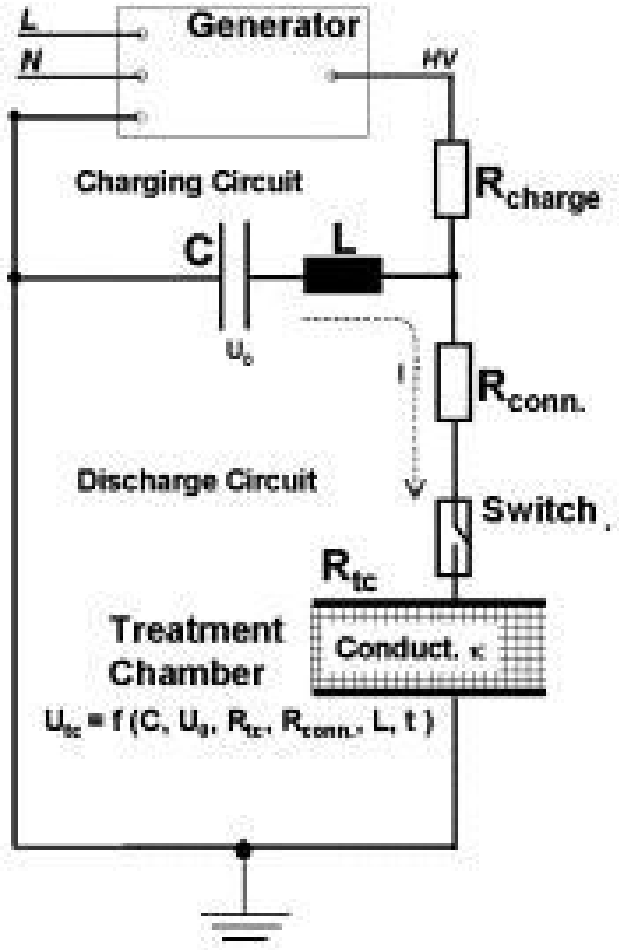

Figure 2. A schematic diagram of pulsed electric field application system (from Heinz et al., 2001)

Table 1. The effect of high pressure on the soft plant cells

Plant tissues	Process conditions			Effects	References
	Pressure (MPa)	Processing time (min)	Temperature (ºC)		
Red grape skin	200-600	30-90	20-70	Increased extraction	Corrales et al

Plant tissues	Process conditions			Effects	References
	Pressure (MPa)	Processing time (min)	Temperature (ºC)		
				yield (3-fold greater than control)	(2009)
Green tea leaves	100-500	1-10	Room temperature	Much shorter extraction time than that of conventional extraction (20 h to 1 min)	Xi (2009)
Longan fruit pericarp	200-500	2.5-30	30-70	Doubled phenolic compound recovery	Prasad et al. (2010)
Panax quinquefolium L. (American ginseng) root	100-600	1-5	25	Much higher extraction yield (0.8661% to 0.661%) and shorter time (8 h to 2 min) than that of heat reflux extraction.	Zhang et al. (2006)
Fresh carrots	100-550	2-30	20	Cell wall breakage as a result of the applied hydraulic pressure (50% loss in hardness)	Trejo Araya et al. (2007)
Rice endosperm cells	100-400	15-60	60	Enhanced the cell permeability and thus facilitated the release of rice allergens	Aertsen et al. (2009).

Table 2. The effect of pulsed electric field on the soft plant cells

Plant tissues	Process conditions			Effects	References
	Field strength (kV/cm)	Pulse duration (μs)	Pulse number		
Morinda	0-1.6	--	0-30	The cell permeability was	Dörnenburg &

Plant tissues	Process conditions			Effects	References
	Field strength (kV/cm)	Pulse duration (μs)	Pulse number		
citrifolia cells				not improved under 0.5 kV/cm	Knorr (1993)
Beetroots	1	10	24, 54, 270	Low level cells damage occurred	Fincan et al. (2004)
Maize germ	0.6-7.3	280	120	Increase in oil yield (23.2% to 43.7%) and phytosterol content (785 mg/100 g to 1039 mg/100 g oil)	Guderjan et al. (2005)
Apple slices	0.2-2.2	10-100	1-100,000	Higher cell destruction degree at a higher level of field strength	Lebovka et al. (2000)
Apple	0.-1	100	1000	Enhanced permeability and improved mass transfer (2.5×10^{-10} m^2 /s to 3.9×10^{-10} m^2 /s)	Jemai et al. (2002)
Beetroots	0-23.9	--	--	Improved the mass transfer (betanin concentration: 2.87 ppm to 3.04 ppm)	Kulshrestha & Sastry (2003)
Red beetroot	1-9	2-5	5-40	Five times higher extraction rate than control	Lopez et al. (2009)
Carrots	0-2.6	--	--	Enhanced juice yield (30% to 70.3%)	Knorr et al. (1994)

Table 3. The effect of ultrasound as a pretreatment method on food materials

Plant tissues	Process conditions	Dissipated Power (Watt)	Effects	References

	Frequency (kHz)	Processing time (min)	Temperature (°C)			
Olive paste	24 or 25,	0-30	20-35	---	Better properties of extracted oils (Bitterness: 4.0 to 2.4).	Jiménez et al. (2007)
Seeds of Jatropha curcas L	42	5-15	37-50	---	Shorten the process time (18 h to 6 h).	Shah et al. (2005)
Almond and apricot seed	42	2.5-15	40	---	Increased Oil yields (75% to 95%) and shorten extraction time (18 h to 6 h)	Sharma & Gupta (2006)
Corn slurry	20	1/3-2/3	10	Low: 274 ± 5 High: 475 ± 15	20-fold decrease in the corn particle size And 3-fold increase in the glucose release rate	Khanal et al. (2007)
Defatted soy flakes	20	0.25-2	4	Very low: 154 ± 1.5 High: 1280 ± 21	Broke the cell walls, and reduced particle size around 10-fold.	Karki et al. (2010)

Table 4. Chemical pretreatment on plant tissues

Plant	Process conditions	Effects	References

	Chemicals	Processing time (h)	Temperature (°C)		
Wheat straw	H_2O_2, NH_4OH, $Ca(OH)_2$, KOH, LiOH, NaOH,	0.5-144	0-80	Effective delignification with the pretreatment of 1.5% NaOH (80% hemicellulose removed)	Sun et al. (1995)
Bamboo	NaOH, H_2O_2	1	90 & 121	Enhanced the digestibility of the cellulose (84.3% to 90.1%)	Yamashita et al. (2010)
Sugarcane bagasse	NH_4OH, HCl, H_2SO_4	1-5	90-120	Obtained the optimal catalytic efficiency (up to 10.85%) with 0.5% of HCl at 100°C	Laopaiboon et al. (2010)
Cotton stalks	H_2SO_4, NaOH, H_2O_2, O_3	0.5-1.5	4, 90 & 121	Sodium hydroxide pretreatment resulted in higher cellulose conversion than acid pretreatment (60.8% to 23.85%).	Silverstein et al. (2007)

