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Review of Green Food Processing techniques. Preservation, transformation, and extraction

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A B S T R A C T

This review presents innovative food processing techniques and their role in promoting sustainable food industry. These techniques (such as microwave, ultrasound, pulse electric field, instant controlled pressure drop, supercritical fluid processing) in the frontiers of food processing, food chemistry, and food microbiology, are not new and were already used for > 30 years by academia and industry. We will pay special attention to the strategies and the tools available to make preservation, transformation and extraction greener and present them as success stories for research, education and at industrial scale. The design of green and sustainable processes is currently a hot research topic in food industry. Herein we aimed to describe a multifaceted strategy (innovative technologies, process intensification, bio-refinery concept) to apply this concept at research, educational, and industrial level.

Industrial relevance: Green Food Processing could be a new concept to meet the challenges of the 21st century, to protect both the environment and consumers, and in the meantime enhance competition of industries to be more ecologic, economic and innovative. This green approach should be the result of a whole chain of values in both senses of the term: economic and responsible, starting from the production and harvesting of food raw materials, processes of preservation, transformation, and extraction together with formulation and marketing.

1. Introduction

Food products, such as fruit and vegetables, fat and oils, sugar, dairy, meat, coffee and cocoa, meal and flours, are complex mixtures of vitamins, sugars, proteins and lipids, fibres, aromas, pigments, antioxidants, and other organic and mineral compounds. Before such products can be commercialized, they have to be processed and preserved for food ready meals and extracted for food ingredients. Different methods can be used for this purpose, e.g. frying, drying, filtering, and cooking. Nevertheless, many food ingredients and products are well known to be thermally sensitive and vulnerable to chemical, physical and microbiological changes. Losses of some nutritional compounds, low production efficiency, time- and energy-consuming procedures (prolonged heating and stirring, use of large volumes of water…) may be encountered using these conventional food-processing methods. These shortcomings have led to the use of new sustainable "green and innovative" techniques in processing, pasteurization and extraction, which typically involve less time, water and energy, such as ultrasound-assisted processing, supercritical fluid extraction and processing, microwave processing, controlled pressure drop process, and pulse electric field. The tremendous efforts made on greening food process can be evaluated through the consideration of books and journals devoted to these aspects (Chemat, Huma, & Khan, 2011).

Food technology under extreme or non-classical conditions is currently a dynamically developing area in applied research and industry. Alternatives to conventional processing, preservation and extraction procedures may increase production efficiency and contribute to environmental preservation by reducing the use of water and solvents, elimination of wastewater, fossil energy and generation of hazardous substances. Within those constraints, "Green Food Processing" has to be introduced on the basis of green chemistry and green engineering: "*Green Food Processing is based on the discovery and design of technical processes which will reduce energy and water consumption, allows recycling of by-products through bio-refinery, and ensure a safe and high quality product*" (Fig. 1).

This review presents a complete picture of current knowledge on Green Food Processing techniques for preservation, transformation and extraction as success stories for research, education and at industrial scale. The readers like chemists, biochemists, chemical engineers,

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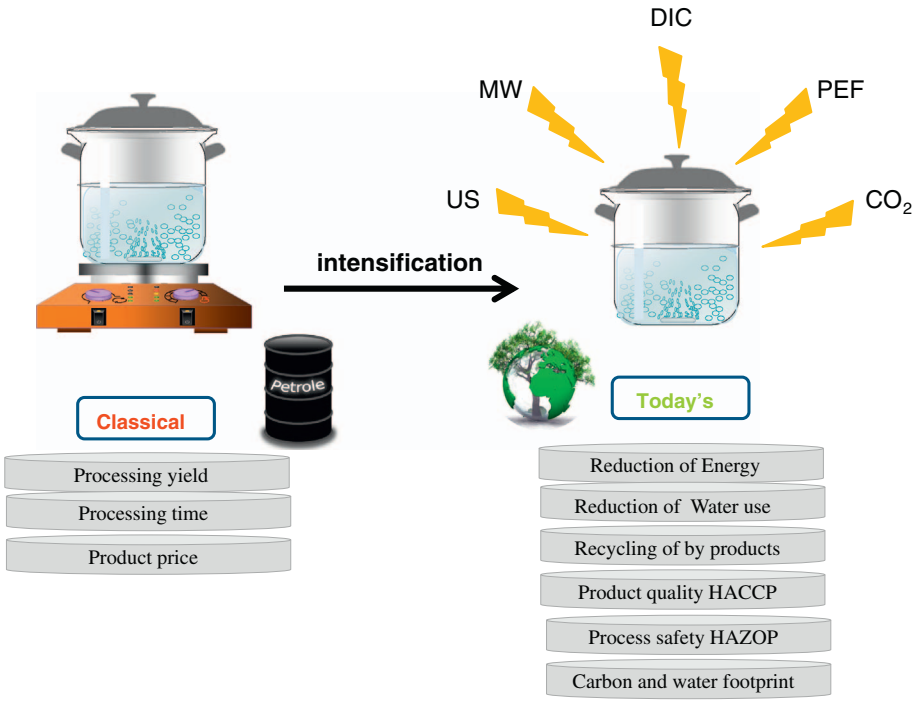


Fig. 1. Green Food Processing: evolution or revolution.

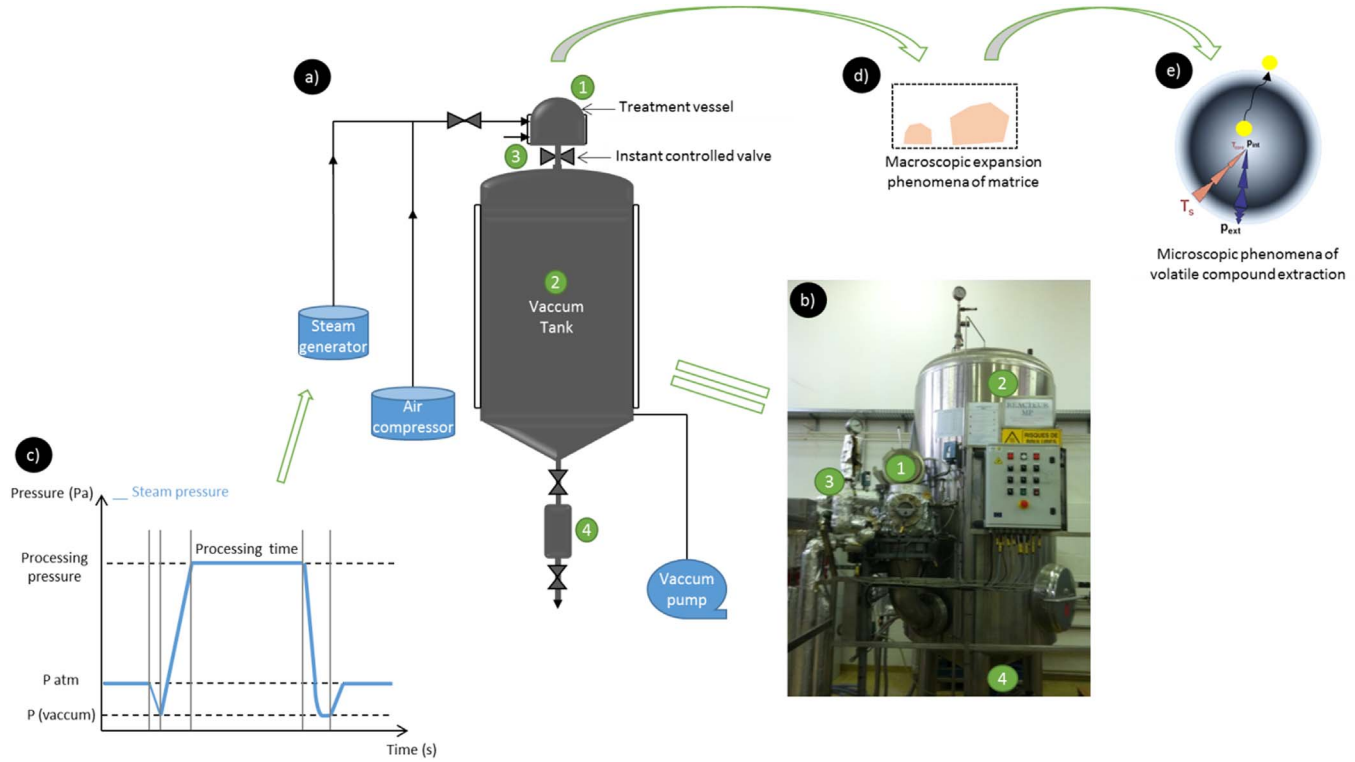


Fig. 2. Schematic representation (a) and photography (b) of DIC process, from experimental conditions (c) to an example of a major macroscopic (d) and microscopic (e) phenomena generated by DIC treatment.

physicians, and food technologists even from academia or industry will find the major solutions identified to design and demonstrate Green Food Processing on laboratory, classroom and industrial scale to approach an optimal consumption of raw food materials, water and energy: (1) improving and optimization of existing processes; (2) using non-dedicated equipment; and (3) innovation in processes and procedures.

2. Instant controlled pressure drop technology

2.1. Process and procedure

DIC 'Détente Instantanée Contrôlée', French for Instant Controlled Pressure-Drop is based on the main principle of the thermodynamics of instantaneity and auto-vaporization processing combining with hydro-

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thermo-mechanical evolution of many biopolymers for food, cosmetic, and pharmaceutical purposes. Developed by Allaf and Vidal (1989), DIC's research began by fundamental studies regarding expansion through alveolation and has targeted several applications in response to issues of control and quality improvement. DIC is considered as a high temperature/high pressure - short time (HTST) treatment and consists of thermo-mechanical processing induced by subjecting the raw material to saturated steam for a short period, and followed by an abrupt pressure drop towards vacuum (about 5 kPa with a rate > 0.5 MPa.s^{-1}). Typically, the sample was adjusted to about 30% dry basis and submitted to a first pressure drop in the treatment vessel to be pre-conditioned. Then, the sample is subjected to heating under high saturated pressure (up to 1 MPa) at high temperature (up to 180 °C) during a short time (5 to 60 s) and followed by an abrupt pressure drop to vacuum (3–5 kPa, $\Delta t = 20$–200 ms). The abrupt pressure drop ($\Delta P/\Delta t > 25.10^6$ Pa.s^{-1}) induces a significant mechanical stress related to instant auto-vaporization of water, an instantaneous cooling of the sample, and swelling phenomenon, causing the rupture of cells and secretion of metabolites through cell walls (Allaf et al., 2011). The purpose of these effects leads to texture change, which results in higher porosity, as well as increased specific surface area and reduced diffusion resistance of the sample. Experimental conditions of DIC extraction allow reduced processing time and the instant reducing temperature drops prevent further thermal deterioration and ensure a high quality of extract (Haddad, Louka, Gadouleau, Juhel, & Allaf, 2001)

DIC equipment is composed of four major components (Fig. 2): (1) an extraction vessel, which is an autoclave with a heating jacket where the sample to be treated is placed; (2) a controlled pressure-drop valve, which ensures a quick and controlled liberation of steam pressure contained in the extraction vessel to the vacuum pump; (3) a vacuum system composed of a vacuum pump and a tank with a volume 50-fold higher than the volume of the treatment vessel; (4) an extract collection trap used to recover condensates; a water ring pump maintains the tank pressure at about 5 kPa. At the beginning, humidified product is placed in the autoclave at atmospheric pressure before vacuum setting. Initial vacuum ensures closer contact of the fluid heating with the exchange surface, which enhances the heat transfer in raw material. After closing the valve (between the autoclave and the vacuum tank), the autoclave is filled with steam up to a processing pressure. After this treatment time, the controlled pressure-drop valve is instantaneously opened (in < 200 ms), resulting in an abrupt pressure drop inside the treatment vessel. After steam release, the atmospheric pressure is returned back inside the reactor.

2.2. Applications in food processing

DIC treatment is employed in several industrial fields such as food, cosmetic, pharmaceutic in response to issues of control and quality improvement, coupled with reduced energy costs (Allaf & Allaf, 2014). As shown in Table 1, DIC can be used in various operations such as transformation, preservation, and extraction. For each operation the approach has always induced the integration of phenomena of instantaneity to intensify the elementary processes of transfer.

The DIC treatment combined to classical hot air drying may be considered as the tool of intensifying the drying when the kinetics of dehydration is particularly low due to difficulty of water transfer through material because of resistance of the natural structure of the material. Recently, Mounir and Allaf (2008) propose an innovative process of 3-stage spray-drying using DIC treatment of powders (sodium caseinates, whey proteins), using saturated steam as a texturing fluid which can permit the modification of powder granule structure, and allows the formation of vacuoles and pores. DIC increases the specific surface area of spray-dried powder and consequently overcomes the problems related to the presence of fine powder (dustiness). DIC treatment appears to be a good alternative to expand granule powder of heat-sensitive food such as apple and onion (Mounir, Besombes, Al-Bitar, & Allaf, 2011). After an initial partial drying step, DIC treatment permits the improvement of dehydration kinetic inserting a texturing process allowing the partially dried product to be expanded. The second step of drying (after DIC treatment) is greatly reduced from 6 h (untreated apple) to 1 h in the case of treated-sample. In the case of onion, the effective diffusivity is accelerated after DIC treatment ($7.56{\cdot}10^{-10}$ as against $0.46{\cdot}10^{-10}$ m^2·s^{-1} for untreated sample. At equal water content (100%db.), DIC pretreatment with a pressure comprised between 0.1 and 0.3 MPa and a short heating period of few seconds (5 to 45 s) combined to freezing and thawing allows improvement of drying/rehydration operations for apple with a good preservation of textural properties. DIC is considered as a good alternative process to classical hot air drying and freeze-drying, especially for drying fragile fruit, such as strawberries (Alonzo-Macías, Cardador-Martínez, Mounir, Montejano-Gaitán, & Allaf, 2013). Furthermore, DIC coupled to hot air drying allows to preserve the nutritional value and bioactive molecules, at optimal DIC conditions (0.35 MPa for 10 s), treated strawberries were richer in anthocyanins and phenolic compounds as compare to other classical drying methods.

DIC is recognized as a process for decontamination, debacterization of foodstuffs. Three patents protect this application (Allaf, Debs-Louka, Louka, Cochet, & Abraham, 1994; Allaf et al., 1998). The treatment allows DIC the elimination of micro-organisms (even in spore forms) through two main mechanisms: a controlled thermal treatment; pressure relaxation excessively stressed on microorganisms that cause their explosion (Debs-Louka, Louka, Abraham, & Allaf, 2010). Archeological investigations often renew pieces of wood having spent long periods in water (mostly seawater). The DIC treatment can stop these degradations and stabilize the archeologiocal waterlogged woods originated from different museums (Allaf, Rezzoug, Cioffi, Louka, & Sanya, 1999; Sanya, Rezzoug, & Allaf, 1998). In the case of thermal treatment for allergen in peanuts, lentil, chickpeas and soybeans proteins, DIC treatment produces a reduction in the overall in vitro IgE binding. Immunoreactivity of soybean proteins was almost abolished with a treatment at 6 bars for 3 min. Additionally, DIC treatment (0.4 MPa during 25 s) showed decrease in the IgE binding of whey proteins (β-lactoglobulin and α-lactalbumin) and a greater reduction of the allergenicity of whey proteins (Boughellout et al., 2015).

DIC pretreatment is considered an efficient method for extraction and texturing from various vegetal materials. Indeed, Mkaouar, Bahloul, Gelicus, Allaf, and Kechaou (2015) showed that DIC texturing on the solvent extraction of polyphenols from olive leaves improves the yield of extraction of 312% and permits generating extract richer in bioactive compounds. Another study has proved that DIC allows enhancing lipid extraction from jatropha and rapeseed seeds without significant modification of fatty acids composition in comparison with conventional Soxhlet extraction (Nguyen Van, 2010). Allaf et al. (2014) have shown that enhancing of lipid extraction by DIC treatment is clearly noticed by calculation of effective diffusivity. DIC process is a good solution for deodorizing and expanse the vegetal matrix in the same time before improving antioxidant extraction from rosemary leaves (Allaf et al., 2013). Expansion of raw material permits a better diffusion of solvent through the material and accelerates the extraction of bioactive compound from 4 h for hydrodistillation to 3 min with DIC. More recently, DIC was endorsed as a pretreatment for in-situ transesterification in the case of microalgae. Optimized DIC treatment ($P = 0.16$ MPa and $t = 68$ s) allows increasing of 27% in total lipid and $> 75\%$ in fatty acids methyl esters yield (Kamal, Besombes, & Allaf, 2014). Additionally, to lipid extraction, it was observed that the residual microalgae allow increasing of lutein extraction. Moreover, DIC allows reducing the energy consumption and manufacturing cost compared to conventional processes of lipid extraction.

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Table 1
Example of applications and experimental conditions of DIC.

Applications	Matrix	Experimental conditions	Benefits	Reference
Transformation				
Spray-drying (spraying coupling to DIC and final drying)	Milk, sodium caseinates Whey protein powders	1) Spraying: T = 60–180 °C, final powders humidity: 4 and 22%db. 2) DIC: P = 0.3–0.7 MPa, t = 9–25 s 3) Drying: 50 °C, air stream 1.2 m/s at 265 Pa initial humidity	Formation of vacuoles, which increased the specific surface area Better functional quality Reduce the specific problems of powder flowability Improving the kinetics of final drying	(Mounir & Allaf, 2008)
Puffing (Hot air drying and DIC treatment coupling to snaking)	Onion chips Apple	1) Drying: T = 40 °C, air flow: 1 m/s, humidity: 267 Pa 2) DIC treatment: P = 0.2–0.6 MPa, t = 5–55 s (apple) P = 0.2–0.5 MPa, t = 5–15 s (onion) 3) Hot air drying (snaking): T = 40 °C, air flow: 1 m/s, humidity: 267 Pa, final moisture content: 5%db.	Greater effective diffusivity and initial starting accessibility Expand the compact structure Vitamins preservation Low-cost, high-quality snaking and powder	(Mounir et al., 2011)
Texturation (pre-drying coupling DIC and freezing)	Apple	1) Pre-drying at 45 °C, 2 m/s, final water content: 30–200% db. 2) DIC: P = 0.1–0.3 MPa, t = 5–45 s, 3) Freezing: − 30 °C for 600 min, after thawed at 4 °C	Improvement of drying/rehydration operations Intensifying the internal resistance to water transfer Good preservation of textural properties	(Ben Haj Said, Bellagha, & Allaf, 2015)
Swell-drying (coupling DIC to standard hot air drying)	Strawberry Green Moroccan pepper	1) Partially hot air dried at 50 °C until 18% db., P_{vapor} = 265 Pa, air flow: 1.2 m/s, t = 8 h 2) DIC conditions: P = 0.1–0.6 MPa; t = 10–30 s 3) Same conditions of hot air drying before	Efficient technique for fragile fruit Preserve nutritional value Improve quality Decrease consumed energy and coast	(Alonzo-Macías et al., 2013)
Preservation				
UHT decontamination (DIC coupling to hot air drying)	*Bacillus stearothermophilus*	1) Initial DIC stage: P = 0.7 MPa, 5 mm thick dry, t = 3 s 2) Heating stage: T = 100–150 °C t = 5–60 s	Destruction of microorganism cell walls and more specifically on the spore wall	(Debs-Louka et al., 2010)
Conservation (successive pressure drops dehydration)	Archeological waterlogged wood	1) Starch impregnation 2) DIC thermal treatment: T = 18–21 °C End of pressure drop T = − 1–3 °C	Small shrinkage Very good surface aspect Initial color maintain Lower moisture content More rapid than freeze-drying	(Sanya et al., 1998)
Thermal treatment for allergen (DIC + solvent extraction)	Peanuts, lentil, chickpeas, soybean proteins	1) DIC treatment: P = 0.3–0.6 MPa, t = 1–3 min, constant initial water content of 50%db. 2) Protein extraction with n-hexane	Drastic reduction in immunoreactivity Reduction in the overall in vitro IgE Time and energy reduction Good alternative to intact proteins in the development of different food products	(Cuadrado et al., 2011)
Protein's immunoreactivity	Milk	P = 0.4 MPa T = 144 °C t = 25 s Pressure drop towards 5 kPa, 32 °C	Enhance the antigenicity of treated caseins Decrease in the IgE	(Boughellout et al., 2015)
Extraction				
Diffusion (DIC + solvent extraction)	Olive (*Olea europaea* L.) leaves	1) DIC pretreatment conditions: P = 0.1 MPa, number of cycles C = 1, t = 11 s 2) Solvent extraction	Destruction of cell walls after DIC texturing Intensification of solvent extraction	(Mkaouar et al., 2015)
Steam extraction (DIC)	Oak wood *(Quercus alba)* chips	P = 0.1–0.6 MPa, t = 30–300 s t = 5 min Initial moisture content: 20%, thickness of chips: 0.5 mm	Degradation of wood cells and subsequent rapid liberation of volatiles Swelling and creation of alveoles within wood microstructure Economy in terms of time and energy	(Mellouk, Meullemiestre, Maache-Rezzoug, Allaf, & Rezzoug, 2013)
Deodorization (DIC + solvent extraction)	Rosemary leaves	1) DIC conditions: P = 0.6 MPa, time per cycle: 6–40 s, number of cycle: 1–11 2) Solvent extraction	Expansion of raw material inducing a better extraction of antioxidant Structural alterations eased secondary metabolite extraction	(Allaf et al., 2013)

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Table 1 *(continued)*

Applications	Matrix	Experimental conditions	Benefits	Reference
Transesterification in situ (DIC + Folch extraction)	Microalgae	1) DIC conditions: P = 0.2–0.6 MPa, t = 20–60 s, water content: 30 and 100%db. 2) Modified conventional Folch extraction	Higher specific surface area Positively affects porosity, diffusivity and lipid availability More safer extraction Quick and effective process	(Kamal et al., 2014)

2.3. Success story

The technology of instant controlled pressure drop has been generated by Allaf group since 1988, several industrial projects have been developed. Many patents have been filed since 1993 (Allaf et al., 1994) and more than twenty PhD theses have treated the subject from different angles. Today, the Allaf group provides research while participating in the design of machinery and the transition from laboratory studies to the industrial stage. The process is operated by ABCAR-DIC Process company, localized in La Rochelle (France). Swell-drying is largely used at industrial scale to produce swell-dried products of > 200 varieties such as apple, banana, strawberry, onion, tomato etc. in the form of cubes, slices and powder that is found in healthy food and unique bands known like “greedy snacking”, “fruit snacks” or “vegetable petals.” This process was also used for decontamination, dehydration and texturation of many foodstuffs. Furthermore, intermediate food products obtained after DIC treatment are used for the development of dehydrated meals or dairy products. The DIC technology is also largely used for the post-harvest rice processing. The USDA report says that Egypt's rice paddy production in the year May 2012 to April 2013 is expected to rise to 6.37 million tons of dried paddy rice, i.e., 4.5 million tons of dried unbroken white grain DIC rice. In China, many teabags treated by DIC have been commercialized and allow a greater diffusion of tea in water and even in cold water (Allaf & Allaf, 2014).

3. Pulsed Electric Field

Pulsed Electric Field (PEF) treatment, also referred as electroporation or electropermeabilization, is a nonthermal process where an external electric field is applied to a living cell for a very short duration (from several nanoseconds to several milliseconds) (Fig. 3). The exact mechanism of membrane permeabilization is not precisely understood yet, but it is accepted that electroporation consists of four different stages including (Saulis, 2010): (a) increase of the transmembrane potential of the cytoplasmic membrane due to cell membrane charging by the applied external electric field, (b) creation of small metastable hydrophilic pores if a threshold of transmembrane potential is reached (0.2–1.0 V), (c) evolution of the number and/or size of the created pores during the PEF treatment, and (d) PEF post treatment stage with leakage of intracellular compounds, entrance of extracellular substances i.e. as irreversible electroporation or pore resealing and integrity recovering of membrane i.e. reversible electroporation.

The effectiveness of cell membranes electropermeabilization depends on several process parameters (electric field strength, treatment time, specific energy, pulse shape, pulse width, frequency and temperature), treatment mode (batch, continuous), configuration of treatment chamber (collinear, coaxial and parallel) (Van den Bosch, 2007), physicochemical characteristics of the treated matrix (pH and conductivity), characteristics of the treated cells (size, shape, membrane, and envelope structure) and state (suspension, solid, semi-solid) (Vorobiev & Lebovka, 2009).

PEF is a promising green tool in food processing as its opens a wide range of application due to the described phenomenon of cell membrane increased permeability or disruption via electroporation. The application can be classified depending on the extent of the applied external electric field and specific energy (Toepfl, Heinz, & Knorr, 2006). For instance, the application of low electric treatment ($E < 2$ kV/cm; $Q < 5$ kJ/kg) is known to induce stress response on the cellular level and is routinely used in molecular biology to gain access to the cytoplasm in order to introduce different molecules. The applications in this field are rather scarce and are limited to biological reaction enhancement of vegetable and microbial cells. The improvement of mass transfer is generally dependent on higher electric fields ($0.1 < E < 50$ kV/cm; $0.4 < Q < 60$ kJ/kg). Food preservation due to microbial or enzyme inactivation requires the highest level of

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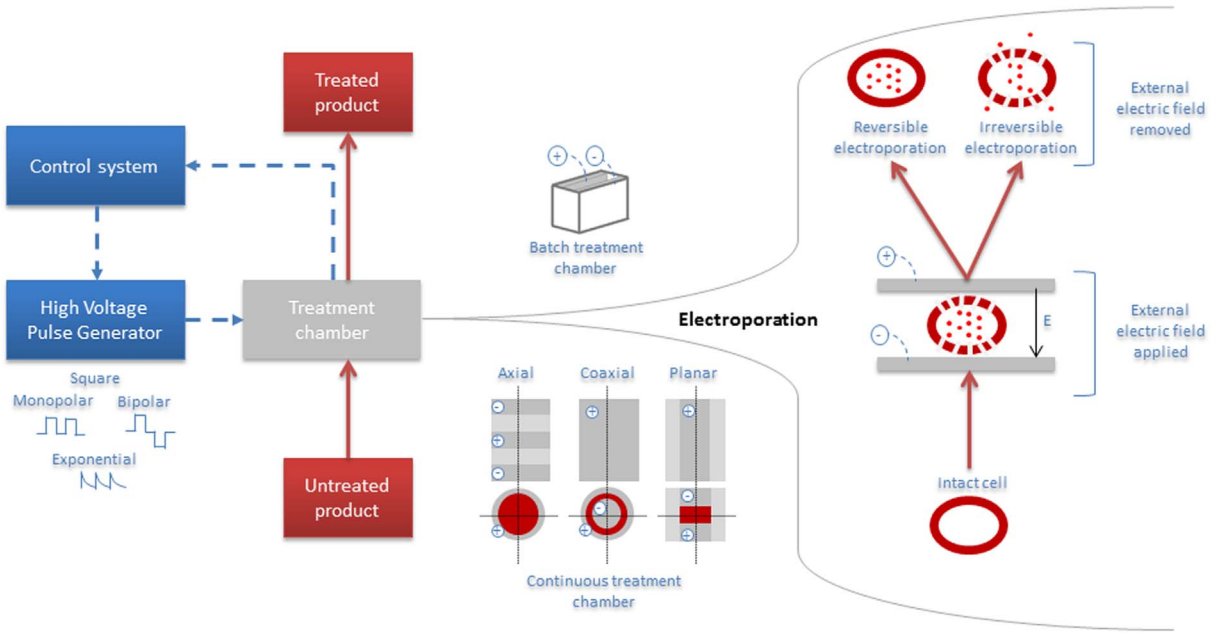


Fig. 3. Schematic view of a PEF treatment system with a representation of different types of treatment chambers and a brief description of electroporation phenomenon during the electric treatment.

Table 2
Example of applications and experimental conditions of PEF.

Application	Matrix	Treatment conditions	Benefits	Reference
Preservation				
Inactivation	Ringer solution contaminated with *B. subtilis* spores	(PEF + heat) $6 < E < 11$ kV/cm; $Q < 350$ kJ/kg	Inactivation of spores with reduced heat load	(Siemer, Toepfl, & Heinz, 2014)
	Carrot puree	(Chilling + PEF) $0.1 < E < 1.1$ kV/cm; $0.15 < Q < 15.58$ kJ/kg	Improvement of the stability of vitamin C and reduction of the residual activity of AAO and POD	(Leong, Oey, Clapperton, Aganovic, & Toepfl, 2015)
Freezing/thawing	Apple; spinach	(PEF + impregnation + freezing + thawing) $0.58 < E < 0.8$ kV/cm; Q: n.d.	Acceleration of freezing/thawing process and comparable texture after defrosting to fresh samples	(Parniakov et al., 2016a; Phoon et al., 2008)
Osmotic dehydration	Apple, carrot	(PEF + impregnation) $0.22 < E < 10$ kV/cm; $0.15 < Q < 106.7$ kJ/kg	Increase of water loss Solute uptake by the matrix depends on the matrix and operational conditions	(Rastogi et al., 1999; Wiktor et al., 2014)
Convective drying	Carrot, red pepper	(PEF + hot air drying) $0.5 < E < 2.5$ kV/cm; $1.8 < Q < 56.5$ kJ/kg[a]	Increase of drying rates and color quality (red pepper)	(Gachovska et al., 2008; Won et al., 2015)
Extraction				
Diffusion	Grape pomace, sugar beet	(PEF + extraction by diffusion) $0.6 < E < 3$ kV/cm; $Q < 19.4$ kJ/kg	Increase of polyphenols and sucrose concentration; selective extraction towards anthocyanins, lower coloration and better filtrability of juices (sugar beet)	(Brianceau et al., 2015; Loginova et al., 2011)
Expression	Apple, grape	(PEF + extraction by pressing) $0.4 < E < 0.65$ kV/cm; $15 < Q < 32$ kJ/kg	Increase of juice and polyphenol yield, decrease of juice turbidity and better odor intensity	(Grimi, Lebovka, Vorobiev, & Vaxelaire, 2009; Turk et al., 2012)
Filtration	BSA suspension	(PEF + cross flow UF) $E = 4.5$ kV/cm, Q: n.d.	Improvement of concentrating rate of protein in retentate and reducing the solute-related resistance to the permeate flux	(Robinson et al., 1993)
Distillation	Roses (*R. alba* L.)	$E = 25$ kV/cm, $10 < Q < 20$ kJ/kg	Increase of oil essential oil yield and possible reduce of distillation time	(Dobreva et al., 2010)
Transformation				
Cutting	Carrot	$E = 0.8$ kV/cm, $Q < 166$ kJ/kg	Decrease of the cutting force	(Leong et al., 2014)
Softening	Meat	$0.32 < E < 0.48$ kV/cm; Q/n.d.	Improving meat tenderness	(Bekhit et al., 2016)
Frying	Potato	$0.75 < E < 2.5$ kV/cm; Q: 18.9 kJ/kg	Improving potato color and reducing oil uptake after frying	(Ignat et al., 2015)
Fermentation	*S. cerevisiae*	$100 < E < 6$ kV/cm; Q: n.d.	Increase of sugars consumption, decrease of fermentation time	(Mattar et al., 2015)

[a] Data not available in (Won et al., 2015).

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treatment (2 < E < 90 kV/cm; 50 < Q < 44,000 kJ/kg).

3.1. Food preservation

Food preservation (Table 2) is achieved whether by controlling or by inhibiting external contaminants and/or internal biological reactions that could alter the organoleptic and nutritional quality of food. For this, two main strategies are being used: a) applying thermal treatments; b) decreasing the water activity of the food matrix to inhibit biological reactions.

Non-thermal PEF processing in liquid foods and beverages preservation has been thoroughly studied as an alternative method to heat preservation. A wide variety of vegetative microorganisms and enzymes have been successfully treated in different food matrices (Griffiths & Walkling-Ribeiro, 2014; Martín-Belloso, Marsellés-Fontanet, & Elez-Martínez, 2014; Terefe, Buckow, & Versteeg, 2015). Better quality retention in PEF-processed products compared to thermal processing has been observed in many cases. However, PEF has often little or a limited effect on enzymes at processing conditions sufficient for microbial inactivation (50–1000 kJ/kg). At sufficiently high-specific energy input (e.g. > 1000 kJ/kg), PEF causes significant inactivation of enzymes at ambient and mild temperature conditions (Terefe et al., 2015). Commercial PEF treatment systems operate in continuous mode for high productivity. Generally, PEF treated liquid food is packaged after their preservative treatment. A batch treatment mode, in conductive plastic material, could also be achieved with comparable levels of inactivation (Roodenburg et al., 2013).

Depending on the required inactivation, target, product composition and initial temperature, it may be advantageous to combine PEF treatment with other treatments (heat, pH, antimicrobial). Such combinations may provide the required lethality at lower field strength and with less electrical energy (Álvarez & Heinz, 2007).

Because of their rigid structures, bacterial spores can survive harsh environments for a long period of time. The combination of temperature and electric fields > 60 °C and 30 kV/cm respectively was effective on spore inactivation (Siemer, Aganovic, Toepfl, & Heinz, 2015).

The main strength seems to be in PEF ability to affect less the nutritional and sensory properties of food material as compared to thermal treatment. For instance, PEF treated beverages seem to have higher contents of polyphenols, carotenoids and vitamins compared to heat pasteurization (Tokusoglu, Odriozola-Serrano, & Martín-Belloso, 2014).

Freezing is a widespread method for food preservation. Unluckily, such treatment leads to deterioration of food texture and flavors during subsequent transformation operations. The formation, the size of crystals and recrystallization after freezing are the main reasons of the quality loss of frozen foods. Reversible electroporation, due to its transient increase in membrane permeabilization, enables introduction of cryoprotectants into biological cells. This family of molecules prevents crystal formation during freezing. This combination leads to a noticeable acceleration of the freezing/thawing process (Jalté, Lanoisellé, Lebovka, & Vorobiev, 2009; Parniakov, Bals, Lebovka, & Vorobiev, 2016a), increase of freezing temperature and a decrease of the ice propagation rate (Dymek, Dejmek, Galindo, & Wisniewski, 2015). Texture and firmness of spinach leaves (Phoon, Galindo, Vicente, & Dejmek, 2008), potato strips were retained by impregnating the material with trehalose (Shayanfar, Chauhan, Toepfl, & Heinz, 2013) and apples with glycerol (Parniakov et al., 2016a).

Dehydration is probably the oldest means for food preservation. The intact cell membranes in food materials represent a highly limiting factor (barrier) to water transport during drying of food matrices. Pore formation during PEF treatment increases cell membrane permeability which enhances the mass transport phenomena. PEF treatment was successfully combined with traditional processes such as osmotic dehydration, freeze drying, radiant and convective heat. The results are encouraging as the combination of PEF and osmotic dehydration resulted in an increase of water loss and migration of solutes into the food matrix was observed (Rastogi, Eshtiaghi, & Knorr, 1999; Wiktor, Śledź, Nowacka, Chudoba, & Witrowa-Rajchert, 2014). A significant reduction of energy consumption and an acceleration of cooling and drying time could also be achieved when apples and potatoes are electrically treated prior to freeze drying without alteration of the dried samples shape (Parniakov, Bals, Lebovka, & Vorobiev, 2016b; Wu & Zhang, 2014). Similar observations were reported for radiant (Baier, Bußler, & Knorr, 2015) and convective air drying (Gachovska, Adedeji, Ngadi, & Raghavan, 2008; Won, Min, & Lee, 2015). PEF treatment was beneficial to color quality of air-dried products (Won et al., 2015).

3.2. Extraction

Extraction by solvents (diffusion) and force fields (pressing, filtration, and centrifugation) is widely used for production of liquid foods and beverages as well as for extraction of molecules of industrial interest. Pretreatments that modify the permeability of the cell membranes, such as grinding, heating, or enzymatic treatment, enhance the mass transfer. However, these techniques may require a significant amount of energy and can cause losses of valuable food compounds.

The use of PEF became very popular in this field as it allows critical acceleration of the solid–liquid extraction. Different technologies of agro-industrial extraction become more selective and less energy consuming if PEF is applied (Vorobiev & Lebovka, 2015).

The combination of PEF and extraction by diffusion has been investigated for improving the extraction of different compounds located on the inside of plant cells, such as colorants (chlorophylls, carotenoids, betalains…), sucrose, polyphenols and other secondary metabolites (Puértolas, Luengo, Álvarez, & Raso, 2012). PEF pretreatment can be applied for winemaking prior to the macerating fermentation step, the extraction of polyphenols is improved and the wine resulting has different organoleptic (color) attributes (El Darra et al., 2016). The same pretreatment is applied to traditional wine making residues; an enhancement of the selectivity of colorant (anthocyanin) extraction is also highlighted (Brianceau, Turk, Vitrac, & Vorobiev, 2015). PEF application has a large potential for replacement or modification of the conventional thermal technology for sugar extraction from sugar beets. PEF pretreatment assisted “cold” extraction results in higher concentration of sucrose, lower concentration of colloidal impurities (especially, pectins), lower coloration and better filterability of juice (Loginova, Loginov, Vorobiev, & Lebovka, 2011).

Traditionally, the increase of the yield in the juice and oil extraction industry has been one of the most important priorities. Gentle techniques that do not cause losses in nutritionally and organoleptic attributes should be used in the procedures to improve the extraction yield. Different plant based matrix was successfully studied for pressure expression combined with PEF pretreatments. Fruit juices (apple, grape…) and vegetable oils (olive) yield is significantly increased when moderate PEF treatments are applied before mechanical expression (Vorobiev & Lebovka, 2015). The electric treatment does not induce bad flavors or taste in the oil (Abenoza et al., 2013) and can produce less turbid, significantly odorant and high polyphenols content apple juices (Turk, Vorobiev, & Baron, 2012).

Pulsed Electric Field can also be combined with other mechanical separation operation such as filtration. The electric treatment helps reducing the solute-related resistance to the permeate flux for concentrating proteins. Significant improvements in the rate of concentrating the protein in the retentate can be obtained, resulting in reduced membrane surface area requirements for a specific degree of separation (Robinson et al., 1993).

The combination of PEF with extraction methods such as distillation is also a successive method that leads to an increase of essential oil yield

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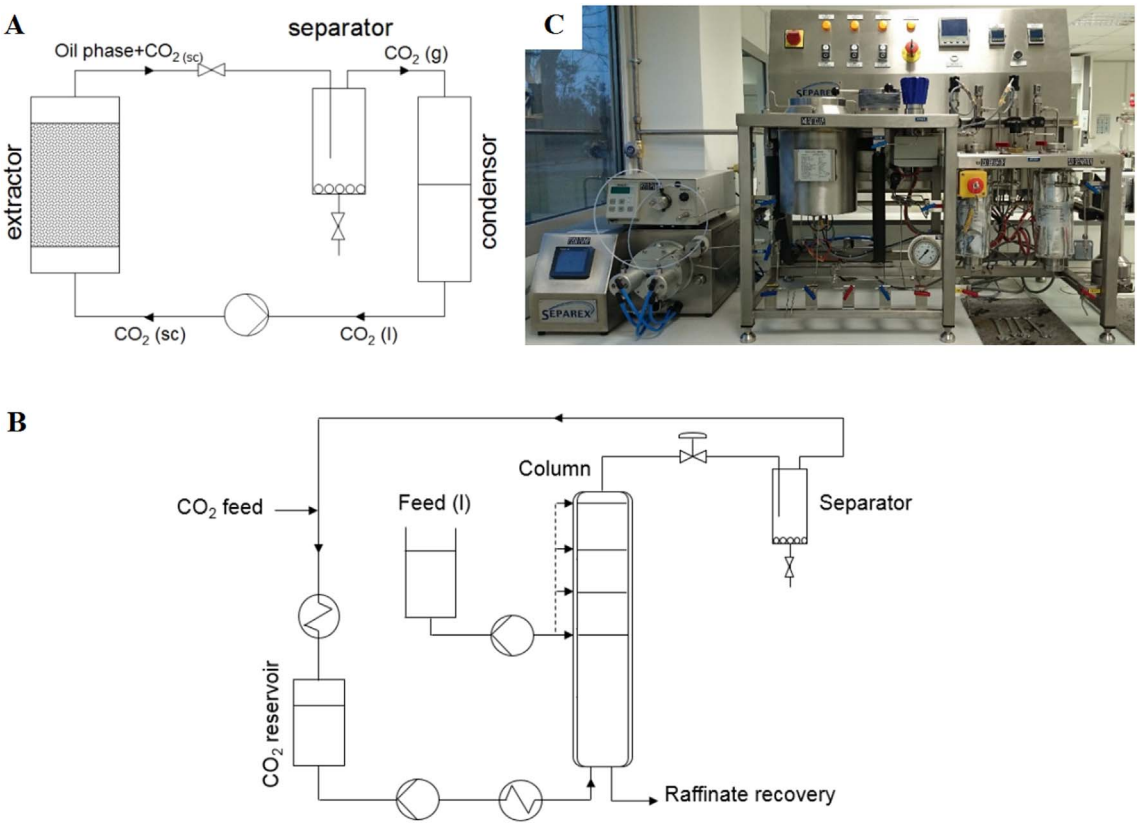


Fig. 4. Simplified schematic representation of supercritical CO_2 installation for solid extraction (A) and liquid extraction (B), example of supercritical fluid lab scale equipment – 1 L autoclave (C).

and reduces the distillation time (Dobreva, Tintchev, Heinz, Schulz, & Toepfl, 2010).

3.3. Transformation

In addition to preservation and extraction applications, other methods were proposed to enhance transformation processes in the latest years. PEF is successfully applied to enhance the mechanical removal of undesired food parts. Skin removal of some fruits (tomato, mango…) gave results equal to steam peeling but with low applied energy (Toepfl, 2012). The data which are available on this topic are rather scarce.

Several studies have demonstrated interesting effects of PEF on softening vegetables and animal tissues. The viscoelastic and textures properties were changed after the electric treatment probably due to loss of turgor pressure (Lebovka, Praporscic, & Vorobiev, 2004). These modifications have direct impact on decreasing the cutting force of fruits (Leong, Richter, Knorr, & Oey, 2014) and improving meat tenderness (Bekhit, Suwandy, Carne, Van de Ven, & Hopkins, 2016).

Several applications in preparation, curing and cooking of meat (McDonnell, Allen, Chardonnereau, Arimi, & Lyng, 2014) and vegetable products have also been proposed (Toepfl, Siemer, & Heinz, 2014). For instance, the application of moderate PEF treatment to potatoes improves its color and reduces oil uptake after frying (Ignat, Manzocco, Brunton, Nicoli, & Lyng, 2015).

Low intensity PEF treatment, inducing reversible electroporation, was recently presented as stressing method to promote production of metabolites in vegetables or to accelerate biological reactions (Toepfl et al., 2006). Mattar et al. (2015) showed that the electric treatment prior to fermentation increases fructose consumption up to 3.98 times at the end of the lag phase and 20 h decrease of the overall fermentation time can be achieved compared to control (without electric treatment).

3.4. Success stories

The numbers of applications related to pulsed electric fields are constantly increasing. New ideas are being tested in laboratory and at industrial scale as reliable pulse modulators and tum-key systems. Recently, new PEF equipment manufacturers, such as Elea, Steribeam, Scandinova, and PurePulse, located in Germany, Sweden, and The Netherlands, respectively, have emerged, thus indicating the growing interest of the food industry in the application of the technology. A cooking device Nutri-Pulse® e-Cooker®, commercialized by IXL Netherlands B.V. is advertised as capable of preparing food with help of electroporation and pulse ohmic heating, results in better conservation of the original nutritive value and the original flavor, color, structure and taste.

4. Supercritical fluids

4.1. Principle, process and procedure

4.1.1. Principle

Supercritical fluids (SCF) represent an alternative to organic solvents in processes using solvents (Badens, 2012). A fluid is considered to be in its critical state when it is both heated above its critical temperature (Tc) and pressurized above its critical pressure (Pc) (Brunner, 2005). The specificity of SCF relies in their physical properties, which can be modulated by an increase of pressure and/or temperature, beyond their critical values. SCF have a density close to liquids, which induces a solvating power close to liquids. Their viscosity, close to gases and a diffusivity that is intermediary between liquids and gases, leads to an increase of mass transfer between the solute to extract and the SCF. These properties enable adjustment of solvent selectivity of a SCF towards a target compound, which is particularly interesting in the case of extraction.

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Supercritical CO_2 (SC-CO_2) is the fluid mostly used in SCF processes (Rozzi & Singh, 2002). Process implementation is eased due to its low critical coordinates (Tc: 31 °C, Pc: 7.38 MPa). Moreover, some of its advantages include non-inflammability, cheapness, abundance and its volatility at atmospheric pressure implies that after depressurization extracts are solvent-free. SC-CO_2 is a non-polar solvent; its solvent power is comprised between the one of pentane and toluene (Lumia, 2011). To enhance solubilization of polar substances, a polar modifier (ethanol, methanol for example) can be added to SC-CO_2.

4.1.2. Process implementation

Concerning extraction and fractionation, two types of equipment settings can be found. Supercritical fluid extraction (SFE) from solid materials is achieved with autoclaves (industrial units may be composed of several autoclaves for semi-continuous processing). Liquid fractionation with SCF is performed with countercurrent columns (for continuous processing).

SCF extraction installations for solid processing are composed of four main parts: (i) a volumetric pump, to ensure a correct pumping of the fluid, the pump can be preceded by a cooler which brings gaseous component in a liquid state (ii) a heat exchanger, (iii) an extractor, where pressure is established and maintained by a back pressure regulating valve, (iv) a separator (Fig. 4A and C). Up to three separators can be put in series, to achieve multiple fractioning of the molecules contained in the extracts.

Extraction by SCF from solids is divided in two main steps: the extraction and the separation of the solute from the solvent. To perform SFE, the fluid has to be brought in its supercritical state. To achieve this, the fluid is usually sequentially pressurized and heated before entering the extractor. Brought at the desired pressure and temperature, the SCF percolate in the extractor, with an ascending or descending flux. The SCF extract the solute contained in the matrix. Separation of the solute from the SCF will be achieved in the separator, where the SCF will turn into a gaseous state, and the solute no longer solubilized in the SCF will be separated by gravity. Extracts are therefore collected at the bottom of the separator. Depending on the equipment, the gas can be recycled by being re-injected in the system, or released to atmosphere.

Fractioning from a liquid feed can be performed in batch mode, where extraction is performed by desorption of a liquid placed on an absorbent using the same equipment as for solid extraction (Benaissi, 2013). In a continuous mode, a countercurrent column is used to selectively recover a solute from the feed (Fig. 4B). Regarding process implementation, the feed is introduced in the middle or on the top of the column, the supercritical phase being introduced at the bottom of the column. The extract to be recovered and the fluid leave at the top of the column and the raffinate (heavier phase) is recovered at the bottom of the column. SCF preparation and regeneration are similar to SFE from solid material.

Apart from solid/fluid or fluid/fluid extraction processes, SCF are used for particle formation process and are the subject of extensive reviews (Jung & Perrut, 2001; Reverchon, 1999; Reverchon & Adami, 2006; Rodríguez-Meizoso & Plaza, 2015). The general concept of two processes is briefly described below:

RESS (Rapid Expansion of a Supercritical Solution). The solute of interest is diluted in a supercritical phase and the resulting mixture is rapidly depressurized through a nozzle. This process has the advantage to produce very fine solid particles, but its applications are limited by the polarity of the solutes to precipitate (low polarity solutes).

GAS or SAS (Gas or Supercritical fluid Anti Solvent) which concept is to "decrease the solvent power of a polar liquid solvent in which the substrate is dissolved, by saturating it with carbon dioxide in supercritical conditions" (Jung & Perrut, 2001). This causes the substrate to precipitate or to recrystallize.

Compared to conventional processes, several key advantages result from the use of SCF. Absence or limited solvent consumption (in the case of co-solvent use) leads to production of a solvent-free extract. The depressurization step in supercritical fluid processes (SFP) enables limiting the number of unit operations, since no separation or purification step is necessary. By operating at low temperatures during the whole process, SFP are adapted to production of heat-sensitive biomolecules. Additionally, SCP are intrinsically sterile (Badens, 2012).

4.2. Applications in Green Food Processing

Since the 1970s, a great number of applications using SCF have emerged and have been developed at laboratory and pilot scale. In this section, some applications from published studies related to food processing are reviewed, with a special emphasis on those dedicated to transformation, preservation and extraction by SCF. Far from being exhaustive, the examples given, when possible, will be supported by key related reference reviews or papers.

4.2.1. Transformation

Transformation processes can be achieved with the use of SCF, and most of them are related to particle formation processes. Precipitation or crystallization of food compounds can be achieved by SAS-type processes (e.g. carotenoids). To enhance biomolecules properties preservation, encapsulation with SCF has been investigated (Cocero, Martín, Mattea, & Varona, 2009; Rodríguez-Meizoso & Plaza, 2015). RESS and SAS processes have been successfully applied for anthocyanins and antioxidant encapsulation (Table 3). Process performances are generally evaluated according to particle characteristics (size and morphology), encapsulation efficiency and release of encapsulated compounds in a matrix.

Textural modifications of food matrix have been produced by combining extrusion and SCF (Maskan & Altan, 2011; Rizvi, Mulvaney, & Sokhey, 1995). By injecting SC-CO_2 during the extrusion process, the processing conditions are milder than conventional extrusion (lower shear and starch expansion is possible below 100 °C). Therefore, torque, equipment wear and heat-sensitive compound degradation can be minimized. Expansion of extrudates can be controlled according to the amount of SC-CO_2 injected, improving the structural characteristics of extrudates (Table 3).

Fractioning by SCF is used for aroma fractioning, where waxes (compounds of high molecular weight) can be separated from the volatile fraction or for lipid fractionation from oils (Reverchon, 1997). Fractioning can be performed after extraction in the depressurization stages through the separators or with a countercurrent column. Fractional extraction can also enable a selective recovery of compounds (Table 3, Palma, Taylor, Varela, Cutler, & Cutler, 1999).

The use of SCF for micronization of particles for food applications has been described by Weidner (2009). Production of powdered food such as chocolate or lecithin at different particle size can be obtained through RESS, SAS and PGSS (Particle from Gas Saturated Solutions) (Weidner, 2009).

4.2.2. Preservation

Food preservation aims at conserving organoleptic properties and guarantees safety in food consumption. Food deterioration may be caused by several factors such as micro-organisms development and endogenous enzymatic activity. The use of SCF or high pressure gases for preservation by sterilization, for microbial, virus and spore inactivation has been the subject of some extended reviews (Garcia-Gonzalez et al., 2007; Perrut, 2012; Spilimbergo, Elvassore, & Bertucco, 2002).

High hydrostatic pressure has been known to enable sterilization and pest control (Perrut, 2012), but the required pressure is quite high e.g. between 2 and 3000 bars (Spilimbergo et al., 2002). Requiring milder conditions for similar results, the use of SCF appears as a suitable alternative for food preservation. Early studies reported that relatively

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Table 3
Applications of supercritical fluids in food transformation, preservation and extraction.

Application	Matrix (target molecule)	Processing conditions	Benefits	Reference
Transformation				
Encapsulation	Jabuticaba skins (anthocyanins)	RESS process P_{CO2}: 200 bars, 40 °C, co-solvent: EtOH, encapsulation in polyethylene glycol	79.78% encapsulation efficiency Stability of anthocyanins to light and temperature, ease of dissolution in solvent	(Santos, Albarelli, Beppu, & Meireles, 2013)
	Rosemary (antioxidants)	SAS process P_{CO2}: 80 to 100 bars, 25 to 50 °C, solvent: EtOH, encapsulation in poloxamers (Pluronic® F88 or Pluronic® F127)	100% encapsulation efficiency Quick dissolution in aqueous solution (1 h), increased protection against degradation factors during dissolution.	(Visentin, Rodríguez-Rojo, Navarrete, Maestri, & Cocero, 2012)
Fractionation	Oregano, sage, thyme, marigold (essential oils)	Extraction: P_{CO2}: 300 bars, 40 °C Fractionation in separators (S): 100 bars (S1) and 50 bars (S2)	Separation of waxes in first separator and maximal recovery of essential oil obtained in second separator (> 70%)	(Fornari, Vicente, Vázquez, García-Risco, & Reglero, 2012)
	Grape seed (phenolic and lipid compounds)	Fraction 1: pure CO_2 (456 bars, 35 °C, 15 min static, dynamic phase: solvent to feed ratio: 16.6) Fraction 1: CO_2 + methanol (5:1, v/v) (456 bars, 35 °C, 15 min static, dynamic phase: solvent to feed ratio: 16.6)	Fraction 1: composed of fatty acids, aliphatic aldehydes and sterols (10.6% yield) Fraction 2: phenolic compounds 7.9% yield (catechin, epicatechin and gallic acid).	(Palma et al., 1999)
Textural modification	Corn and potato (extrudate production)	SCFX process with whey powder/egg white incorporation. Die temperature: 60 °C, screw speed: 100 rpm, die CO_2 pressure: 100 to 150 bars	Enhanced expansion, reduction of starch degradation and homogeneous microcellular structure compared to steam extrudates	(Alavi, Gogoi, Khan, Bowman, & Rizvi, 1999)
	Wheat flour	SCFX process, twin screw extruder, Die temperature: 80 °C, screw speed: 300 to 400 rpm, die CO_2 pressure: 10 bars	CO_2 injection allowed lower processing temperatures and reduced loss of thiamine (3 to 11% against 10 to 16%) and lower water absorption index.	(Schmid, Dolan, & Ng, 2005)
Crystallization/ precipitation	Lycopene	SAS process, P_{CO2}: 7 to 150 bars, 35 to 45 °C, solvent: dichloromethane	Yields above 95% Increasing pressure leads to increase of particle size and higher initial concentration leads to smaller particles.	(Miguel, Martín, Gamse, & Cocero, 2006)
	Shrimp residues (astaxanthin)	SAS process, P_{CO2}: 100 to 120 bars, 35 to 40 °C, solvent to feed ratio: 1, solvent: acetone, co-precipitation with Pluronic® F127	74% encapsulation efficiency, higher color preservation compared to crude extract	(Mezzomo et al., 2012)
Preservation				
Bacteria inactivation	*Escherichia coli* (carrot)	P_{CO2}: 120 bars, 35 °C, 10 min	Non detectable levels	(Galvanin et al., 2014)
	Listeria monocytogenes (dry cured ham)	P_{CO2}: 120 bars, 50 °C, 25 min	Non detectable levels	(Galvanin et al., 2014)
Spore inactivation	*Alicyclobacillus acidoterrestris* (apple juice)	P_{CO2}: 80 bars, 70 °C, 30 min	Non detectable levels	(Bae, Lee, Kim, & Rhee, 2009)
	Bacillus subtilis (suspension)	P_{CO2}:80 bars, 35 °C, 30 min (30 cycles)	Complete inactivation	(Spilimbergo et al., 2002)
Enzyme inactivation	Polyphenol oxidase (red beet)	P_{CO2}: 75 bars, 55 °C, 30 min	93% loss of activity	(Liu et al., 2010)
	Pectinesterase (orange juice)	P_{CO2}: 269 bars, 56 °C, 145 min	100% loss of activity	(Balaban et al., 1991)
Drying	Carrot	P_{CO2}: 200 bars, 40 °C to 60 °C, 50 min to 15 min, co-solvent: EtOH (6% mol)	Microstructure and shape conservation, favorable rehydrated textural properties	(Brown, Fryer, Norton, Bakalis, & Bridson, 2008)
Extraction				
Percolation	Tomato wastes (lycopene)	P_{CO2}: 344 bars, 86 °C, 200 min	61% extraction of lycopene	(Rozzi, Singh, Vierling, & Watkins, 2002)
	Almond (oil + tocopherols)	P_{CO2}: 350 to 550 bars, 35 to 50 °C, 10 to 30 kg/h, maximum recovery at 2 to 3 h of extraction	Highest tocopherol enrichment in oil obtained in the first 2 h of extraction. Co-extraction of tocopherol and oil favored at the highest pressures tested.	(Leo, Rescio, Ciurlia, & Zacheo, 2005)
Liquid/liquid extraction	Soybean oil (lecithin)	GASC process, P_{CO2}: 50 to 65 bars, 24.85 °C, soybean oil diluted in 95% hexane	Enrichment of 98.6% of lecithin in solid product	(Mukhopadhyay & Singh, 2004)
	Propolis tincture (essential oil and flavonoid)	SAS process, P_{CO2}: 300 bars, 60 °C, optimum tincture concentration: 10% mass	100% recovery of flavonoids	(Catchpole, Grey, Mitchell, & Lan, 2004)
Pressing	Cocoa nibs (oil)	GAME process, P_{CO2}: 100 bars, 100 °C, effective mechanical pressure: 500 bars	87.1% oil recovery against 71.8% oil recovery for conventional pressing at the same experimental conditions	(Venter et al., 2006)
	Linseeds (oil)	GAME process, P_{CO2}: 100 bars, 40 °C, effective mechanical pressure: 100 bars	30% increase using GAME process by comparing with conventional pressing at the same experimental conditions	(Willems et al., 2008)
Filtration	Carrot oil (beta-carotene)	310 bars, 40 to 60 °C, ΔP: 30 to 50 bars, membranes are nanofilters	Permeate enrichment in beta-carotene (from 9.4 up to 24.2 ppm)	(Sarrade et al., 1998)

GAME: Gas Assisted Mechanical Expression, GASC: Gas Anti-Solvent Crystallization, P_{CO2}: CO_2 pressure, RESS: Rapid Expansion of a Supercritical Solution, SAS: Supercritical Anti-Solvent, SCFX: Supercritical Fluid Extrusion.

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mild conditions were sufficient to inhibit the growth and increase the inactivation rate (heat treatment of 50 to 55 °C with 6 bars of CO_2) (Fraser, 1951; Perrut, 2012). Generally, the microbial inactivation is greatly affected by pressure, temperature, exposure duration and compression/decompression cycles (Garcia-Gonzalez et al., 2007; Melo Silva et al., 2013; Perrut, 2012; Spilimbergo et al., 2002). The presence of water is reported to increase the bactericidal effect of CO_2, most probably due to its relationship with pH, where acidic pH tends to favor inactivation (Garcia-Gonzalez et al., 2007; Garcia-Gonzalez et al., 2009). Some other authors investigated the positive effect of condensed gases (e.g. CO_2, N_2) on microorganisms inactivation. However, it must be underlined that the matrix effect plays a major role during the inactivation process (Garcia-Gonzalez et al., 2007; Wei, Balaban, Fernando, & Peplow, 1991). Recent investigations on food matrices (Table 3) report that inactivation can be obtained with moderate conditions (CO_2 pressure between 80 and 120 bars and below 70 °C). Such conditions enable a complete inactivation or no detectable levels in foods such as carrots, cured ham, apple and orange juice and red beet after treatment.

Enzyme inactivation can be obtained when exposed to SC-CO_2 conditions or dense phase conditions. Reported factors leading to enzyme inactivation are pH lowering and inhibitory effect of molecular CO_2. In that sense, although inactivation can be achieved with others gases, CO_2 is suggested to have a unique role in inactivation (Damar & Balaban, 2006). At very mild conditions (between 1 and 100 bars at temperatures below 60 °C), inactivation of enzymes such as pectin esterase and polyphenol oxidase can be achieved (Damar & Balaban, 2006). It was noted that some enzymes such as lipoxygenase and peroxidase in sucrose solutions required higher pressures for inactivation (100 to 600 bars, below 55 °C) so some enzymes are more pressure-sensitive than others (Hendrickx, Ludikhuyze, Van den Broeck, & Weemaes, 1998; Tedjo, Eshtiaghi, & Knorr, 2000). Early studies report the use of CO_2 microbubbles in batch and continuous systems for enzyme inactivation in liquid materials such as fruit juices (Ishikawa, Shimoda, Shiratsuchi, & Osajima, 1995; Ishikawa et al., 1997; Wimmer & Zarevúcka, 2010).

4.2.3. Extraction

Extraction by SCF is well-known at both academic and industrial level and therefore is the subject of numerous publications. Four types of processes related to extraction are presented in Table 3.

Supercritical extraction of natural products (such as oils and fats, antioxidants, pigments and aromas) by percolation is described in several reviews (Díaz-Reinoso, Moure, Domínguez, & Parajó, 2006; Herrero, Cifuentes, & Ibañez, 2006; Reverchon, 1997; Reverchon & De Marco, 2006). From literature survey, it can be identified that usually high pressures are required (above 280 bars) for extraction of high molecular weight compounds such as oils. Aromatic fractions such as essential oils are extracted using moderate conditions (pressures from 70 to 200 bars and temperatures from 40 to 60 °C) (Reverchon, 1997; Sovová, Aleksovsk, Bocevska, & Stateva, 2006). Focus in extraction by percolation is also set on by-product valorization or on the use of SCF for co-extraction of compounds for enhancement of final product attributes (Table 3). Liquid-liquid extraction is applied for numerous applications such as concentrated aromas production from beverages and alcohol removal (Brunner, 2005; Macedo et al., 2008), oil fractionation and deodorization (Shimoda et al., 2000; Torres, Torrelo, Señoráns, & Reglero, 2009), and hexane removal from vegetable oils (Eller, Taylor, & Curren, 2004). Processing pressures rarely exceed 300 bars.

Combined processes with SCF have been investigated to increase extraction performances. A process combining pressing and use of gases in a supercritical state (Gas Assisted Mechanical Expression, GAME) has been recently investigated to increase oil extraction yield (Voges, Eggers, & Pietsch, 2008). This process has been successfully applied on various seeds (cocoa, linseeds, sesame, Table 3). Authors have noticed that pressing was greatly favored by SCF or dense gases, indeed a low mechanical pressure is necessary (around 10 MPa) to increase oil yield from 10 to 20% (all other conditions equal) (Venter, Willems, Kuipers, & Haan, 2006; Willems, Kuipers, & de Haan, 2008). Nanofiltration coupled to SC-CO_2 extraction for the purification of low molecular weight compounds (1500 $g \cdot mol^{-1}$) was introduced by Sarrade, Rios, and Carlès (1998). This process has been applied to purification of beta-carotene from carrot oil and to fractionation of fish oil triglycerides. Further developments on combination of membrane technologies and SCF are still on going in the field of edible oil refining (Temelli, 2009).

4.3. Success story in the use of supercritical fluids: industrial production

Industrial processing with SCF has been a reality for several years. Since early patents on coffee decaffeination or hops extraction in the 1970's, a great number of industrial units have emerged. In 2009, Perrut estimated that 300 industrial units were using SCF. For supercritical extraction performed on solid materials, the main applications are related to food and perfume industry: aromas and flavors extraction (hops, vanilla, ginger, roses…), coffee and tea decaffeination. For example, Maxwell House Coffee (a division from General Foods) has reported processing 80,000 tons of coffee per year. The plant is equipped with a 60 m^3 extractor and to function in a semi-continuous scale (Benaissi, 2013). The removed caffeine is further sold to pharmaceutic or food companies.

5. Microwave extraction

5.1. Process and procedure

Microwave heating results from the dissipation of the electromagnetic waves in the irradiated medium. The dissipated power in a medium depends on the dielectric properties and the local time-averaged electric field strength. So, there is a fundamental difference between microwave and conventional heating: in conventional heating, heat transfers occur from the heating device to the medium, whereas in microwave heating, heat is dissipated inside the irradiated medium. In contrast with conventional heating, microwave heat transfer is not limited to thermal conduction or convection currents (Fig. 5). In practice, this means that a much faster temperature increase can be obtained. Furthermore, the maximum temperature of the material heated by microwaves is only dependent upon the rate of heat loss and power applied.

Although microwaves create volumetric heating, the field distribution is not even throughout the irradiated material. Therefore, the energy is not homogeneously dissipated. The electric field distribution depends on the geometry of the heated object and the dielectric properties. For media which readily absorb microwaves, the depth at which power density is reduced to 1/e of original intensity might be a limiting factor.

For more transparent media, the occurrence of standing wave patterns will result in 'hot spots' if the power dissipation is faster than the heat transfers to surrounding colder areas. As a general rule a standing wave pattern can occur if multiples of a half wavelength fit in the typical dimension (d) of the irradiated object.

Microwave ovens can have monomode or multimode cavity. The monomode cavity can generate a frequency which excites only one mode of resonance. Their use for food processing is limited because the volume has to be extremely small in order to maintain the resonance. The majority of food heating applications (Edgar & Osepchuk, 2001) use a multimode resonance cavity applicator because it permits large volumes. The incident wave is able to affect several modes of resonance, and this superimposition of modes allows the homogenization of field.

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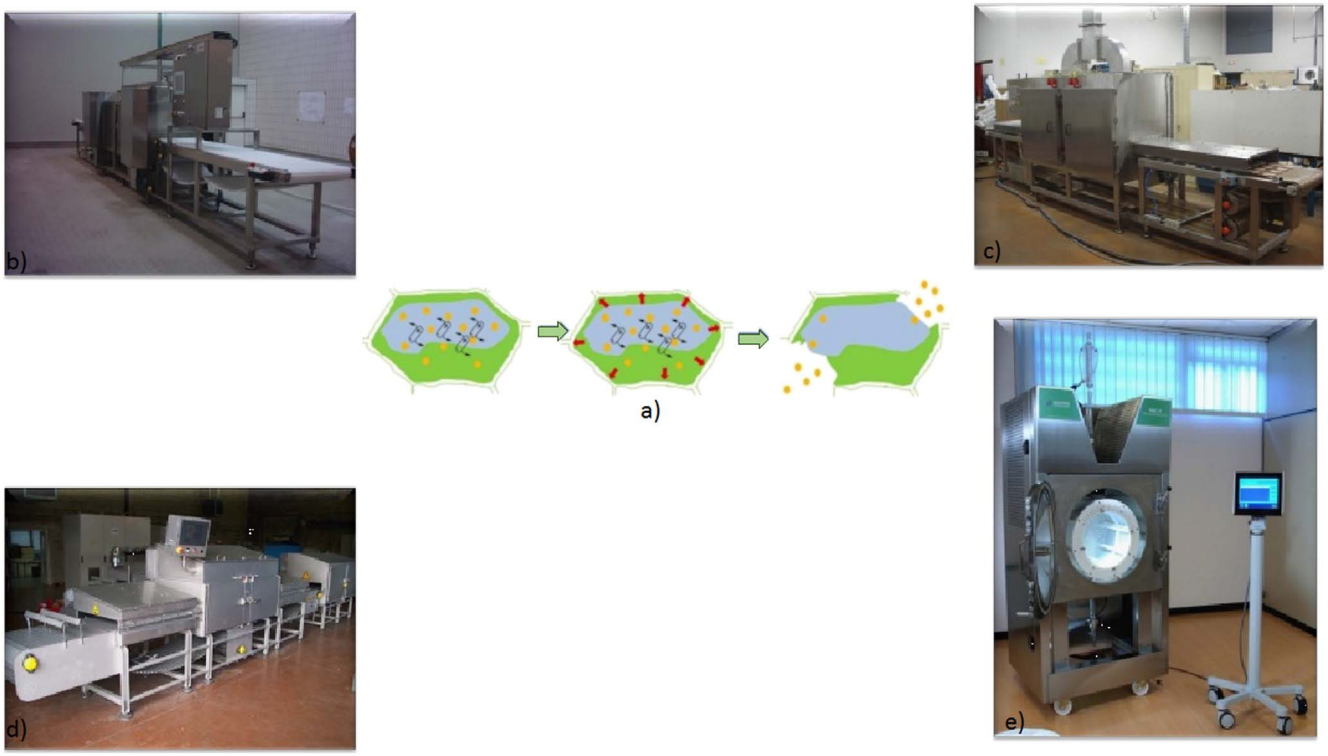


Fig. 5. (a) A brief description of phenomenon in the cell generated by microwave irradiation, (b) microwave tunnel to temper, reproduced with permission from SAIREM, (c) microwave cooking of desserts in containers, reproduced with permission from SAIREM, (d) microwave tunnel for pasteurization of liquid food, reproduced with permission from MES Technologies, (e) microwave extraction: SFME and MHG, reproduced with permission from Millestone.

Microwaves are only absorbed by dipoles, transforming their energy into heat. Heat transfer advantages of applying microwave power, a non-contact energy source, into the bulk of a material include: faster energy absorption, reduced thermal gradients, selective heating and virtually unlimited final temperature. Several processes such as drying, tempering, thawing, blanching, sterilization, pasteurization, baking and

Table 4
Example of applications and experimental conditions of MW.

Application	Matrix	Treatment conditions	Benefits	Reference
Preservation				
Pasteurization	Kiwifruit puree	P: 1000 W, t: 200 s and P: 900 W, t: 225 s; m: 500 g	Inactivation 90% of peroxidase enzyme	Benlloch-Tinoco, Martínez-Navarrete, & Rodrigo, 2014
Thawing	Strawberries	P: 700 W, t: 10 min, m: 250 g	A reduction in processing time, No influence on the quality indices (color, ascorbic acid and anthocyanins contents)	Holzwarth, Korhummel, Carle, & Kammerer, 2012
Sterilization	Palm fruit	P: 800 W, t: 2 min	Increment in lauric acid (C12: 0), Highest concentration of vitamin E and carotene content, Clean technology due to zero water effluent discharge	Cheng, Mohd Nor, & Chuah, 2011
Extraction				
MHG	Grape juice by-products	P: 400 W, t: 20 min, m: 400 g	Green extraction method, Efficiency of MHG in the extraction of polyphenols and anthocyanins from grape by-products	Al Bittar, Périno-Issartier, Dangles, & Chemat, 2013
SFME	Lavender flowers	P: 500 W, t: 10 min	Extraction of essential oils Short extraction time	Chemat et al., 2006
MHG	*Rosmarinus officinalis* L.	P: 500 W, t: 15 min	Extraction of antioxidants Economy in term of time and energy More safer extraction	Abert Vian, Fernandez, Visioni, & Chemat, 2008
Transformation				
Drying	bananas	P: 400 W, magnetron is « on » for 11 s and « off » for 18 s, m: 86 g.	Creation dried-and-crisp fruits by applying successive cycles of heating and vacuum pulses in a microwave field	Monteiro, Carciofi, & Laurindo, 2016
Baking	Cake batter	P: 250 W, t: 67 s, m: 30 g of freshly prepared batter	93% reduction in baking time/convective baking Improvement textural properties such as moisture content and firmness Highest nutritive value	Megahey et al., 2005
Blanching	Brussels sprouts	P: 700 W, t: 5 min followed by blanching in boiling water for 2 min.	No deleterious effects on total flavonoids and ascorbic acid, Improvement health properties of Brussels sprouts	Vina et al., 2007

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extraction have been applied efficiently in the food industry.

5.2. Applications in food processing (Table 4)

Drying is one of the oldest methods of preserving food and can be specified as a simultaneous heat and mass transfer operation in which water activity of a foodstuff is lowered by the removal of water by evaporation into an unsaturated gas stream. The most important characteristic of microwave dehydration is volumetric heating effect where the microwave energy travels through the food and is absorbed more in the wet region than in the dry region of the product. Consequently, the centre is warmer than the surroundings and the mass transfer is accelerated. Microwave drying results in a high thermal efficiency, shorter drying time and improves the final quality of the dried product compared to conventional drying. MW drying is also able to maintain good quality of the product such as color, aroma and texture (Fathima, Begum, & Rajalaksmi, 2001).

Several experimenters have reported microwave-assisted hot-air drying experiments with foodstuffs, where considerable improvements in the drying process have been evident such as better aroma, faster, and better rehydration (Gowen, Abu-Ghannam, Frias, & Oliveira, 2006) than hot-air drying.

Quality can often be improved further by the addition of vacuum: Vacuum Microwave Drying (VMD) offers an alternative way to retain volatile compounds sensitive to losses through thermal and oxidative degradation. Moreover, the absence of air during drying reduces oxidation and therefore, color, texture and nutrient content of products are all improved (Gunasekaran, 1999). The VMD technique was also successfully applied to cranberries, (Yongsawatdigul & Gunasekaran, 1996) model fruits gels (Drouzas, Tsami, & Saravacos, 1999), garlic (Cui, Xu, & Sun, 2003), cabbages (Yangyang, Mujumdar, Le-qun, & Jin-cai, 2004) and button mushrooms (Giri & Prasad, 2006).

In addition to hot air, microwaves can also be combined with freeze drying. Freeze drying is used for heat sensitive product and almost no oxygen is involved in the process. However, this technique is costly, time and energy consuming. In this system, the energy is directly absorbed by the water molecules for sublimation within the material and increases the drying rate in freeze drying.

Blanching is a thermal treatment prior to freezing with the aim of inactivation of enzymes such as polyphenoloxidase (PPO) and peroxidase (POD) that are responsible for browning reactions and lead to off-flavor development. Blanching also destroys microorganisms on the product surface and makes some vegetables more compact. Microwave blanching could be an alternative to conventional blanching, with precise process control, shorter processing time and less energy use and decreases the blanching time of the product centre. Many studies concluded that the microwave technique leads to firmer products, equal or better nutrient contents and similar colors when compared to conventional processing (Brewer & Begum, 2003; Lin & Brewer, 2005).

Tempering can be considered as the initial phase of the complete thawing process. **Thawing** of frozen foods is an important unit operation in the food industry. Large quantities of food have to be preserved by freezing at harvest time for use throughout the year. In microwave thawing, electromagnetic waves are directly absorbed by the product without the use of conductors or electrodes. Therefore, it is a very fast thawing method but its application is limited by product thermal stability. The problem is complex because the water loss factor is approximately 12 whereas that of ice is 0.003, which means that the ice is almost insensitive to microwave energy. In a frozen product (− 18 °C), unfrozen water preferentially absorbs the microwave energy whereas the frozen part is insensitive to microwaves. This leads to localized areas of very hot water, partial thawing and thermal runaway (Schiffmann, 2001). Improvements to maintain a uniform temperature during microwave thawing are necessary.

The advantage of microwave thawing was the reduction in processing time, even when there was no significant effect on product quality. In the food industry, microwave ovens are often part of continuous processes. Thin layers of product circulate on a conveyor under a succession of microwave generators. Microwaves are used to thaw fish fillets and meat blocks.

Microwave tempering reduces costs of manufacturing prepared foods as frozen raw materials can be tempered as needed, with no drip loss, without the need for tempering rooms and reduced processing time.

Baking is a complex process, described as a simultaneous heat and mass transfer. Baking involves a series of physical, chemical and biochemical changes in food, such as starch gelatinization, protein denaturation, carbon dioxide liberation from leavening agents, volume increase, water evaporation, crust formation and non-enzymatic browning (Therdthai & Zhou, 2003).

Microwave baking reduces the baking time and energy (Sumnu, 2001). Megahey, Mcminn, and Magee (2005) illustrated this by comparing the time to bake a cake by microwave and conventional baking. Microwave baking allowed for up to a 93% reduction in baking time, relative to convective baking. Cakes baked by microwaves showed improved textural properties such as moisture content and firmness. Another advantage of microwave baking is that the final product has a higher nutritive value.

Pasteurization is a thermal inactivation of pathogenic microorganisms, notably vegetative cells, yeasts and moulds. **Sterilization** is the inactivation of microorganisms and their spores, which are generally more thermo-resistant than vegetative cells. The microwave heating of food provides an excellent opportunity to pasteurize or sterilize the products. Products such as sweet mash potato (Coronel, Truong, Simunovic, Sandeep, & Cartwright, 2005; Steed et al., 2008), a biphasic food product (salsa con queso) (Kumar, Coronel, Simunovic, & Sandeep, 2007), green beans and mash carrot (Kumar et al., 2008), were treated and the feasibility of microwave sterilization was confirmed. The continuous pasteurization and sterilization of liquids with microwave equipment are a useful alternative processing approach but the price and the energy consumption are relatively high.

5.2.1. Extraction

Use of microwave energy was described for the first time in 1986 by Ganzler (Ganzler, Salgo, & Valko, 1986) and Lane (Lane & Jenkins, 1984) for extraction of food ingredients. In the last decade there has been an increasing demand for new extraction techniques, amenable to automation, with shortened extraction times and reduced organic solvent consumption, to prevent pollution and reduce the cost of sample preparation. Driven by these goals, advances in microwave green extraction have given rise to two classes of techniques: Solvent Free Microwave Hydrodistillation and Microwave Hydrodiffusion and Gravity.

Solvent Free Microwave Hydrodistillation (SFME) was conceived for laboratory scale applications in the extraction of essential oils from different kinds of aromatic plants and fruits (Chemat, Lucchesi, and Smadja (2004a, 2004b)) SFME apparatus is an original combination of microwave heating and distillation at atmospheric pressure. Based on a relatively simple principle, this method involves placing plant material in a microwave reactor, without any added organic solvent or water. The internal heating of the in situ water within the plant material distends the plant cells and leads to rupture of the glands and oleiferous receptacles. Thus, this process frees essential oil which is evaporated by the in situ water of the plant material. A cooling system outside the microwave oven condenses the distillate continuously. The water excess is refluxed to the extraction vessel in order to restore the in situ water to the plant material. Therefore, the SFME method offers a reduced environmental burden as it rejects less CO_2 in atmosphere (200 g CO_2 per gram of essential oil compared to traditional method which was rejecting 3600 g CO_2 per gram of essential oil) (Chemat et al., 2004a, 2004b; Ferhat, Meklati, Smadja, & Chemat, 2006).

Microwave Hydrodiffusion and Gravity (MHG) extraction was

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patented by Chemat et al., 2008. This green extraction technique is an original "upside down" microwave alembic combining microwave heating and earth gravity at atmospheric pressure. MHG was conceived for laboratory and industrial scale applications for the extraction of food ingredients from different kind of fruits, vegetables and aromatic plants. This method involves placing plant material in a reactor inside the microwave oven, without adding any solvent or water. Microwaves induce warming of the water contained in the matrix, which allows the destruction of cells containing essential oil. Essential oils, as well as the internal water of the matrix, are released and transferred from inside to the outside of the plant: this is the hydrodiffusion phenomenon. A cooling system placed outside the microwave oven allows the condensation of the distillate. It is important to note that this green method allows extract of essential oils without distillation and evaporation which are the most energy consuming processes between the unit operations (Périno-Issartier, Giniès, Cravotto, & Chemat, 2013).

5.3. Success story

Using microwaves, full reproducible food processes can now be completed in seconds or minutes with high reproducibility, reducing the processing cost, simplifying manipulation and work-up, giving higher purity of the final product, eliminating post-treatment of waste water and consuming only a fraction of the time and energy normally needed for a conventional processes heated by convection, conduction, or radiation. For food production, the resultant value could include: more effective heating, fast heating of packaged food, reduced equipment size, faster response to process heating control, faster start-up, increased production, and elimination of process steps.

6. Ultrasound assisted food processing

Ultrasound is a sound frequency in the range between 18 and 100 kHz that is above hearing of the human ear. High power ultrasound means application of intensities higher than 1 $W{\cdot}cm^{-2}$ (usually in the range between 10 and 1000 $W{\cdot}cm^{-2}$). High power and low frequency ultrasound (f = 20 to 100 kHz) is considered as "power ultrasound" because its application causes cavitation and is applied in the food industry (Paniwnyk, 2005). The benefits of US are attributed to acoustic cavitation: micro-bubbles created in a liquid phase when subjecting a mixture to US will grow and oscillate quickly before collapsing due to pressure changes (Chemat et al., 2008; Jambrak, Mason, Lelas, Herceg, & Herceg, 2008) (Fig. 6a). These violent implosions will fragment or disrupt the surface of the solid matrix, enhancing mass transfer and accelerating diffusion.

The effectiveness of the ultrasound depends to the acoustic frequency, temperature and pressure applied. Lower frequencies generate larger bubbles and thus a more violent bubble collapse with higher localized temperatures and pressures. However, as frequency is increased, there are more collapse events per unit time. Two different types of ultrasound equipment are commonly used in laboratory (Fig. 6). The first one is the ultrasonic cleaning bath (Fig. 6b) which is commonly used for solid dispersion into solvent (ultrasounds will dramatically reduce the size of the solid particles, which will enhance its solubility), for degassing solutions or even for cleaning small material by immersion of the glassware into the bath. The ultrasonic baths are less used for chemical reactions even if they are easy to handle and economically advantageous because reproducibility of reaction is low. In fact, the delivered intensity is low and is highly attenuated by the water contained in the bath and the walls of the glassware used for the experiment. The second one, the ultrasonic probe or horn system (Fig. 6d), is much more powerful because the ultrasonic intensity is delivered on a small surface (only the tip of the probe) compared to the ultrasonic bath. Another change is that the probe is directly immersed into the reaction flask so less attenuation can happen. This system of probe is widely used for sonication of small volumes of sample but special care has to be taken because of the fast rise of the temperature into the sample. A special sono-extraction reactor (from 0.5 to 3 L) has been developed by REUS (www.etsreus.com, FRANCE) (Fig. 6c) The intensity of ultrasounds is about 1 W/cm^2 with a frequency of 25 kHz. In order to keep constant temperature, the reactor is made of a double mantle into which cooling water can circulate. The main advantage of this type of apparatus is that the natural products and the extraction solvent are mixed into a container and the ultrasounds are directly applied to the mixture. To run out industrial trials or to scale-up laboratory experiments, REUS has also developed reactors from 30 to 1000 L (Fig. 6f, g). Pump systems are coupled to the ultrasonic bath in order to fill the ultrasonic bath, to stir the mixture and to empty the system at the end of the experiment. The manufacturers of high-power ultrasound equipment have been also focusing on designing devices by including specific operational features such as continuous flow mode. The equipment basically consists of a glass tube or stainless steel reactor, through which the fluid mixture is pumped, surrounded by a jacket filled with pressurized water for conduction of the sound waves. Ultrasound is transmitted to the system by a sonotrode attached to the jacket (Fig. 6e).

6.1. Application in food processing

There are a large number of potential applications of high intensity ultrasound in food processing of which a number are discussed below (Table 5).

6.1.1. Degassing/deaeration

A liquid contains gases as a mixed condition, such as dissolved oxygen, carbon dioxide, nitrogen gas etc. Two common methods used for degassing are boiling and reducing pressure while ultrasound has an advantage in the small temperature change. Degassing in an ultrasonic field is a highly visible phenomenon when ultrasound, e.g. an ultrasonic cleaning bath, is used with regular tap-water inside. It occurs when the rapid vibration of gas bubbles brought them together by acoustic waves and bubbles grow to a size sufficiently large to allow them to rise up through the liquid, against gravity, until they reach the surface (Laborde, Bouyer, Caltagirone, & Gerard, 1998; Tervo, Mettin, & Lauterborn, 2006). Several acoustic cavitation structures generated in low-frequency ultrasound fields within the range (20–50 kHz) have been investigated and these have been summarized by Mettin (2005). In the food industry, this technique can be used to degas carbonated beverages such as beer (defobbing) before bottling (Brown & Goodman, 1965).

6.1.2. Demouling

Generally, the industrial cooking of foods leads to adhesion of the products to the cooking vessel or in other operations it must detach from its mould. At present, to solve this problem mechanical methods such as knocking vibration are used to remove the products. An alternative solution to these conventional methods is to release food products by coupling the mould to a source of ultrasound (Scotto, 1988). The device for demoulding industrial food products couples the mould and the ultrasonic source in order to enhance removal of the product contained in the latter by virtue of the high-frequency relative movement between the contact surfaces of the mould and of the product contained in the latter. This technique allows surface coatings to be eliminated and ensures that any residual material in the mould can be cleaned automatically.

6.1.3. Cutting

The introduction of ultrasound in food cutting has improved the performance of overall food processing. Ultrasonic food cutting equipment provides a new way to cut or slice a variety of food products that streamlines production, minimizes product waste and lowers maintenance costs. Ultrasonic cutting uses a knife-type blade attached through

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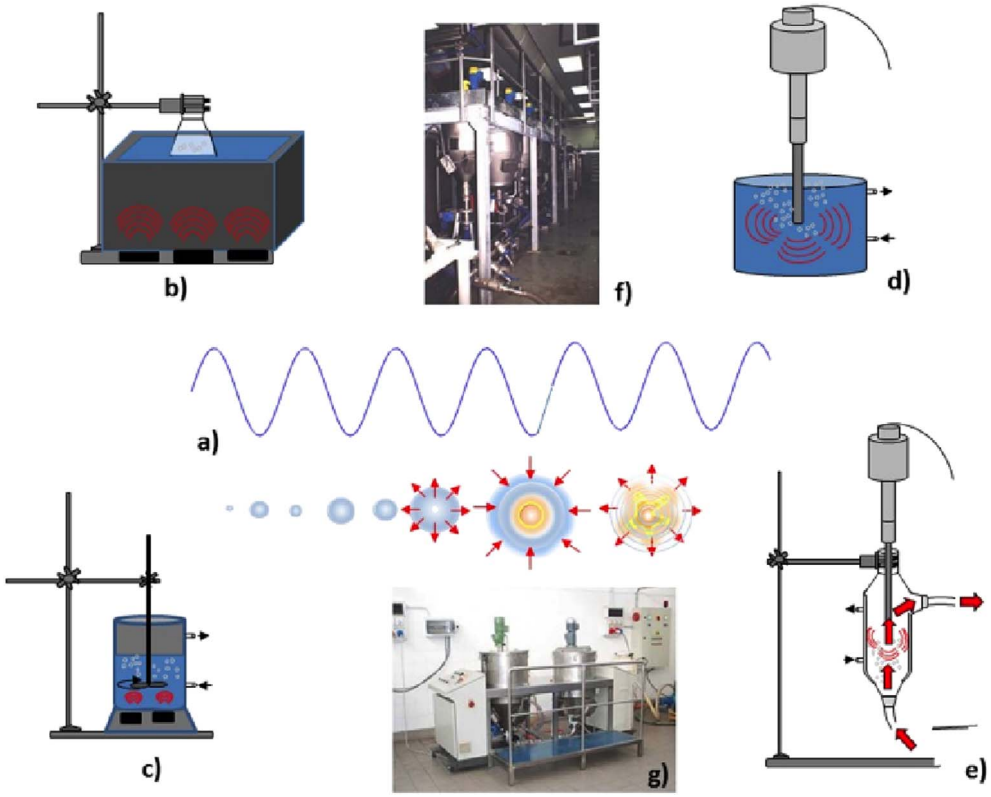


Fig. 6. a) Acoustic cavitation, b) ultrasonic bath, c) sono-extractor by REUS, d) ultrasonic probe, e) continuous ultrasonic probe system, f) industrial ultrasonic equipment (500 L), g) industrial ultrasonic equipment (50 L).

a shaft to an ultrasonic source (Rawson, 1988). The cutting tool itself can be of many shapes and each shape can be considered to be an acoustic horn, part of the whole ultrasonic resonating device. Cutting with the superimposition of ultrasonic vibration is a direct competitor of technologies such as high-velocity water jet cutting and conventional techniques such as using saws or knives. The low energy requirements for ultrasonic cutting have been presented (Schneider, Zahn, & Rohm, 2008; Schneider, Zahn, Schindler, & Rohm, 2009). The ultrasonic cutting characteristics depend on the food type and condition, e.g. frozen or thawed (Brown, James, & Purnell, 2005). The most widespread application of ultrasound is in the cutting of fragile foodstuffs. It uses in the particular cases of fragile and heterogeneous products (cakes, pastry and bakery products) and fatty (cheeses) or sticky products (Arnold, Leiteritz, Zahn, & Rohm, 2009).

6.1.4. *Meat tenderization*

The quality of meat depends on the aroma, flavor, appearance, tenderness and juiciness. Consumer behaviour has shown that tenderness is most important palatability factor in determining meat quality (Smith, Cannon, Novakofski, McKeith, & O'Brien, 1991). The traditional method used for meat tenderization is mechanical pounding, which makes poorer quality meat more palatable. Power ultrasound has also been found to be useful for this process. Ultrasound can act in two ways: by breaking the integrity of muscular cells or by enhancing enzymatic reactions, i.e. via a biochemical effect (Boistier-Marquis, Lagsir-Oulahal, & Callard, 1999). A pilot study involving sirloin steak (Roberts, 1991) showed that sonicating beef muscle at 2 $W{\cdot}cm^2$ for 2 h at 40 kHz produced damage to the perimysial connective tissue, resulting in improved eating texture.

6.1.5. *Food preservation*

Microorganisms and enzymes are the primary factors responsible of food deterioration. Conventional thermal processing kills vegetative microorganisms and some spores, and inactivates enzymes. However, the time and temperature of the process are proportional to the amount of nutrient loss, development of undesirable flavors and deterioration of functional properties of food products. Ultrasound is one of the new preservation techniques that could eliminate microbial activity. High power ultrasound alone is known to disrupt biological cells. When combined with heat treatment, it can accelerate the rate of sterilization of foods. Therefore, it reduces both the duration and intensity of the thermal treatment and the resultant damages. At sufficiently high acoustic power inputs, ultrasound is known to rupture cells (Chisti, 2003; Chisti & Moo-Young, 1986; Dakubu, 1976). A cell can be inactivated at an intensity less than that needed to cause disruption. The mechanism of microbial killing is mainly due to the thinning of cell membranes, localized heating and production of free radicals (Butz & Tauscher, 2002). There are many examples of microorganisms inactivated using ultrasound. Some of these have been studied in culture media and others in food, using ultrasound either combined or alone. The most frequently studied microorganisms, not only in the field of power ultrasound, but also among other methods of food preservation are *Saccharomyces cerevisiae* and *Escherichia coli*. The former has been found to be less resistant to ultrasound than other vegetative cells, which is mostly attributed to its larger size. The inactivation of this microorganism has been proven in such food models as water, phosphate buffers, and sabouraud broth (Ciccolini, Taillandier, Wilhem, Delmas, & Strehaiano, 1997; Guerrero, Lopez-Malo, & Alzamora, 2001; Petin, Zhurakovskaya, & Komarova, 1999). The inactivation of *Staphylococcus aureus*, *Pseudomonas fluorescens*, *Listeria monocytogenes* and *E. coli* has been proven in water and phosphate buffers, as well as in foods such as UHT milk. Ultrasonication in combination with heat was performed to study the inactivation of *Listeria innocua* and mesophilic bacteria in raw whole milk. When applying ultrasound in combination with heat the kill rates were increased when compared to rates of thermal treatment alone and a synergistic rather than an additive effect was observed. Ultrasound produced a good level of inactivation under different treatment

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Table 5
Example of applications and experimental conditions of US.

Application	Matrix	Treatment conditions	Benefits	Reference
Preservation				
Inactivation	Orange juice	500 kHz, 240 W, 15 min	Inactivation of total mesophilic aerobes and fungi	Valero et al., 2007
	Milk	24 kHz, 400 W, 55 °C, 2,5 min	Inactivation of *Listeria innocua*	Noci, Walkling-Ribeiro, Cronin, Morgan, & Lyng, 2009
	Phosphate buffer	20 kHz, 100 W, 40–61 °C, 100–500 kPa, 0,25-4 min	Inactivation of *Escherichia coli*	Lee, Zhou, Liang, Feng, & Martin, 2009
Extraction				
Polyphenolic compounds	Citrus peel	US bath, 60 kHz, 15 °C–40 °C, 2 g/40, 80% methanol, 1 h	Higher extraction efficiency by US compared to maceration. Low extraction temperature leads to higher yields.	Barbero, Liazid, Palma, & Barroso, 2008
Vanillin	Vanilla pods	US horn, 22.4 kHz 1 h pulsed mode (5 s. ON/5 s OFF) 1 g/100 mL solvent	140 ppm vanillin concentration in 1 h vs 180 ppm in 8 h conventional Soxhlet	Jadhav, Rekha, Gogate, & Rathod, 2009
Lycopene	Tomatoes	US bath, 40 kHz, 300 W, 1 g/8 mL of ethyl acetate, sonicated for 29 min at 86 °C;	90% total lycopene extracted in 29 min, yield enhances when coupling US with microwaves	Liangfu & Zelong, 2008
Transformation				
Cutting	Cheese	Guillotine sonotrode, 40 kHz, cutting velocity 2500 mm.min^{-1} and ultrasonic amplitude 12 μm.	Advantages: Faster, more efficient and effective cutting Non-deformation of the products. The knife is self-cleaning due to the ultrasonics which are transmitted from the generator.	Arnold et al., 2009
Degassing	Milk	20 kHz, pulsed ultrasound (1 s/1 s), 20 °C, 3 min	Effective procedure to remove foam and dissolved oxygen in supersaturated milk	Villamiel, Verdurmen, & de Jong, 2005
Meat tenderization	Jumbo squid	25.6 kHz, 186.9 W, 30.8 min	Tenderization of jumbo squid without compromising the other quality parameters. The predicted values of the texture, including the flexibility and the firmness, were 2.40 mm and 435.1 g, respectively.	Hu et al., 2014

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Table 6
Characteristics, main disadvantages and advantages of green extraction techniques.

Technique	Investment	Sample size	Processing time	Main disadvantages	Main advantages
Ultrasound	Low	600 L	Low	Problem for separation	High cell disruption
Microwave	Medium	150 L	Low	Hot spots	Cell disruption
DIC	High	100 L	Low	High energy consumption	High cell disruption
SFE	High	300 L	Medium	Need of know-how	Enhance mass transfer
PEF	High	Continuous	Medium	Difficult ease of operation	Electroporation of wall cells

conditions and media for Bacillus species. The inactivation of *Listeria monocytogenes* by high-power ultrasonic waves (20 kHz) at ambient temperature and pressure has been found to be low with decimal reduction values in 4.3 min. This could be improved however either by an increase in pressure (manosonication) or by increasing the power of sonication. Inactivation by manothermal sonication (MTS) proved to be more effective (Manas, Pagan, Raso, Sala, & Condon, 2000; Pagan, Manas, Alvarez, & Condon, 1999).

For stabilization of some food materials, enzymes must be inactivated or their activity reduced. Enzyme inactivation can be easily achieved by heat treatment. However, in some cases the high heat resistance of enzymes may be a problem as heat can negatively modify some food properties such as flavor, color or nutritional value. This is the driving force for the increased interest in an alternative method of enzyme inactivation: high power ultrasound, i.e. sonic waves above 20 kHz. The effects of ultrasonic waves on proteins are very complex. Polymeric globular proteins are broken down into subunits and if oxygen is present, the quaternary structure is not recoverable. A partial delipidation of lipoproteins can be obtained and if the ultrasonic irradiation is long enough, proteins can be hydrolysed and polypeptide chains can be broken. The influence of the gas on the intensity of enzyme inactivation has been related to the formation of free radicals by cavitation. Sensitivity to ultrasounds depends on the conditions of the treatment (McClements, 1995) as well as on the nature of the enzyme. Generally, ultrasonication in combination with other treatments is more effective in food enzyme inactivation. In fact, MTS treatment has an increased effectiveness compared with ultrasound alone (Manas et al., 2000). MTS treatments inactivate several enzymes at lower temperatures and/or in a shorter time than thermal treatments at the same temperatures.

6.1.6. Extraction

Ultrasound assisted extraction is an emerging potential technology that can accelerate heat and mass transfer and has been successively used in extraction field. Ultrasound waves after interaction with subjected plant material alter its physical and chemical properties and their cavitational effect facilitates the release of extractable compounds and enhances the mass transport by disrupting the plant cell walls. Ultrasounds are successively employed in plant extraction field. Several classes of food components such as aromas, pigments, antioxidants, and other organic and mineral compounds have been extracted and analyzed efficiently from a variety of matrices (mainly animal tissues, food and plant materials). Riera et al. (2004) examined the effect of ultrasound (20 kHz and 50 W) on the particulate almond oil extraction kinetics using supercritical CO_2. As a consequence of the trials (at 280 bar and 55 °C) at the end of the extraction time (8 h 30 min) the yield of the oil was significantly increased (20%) when SFE was assisted by ultrasound. Alternatively, mass transfer was speeded up to such an extent that yields comparable to those obtained by SFE alone could be achieved in about 30% shorter time when using ultrasound.

6.2. Success stories

The considerable interest in high-powered ultrasound is due to its promising effects in food processing and preservation, such as higher product yields, shorter processing times, reduced operating and maintenance costs, improved taste, texture, flavor and color, and the reduction of pathogens at lower temperatures. It can be applied not only to improve the quality and safety of processed foods but offers the potential for developing new products with unique functionality as well. Nevertheless, although conventional cutting, emulsification and cleaning are often bottlenecks, lack of knowledge keeps industry from implementing ultrasound in their processes.

A recent survey and market study of the possible future applications of new process technologies (like microwave, ultrasound) in the food industry has revealed that many companies are reluctant to apply these new technologies. The main reason is poor understanding of these new techniques by food professionals and the reason or weight of tradition.

7. Comparison of techniques

The use of innovative extraction techniques such as ultrasound, microwave, instant controlled pressure drop, supercritical fluid, and pulsed electric fields (Table 6) allows reduced extraction time, energy consumed, and less water or solvent. Conventional techniques are limited by the diffusion of water or solvent into biomass, due to rigid structure of cell walls of microorganisms. The solution could be to enhance the diffusion of water or solvent and to disrupt cell walls. For example, ultrasound and electric pulse fields allow in a high disruption of cell, which permits accelerating the mass transfer, thus, processing time is reduced. In another hand, heating by microwave induces combined mass and heating transfer that permits the destruction of cells and liberation of metabolites. As a future trend is to have a decision tool which permits selecting a technology regarding the initial material. For example, the choice of which technique has to be used to perform extraction of a desired metabolite from a specific plant has to be a result of a compromise between the efficiency and reproducibility of extraction, ease of procedure, together with considerations of cost, time, safety and degree of automation.

Another challenge for the food industry in the coming years, such as consumer and society demand on one side high quality, safe, nutritional processed foods but also in another side reduction of waste and awareness about climate change. Environmental studies are difficult in particular with food products. Ideally a complete LCA study should necessarily include agricultural production, industrial refining, storage and distribution, packaging, consumption and waste management, which comprise large and complex systems (Sonnemann and Margni (2015); Peano et al. (2012); Pardo and Zufía (2012).).

8. Conclusions and perspective

Food processing even preservation, transformation or extraction takes an important place in manufacturing processes and is linked to several drivers such as request for naturally derived ingredients (colors, antioxidants, antimicrobial, aromas…) by the consumers, and the need of standardization. Green Food Processing could be a research thematic that encompasses a comprehensive strategy based on the discovery and the design of processes in order to reduce energy and water consumption. It has been investigated mainly at laboratory scale by several research teams in Europe mostly, and represents a good opportunity to

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Fig. 7. Practical work on Green Food Processing for Master students at Avignon University and Introduction to green extraction for primary schools at Avignon University

rationalize eco-friendly developmental and industrial practices.

The food industry is a very competitive environment and to survive they have to use optimized processes and to reduce the carbon food print. The concept of Green Food Processing meets the demand of the final consumer in term of greener product, an education work will have to be done in order to explain what the benefits are for the final consumer. This educational work will require vulgarization from the scientific community and industry members and avoid the shortcuts of "green washing". For example, in Avignon University to illustrate an application of Green Food Processing in teaching laboratories, we used green procedures employing ultrasound energy and microwave energy as energy source to teach fundamental food processing concepts such as marinating, maceration and extraction. As an example, we have developed new green procedure, using microwave energy as energy source, to teach the fundamental concepts of extraction of essential oils used for aromatisation of food products. The objective of this teaching was to offer students the opportunity to compare the potential of this green technique for extraction of essential oil with a traditional hydro-distillation method (used in all the teaching laboratories all over the world) and to appreciate the benefits of using greener processing method: reduction in time, energy and water consumption. These green food-processing techniques could be easily understood by younger people. Each year, we open our research laboratory during the open week of "La fête de la science" and show the innovations about processing food especially extraction of food ingredients (aromas, colors…) with original techniques such as microwave and ultrasound (Fig. 7).

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