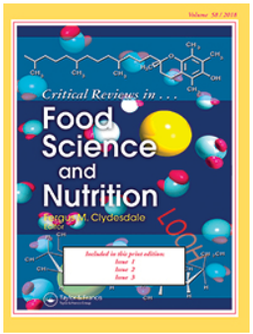

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Current technologies and new insights for the recovery of high valuable compounds from fruits by-products

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ABSTRACT

The recovery of high valuable compounds from food waste is becoming a tighten issue in food processing. The large amount of non-edible residues produced by food industries causes pollution, difficulties in the management, and economic loss. The waste produced during the transformation of fruits includes a huge amount of materials such as peels, seeds, and bagasse, whose disposal usually represents a problem. Research over the past 20 years revealed that many food wastes could serve as a source of potentially valuable bioactive compounds, such as antioxidants and vitamins with increasing scientific interest thanks to their beneficial effects on human health. The challenge for the recovery of these compounds is to find the most appropriate and environment friendly extraction technique able to achieve the maximum extraction yield without compromising the stability of the extracted products. Based on this scenario, the aim of the current review is twofold. The first is to give a brief overview of the most important bioactive compounds occurring in fruit wastes. The second is to describe the pro and cons of the most up-to-dated innovative and environment friendly extraction technologies that can be an alternative to the classical solvent extraction procedures for the recovery of valuable compounds from fruit processing. Furthermore, a final section will take into account published findings on the combination of some of these technologies to increase the extracts yields of bioactives.

KEYWORDS

Fruit waste; bioactive compounds; innovative extraction techniques; combined technologies

Introduction

Food wastes are nowadays considered a serious social, economic, and environmental problem. According to the Food and Agriculture Organization (FAO) data (Santana-Méridas et al., 2012; Fao, 2013; Lipinski et al., 2013), approximately one third of all food produced for human consumption in the world is lost or wasted. This corresponds to approximately 1.6 billion tons per year, 54% of which is lost during the production steps, postharvest handling, and storage. The remaining 46% is further lost along the food chain, mainly during the retail distribution and domestic consumption.

Considering the lost only derived from the processing steps of fruits, about 30–40% (w/w wet basis) is discarded as waste (Galanakis, 2012). The typical kind of fruit waste comprises peels, seeds, and bagasse. Due to their high water content, such materials are prone to microbial spoilage (Djilas et al., 2009); therefore, drying is necessary before further exploitation. However, the equipment cost of drying, storage, and transport hinder the economical sustainability of fruit waste re-utilization; a further drawback is caused by the low revenue of dried fruit-waste when this is utilized just as animal feed or fertilizer in agriculture.

There is a growing evidence that the production of feeds and fertilizers from fruit wastes does not take into account the real potentiality of this material.

Researchers over the past 20 years have effectively demonstrated that many of the food wastes could serve as a source of potentially valuable bioactive compounds such as minerals, vitamins, sugars, carotenoids, fibers, phenols, aromatic compounds, and so on. In human nutrition, there is a growing evidence that these bioactive compounds possess antioxidant, anticancer, anti-inflammatory, and antiviral properties. For example, phenols and carotenoids from fruit wastes could be applied as natural preservatives to extend the shelf life of food or beverage by delaying the formation of off-flavors and rancidity. Pectin, a polysaccharide extracted mainly from citrus peel and apple pomace, is well known as gelling agent in confectionary. Recently, its use was also proposed as fat replacer in meat products (Fernandez-Gines et al., 2005). Furthermore, dietary fibers are also able to improve intestinal motility and thereby are destined to supplement food products or ready meals (Rodríguez et al., 2006).

However, most of the fruit wastes are currently not exploited. One of the reasons is the lack of appropriate and commercially available strategies for the extraction and provision of these compounds (Wijngaard et al., 2012).

Recently, new innovative and technological solutions have been proposed to selectively extract bioactive compounds and, at the same time, meet the environment friendly criteria. In this context, technologies employing fluids at high pressures (subcritical and supercritical states), high hydrostatic pressures, pulsed electric fields, membrane filtration, enzyme extraction, ultrasounds, and microwaves represent good alternatives.

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Based on this scenario, the present review aims to describe the main valuable bioactive compounds occurred in fruit wastes and the most innovative and environment friendly technologies employed for their extraction and recovery. Furthermore, the most recent results dealing with the combination of different extraction technologies to enhance the extract yields will be also presented.

Bioactive compounds from fruits

Bioactive compounds are believed to impart health benefits beyond basic nutrition (Guaadaoui et al., 2014). Fruit wastes are potential sources of these compounds. In the following sections, the main classes of bioactive compounds in fruits are briefly reported.

Phenolic compounds

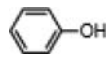
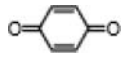
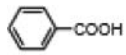
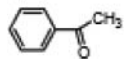
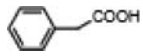
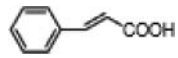
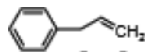
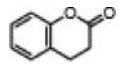
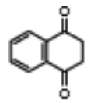
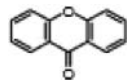
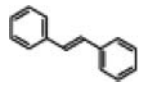
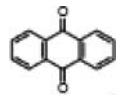
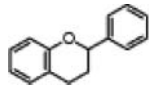
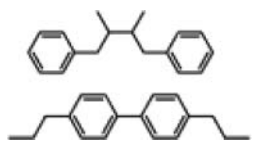
Phenols are secondary plant metabolites contributing to fruit organoleptic and nutritive quality in terms of aroma, taste, flavor, and color. Phenolic compounds include a wide variety of molecules having one or more hydroxyl groups on aromatic ring structure (Lapornik et al., 2005; Ignat et al., 2011). Table 1 shows the main families of phenolic compounds. Their structure ranges from simple molecules to that of high molecular mass polymer often referred as polyphenols (Balasundram et al., 2006). The term "polyphenols" includes more than 8000 compounds, but this number is continually increasing (Boudet, 2007). A possible classification of phenolic compounds is based on the number of phenol rings and the structural elements that bind those rings to one another (Haminiuk et al., 2012). In this respects, the main classes are flavonoids, phenolic acid, tannins, stilbenes, and lignans (D'Archivio et al., 2007).

Apples, grapes, and citrus fruits are a well-known source of polyphenols. Apple pomace contains polyphenols predominantly localized in the peels. Only a minor extent is extracted into the juice during processing. Among polyphenols, apple pomace is a rich source of catechins, hydroxycinnamates, phloretin glycosides, quercetin glycosides, and procyanidins (Djilas et al., 2009). One of the most interesting properties of this material is represented by its antioxidant capacity. Lu and Yeap Foo (2000) determined DPPH and superoxide ion radical scavenging activities of polyphenols in apple pomace, and also in the β-carotene/linoleic acid system. The polyphenols examined were epicatechin, its dimer (procyanidin B2), trimer, tetramer, and oligomer, quercetin glycosides, chlorogenic acid, phloridzin, and 3-hydroxy-phloridzin.

As concern grape pomace, the seeds have been widely appreciated because of their content in phenolic compounds, such as gallic acid, catechins, and epicatechin, together with a wide variety of procyanidins. Iacopini et al. (2008) evaluated the extracts obtained from skin and seeds of 10 native Tuscan and international *Vitis vinifera* varieties, quantifying the content of five phenolic constituents of biological interest: catechin and epicatechin in seeds and quercetin, rutin, and resveratrol in skin extracts.

Phenolic compounds have been also widely detected in waste derived from citrus processing. The peels, in particular, are an abundant source of natural flavonoids. The content in phenolics of the peel of lemons, oranges, and grapefruits is about 15% higher compared to the edible portions (Gorinstein et al., 2001). Furthermore, citrus peels contain a high amount of flavonoids belonging to six peculiar classes according to their

Table 1. Main phenolic compounds families Gracia-Salas, P., Morales-Soto, A., Segura-Carretero, A. and Fernandez-Guierrez, A. (2010) Phenolic-Compound-Extraction Systems for fruit and vegetable samples. Molecules, 15:8813–8826.

Carbon numbers	Class	Basic structure
C_6	Simple phenols	
	Benzoquinones	
C_6-C_1	Benzoic acid	
C_6-C_2	Acetophenones	
	Phenylacetic acid	
C_6-C_3	Cinnamic acid	
	Phenylpropene	
	Coumarins	
	Chromones	
C_6-C_4	Naphthoquinones	
C_6-C_1-C_6	Xanthones	
C_6-C_2-C_6	Stilbenzenes	
	Anthraquinones	
C_6-C_3-C_6	Flavonoids	
$(C_6$-$C_3)_2$	Lignans, neolignans	
$(C_6$-$C_1)_n$	Hydrolysable tannins	Heterogenous polymer composed of phenolic acids and simple sugars
$(C_6$-$C_3)_n$	Lignins	Highly crosslinked aromatic polymer

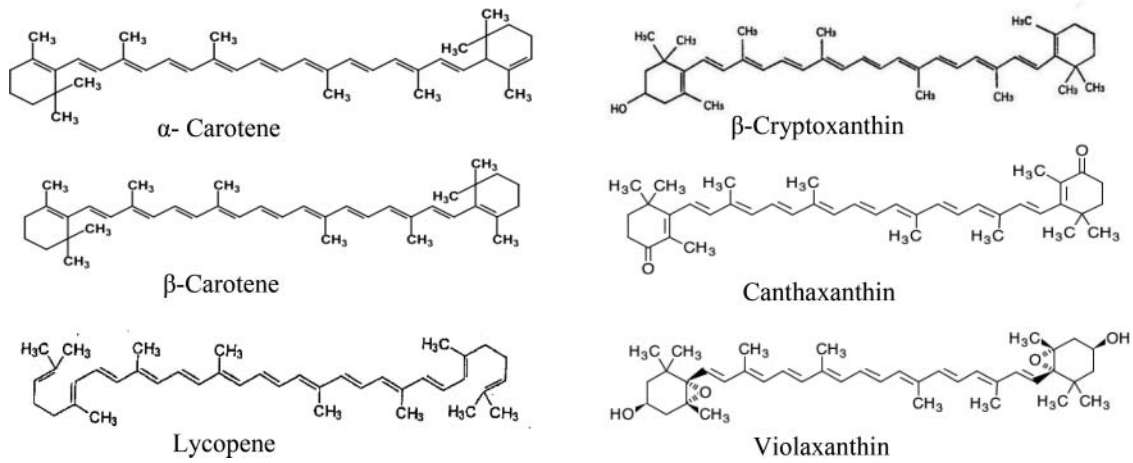


Figure 1. Example of some carotenoids.

structure: flavones; flavanones; flavonols; isoflavones; anthocyanidins; and flavanols.

Carotenoids

Carotenoids comprise a number of naturally occurring yellow, orange, or red compounds notable for their structural diversity, wide distribution, and multiple functions (Fig. 1). They are generally C_{40} tetraterpenoids formed from eight C_5 isoprenoid units joined head-to-tail, except at the center where a tail-to-tail linkage reverses the order, resulting in a symmetrical structure. In nature, carotenoids exist primarily in the more stable all-*trans* (−E) form, although small amount of *cis* (−Z) isomers are also observed. Around 800 carotenoids have been identified in fruits and vegetables. They are split into two classes: the unsaturated hydrocarbon are known as carotenes (e.g., *β*-carotene, lycopene) whereas the oxygenated derivatives (contain hydroxyl, keto, epoxy, and aldehyde substituents) are known as xynthophylls (e.g., *β*-cryptoxanthin, canthaxanthin, violaxanthin). In fruits xanthophyll are in higher proportion (Carbonell-Capella et al., 2014). In general, bioaccesibility of carotenoids is low. However, in some fruits like mango and papaya, they are present in oil droplet in an esterified form with fatty acids. Such structure enhances their extraction and, thus, bioavailability, during digestion (Courraud et al., 2013). Immature citrus peels also show a carotenoid profile characteristic of chloroplastic-containing tissue. Lutein is the main carotenoid. A noticeable decrease in carotenes and lutein occurs at the onset of fruit coloration, with a parallel accumulation of specific xanthophylls. The predominant carotenoid in the peel of mature sweet orange fruit is the *β*, *β*-violaxanthin. The characteristic color of some colored varieties of citrus fruits (sweet orange and mandarins) is provided not solely by *β*, *β*-xanthophylls (violaxanthin and cryptoxanthin) but also by citrus-specific C30-apocarotenoids, *β*-citraurin, *β*-citraurinene, and *β*-apo-8′-carotenal (Rodrigo et al., 2004).

Tocopherols

Tocopherols are important lipid soluble bioactive compounds of fruits and vegetables, characterized by a strong antioxidant activity (Fig. 2). Tocopherols are formed by a 6-chromanol ring structure methylated to varying degrees at C_5, C_7, and C_8 positions and a saturated C_{16} side chain at C_2 position. Different isomers are due to the number and positions of methyl groups on the 6-chromanol ring. Vitamin E is a general term applied for tocopherol and tocotrienols, consisting of four tocopherols, that is, *α*, *β*, *γ*, and *δ*, and four corresponding to tocotrienols (Pertuzatti et al., 2015). Tocopherols are important antioxidants having beneficial effect to inhibit tumor development and to protect membrane fatty acids from fee radical action. Oil extracted from grape seeds has been demonstrated as a valuable source of linoleic acid having also a natural source of vitamin E that provides for considerable

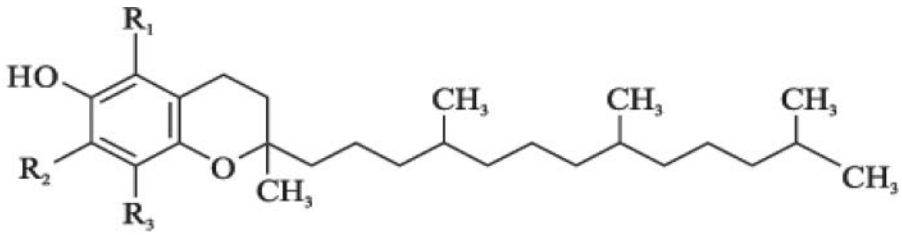


Figure 2. Generic structure of tocopherols.

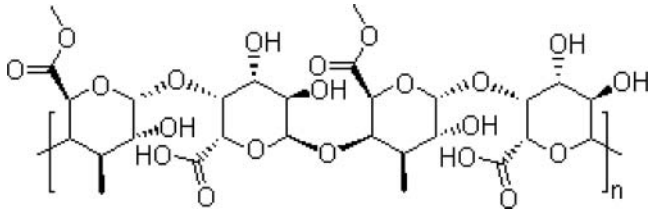


Figure 3. Schematic of pectin structure.

oxidative stability of the extracts. The study of Beveridge et al. (2005) showed that grape seed oil is richer in α-, β-, and γ-tocopherols and α-, and γ-tocotrienols, with γ-tocotrienol being most important quantitatively. Citrus seeds' extracts are a valuable source of terpenoids. However, the meal remaining from the extraction is also shown to be a good source of proteins (Gorinstein et al., 2001).

Dietary fibers

Fruit wastes are also an important source of dietary fibers, commonly known as non-starch polysaccharides. The building blocks of these polysaccharides are glucose, galactose, mannose, arabinose, xylose, rhamnose, fructose, glucuronic acids, galaturonic acids, acetic acid, etc. Dietary fibers consist of a number of heterogenous chemical compound carries a significant amount of polyphenols, carotenoids, and other substances contribute to positive health benefits (Ayala-Zavala et al., 2011). Depending on the extraction process, different dietary fibers show diverse nutritional and functional properties. Dietary fibers can be classified mainly in two groups such as water-soluble, for example, pectin, gums, and mucilages, and water-insoluble, namely, cellulose, hemicellulose, and lignin (Dhingra et al., 2012). Soluble dietary fibers diminish intestinal absorption of blood cholesterol whereas insoluble dietary fiber associates with water absorption and intestinal regulation apart from the well-known probiotic and health benefits (Zhu et al., 2015). Dietary fibers also have some important functional properties in food preparation such as water-holding capacity, increasing viscosity, swelling capacity, and gel formation. For example, pectin (Fig. 3) is one of the most important dietary fibers with an excellent water holding, flavor binding, and gelling properties (Liew et al., 2015).

Apple pomace consists of 10–15% of pectin on a dry weight basis with superior gelling properties compared to citrus pectin but with a brown hue that may limit its incorporation into light-color foods. The most important source of soluble dietary fibers is the wastes derived from citrus processing. These compounds are not only present in high amount in citrus peels but have also important features due to the presence of associated bioactive compounds (flavonoids and vitamin C) with antioxidant properties, which may provide additional health-promoting effects. It has been demonstrated that pectin in the citrus waste contributed to the total amount of chemical fibers in a percentages ranging from 25 to 30% (Marín et al., 2007).

Process technologies

In the subsequent sections, the most innovative and environment-friendly technologies employed for the extraction and recovery of bioactive compounds from fruit wastes are reported. The pros and cons of each technique are presented, widely discussed, and, where possible, compared to the conventional solvent extraction methods.

Pressurized solvent extraction

Pressurized solvent extraction involves the use of solvents at high pressure and with temperature above their boiling points. The high temperatures (between 100 and 374°C) can positively affect the mass transfer rate, the surface equilibria, and the extraction rate thanks to an increasing capacity of the solvent to solubilize solutes, a higher diffusion rate, a better disruption of solute-matrix bonds, a lower surface tension, and a lower viscosity of the solvent. The high pressure, usually ranging from 4 to 20 MPa prevents the evaporation of the solvent and also affects the mass transfer of the solvent into the pores matrices and, thus, the analyte solubility.

The technology is also known as pressurized solvent extraction, subcritical solvent extraction, or accelerated solvent extraction. When 100% water is used as a solvent, the process is generally called superheated water extraction, subcritical water extraction, or pressurized hot water extraction. This is the most common solvent applied in this technology. Under subcritical conditions, water has high diffusivity, low viscosity, and surface tension. This improves the mass transfer kinetics and solutes solubility. At standard pressure and temperature, water is a polar compound with dielectric constant of 80, but as temperature increases, this value decreases and water acts like non-polar compounds. Thanks to these interesting properties, subcritical water extraction is receiving high resonance as excellent alternative medium for extracting non-polar substances thanks to its temperature-dependent selectivity, environmental, non-flammable, and non-toxic acceptability, efficiency, and low cost. Accordingly, subcritical water extraction is receiving high commercial interest as alternative extraction method to obtain bioactive compounds from wastes of fruit processing.

In Table 2, the main findings published so far on the recovery of bioactive compounds with pressurized liquid are listed. One of the first application was published by Wijngaard and Brunton in 2009 applying the technology to extract bioactive (antioxidants and polyphenols) from apple pomace as alternative to conventional techniques. A response surface methodology was applied to select the best temperature ranges and ethanol and water concentrations for enhancing the extraction of antioxidant and polyphenol. Maximum antioxidant activity was obtained at a temperature of 200°C, but unwanted compounds such as hydroxymethylfurfural were formed. Therefore, a lower temperature range between 75 and 125°C was recommended achieving the maximum antioxidant activity at 10.3 MPa and 102°C with 60% ethanol. This result was 2.4 times higher than the one obtained with the traditional solid–liquid extraction. The achievements of this study were so promising that the authors defined the technology as a green extraction technique able to enhance the antioxidant activity or polyphenol levels of a potential food ingredient.

A similar study was performed at 10.3 MPa using 80% aqueous ethanol as the solvent and with the addition of acidified organic acids (formic, acetic, citric, and tartaric acid at a pH of

Table 2. Summary of the technologies applied for the recovery of valuable components from different fruit wastes.

Compound	Source	Applied technology	Recovery yield	Reference
Bioactive compounds	Orange press liquor	Ultrafiltration	Total soluble solids (—)	Ruby Figueroa et al. (2011)
Phenolic compounds	Pigmented orange peels	Nanofiltration	Total anthocyanins (89.2%) Flavonoids (70%)	Conidi et al. (2012)
Phenolic compounds	Fermented grape pomace	Ultrafiltration Nanofiltration	Total phenolic content (—)	Díaz-Reinoso et al. (2009) Díaz-Reinoso et al. (2010)
Phenolic compounds	Winery sludge from red grapes	Ultrafiltration	Total phenolic content (>60%) Sugars (>60%)	Galanakis et al. (2013)
Phenolic compounds	Bergamot juice	Ultrafiltration Nanofiltration	Polyphenols (—) Sugars (—)	Conidi et al. (2011)
Phenolic compounds	Grape seeds	Ultrafiltration	Polyphenols (11.4%)	Nawaz et al. (2006)
Phenolic compounds	Grape seeds	Microwave	Total phenolic content (13.5%)	Hong et al. (2001)
Flavonoids	Grape skins	Microwave	Total anthocyanins (118%)	Liazid et al. (2011)
Phenolic compounds	Mandarin pomace	Microwave	Total phenolic content (99%)	Hayat et al. (2009, 2010a, 2010b)
Phenolic compounds	Grape seeds	Microwave	Total phenolic content (92%)	Li et al. (2011)
Phenolic compounds	Sea buckthorn berries pomace	Microwave	Total phenolic content (—)	Périno-Issartier et al. (2011)
Dietary fibers	*Citrullus lantanus* fruit ring	Microwave	Pectin (25.79%)	Maran et al. (2014)
Dietary fibers	Navel orange peels	Microwave	Pectin (18.13%)	Guo et al. (2012)
Dietary fibers	Apple pomace	Microwave	Pectin (31.5%)	Wang et al. (2007)
Dietary fibers	Orange peels	Microwave	Pectin (19.24%)	Maran et al. (2013)
Bioflavonoids	Penggan peels	Ultrasound	Hesperidin (50%)	Ma et al. (2008)
Polysaccharides	Litchi seeds	Ultrasound	Arabinose (5%) Fructose (38%) Galactose (24%) Glucose (19%) Mannose (10%)	Chen et al. (2011)
Phenolic compounds	Apple pomace	Ultrasound	Flavan-3-ols (30%) Procyanidins (27%) Dihydrochalcones (18%) Phenolic acids (5%) Flavonols (11%)	Virot et al. (2010)
Phenolic compounds	Orange peel	Ultrasound	Flavanone glycosides (11%)	Khan et al. (2010)
Phenolic compounds	Apple pomace	Ultrasound	Total phenolic content (50%)	Pingret et al. (2012)
Carotenoids	Citrus peel	Ultrasound	All-*trans*-β-carotene (8%)	Sun et al. (2011)
Phenolic compounds	Coconut shell powder	Ultrasound	Total phenolic content (2.2%)	Rodrigues et al. (2008)
Phenolic compounds	Longan fruit pericarp	Ultrasound	Total phenolic content (14%)	Prasad et al. (2010)
Phenolic compounds	Litchi fruit pericarp	Ultrasound	Total phenolic content (24%)	Prasad et al. (2009a, 2009b, 2009c)
Flavonoids Essential oils	Grape by-products	Ultrasound	Anthocyanin content (7%) Oils (>90%)	Corrales et al. (2008)
Phenolic compounds	Citrus peels	Ultrasound	Total phenolic content (80%)	Omar et al. (2013)
Dietary fibers	Citrus *junos* peel	Subcritical water	Pectin (78%)	Tanaka et al. (2012)
Dietary fibers	Flavedo citrus *junos*	Subcritical water	Pectin (80%)	Ueno et al. (2008)
Dietary fibers	Apple pomace Citrus peel	Subcritical water	Pectin (16.68%) Pectin (21.95%)	Wang et al. (2014)
Phenolic compounds	Winery waste	Subcritical water	Total polyphenols (32%) Total flavonoids (15%)	Aliakbarian et al. (2012)
Phenolic compounds	Winery by-products	Subcritical water	Gallic acid (70%)	García-Marino et al. (2006)
Flavanones	*Citrus unshiu* peel	Subcritical water	Hesperidin (72%) Narirutin (12%)	Cheigh et al. (2012)
Phenolic compounds	Pomegranate seeds	Subcritical water	Total phenolic content (—)	He et al. (2012a, 2012b)
Phenolic compounds	Red grape skin	Subcritical water	Total anthocyanin content (45%)	Ju and Howard (2005)
Phenolic compounds	Longan fruit pericarp	High hydrostatic pressure	Total phenolic content (18%)	Prasad et al. (2009a, 2009b, 2009c)
Flavonoids	Red grape skins	High hydrostatic pressure	Total anthocyanin content (30%)	Corrales et al. (2009)
Phenolic compounds	Litchi fruit pericarp	High hydrostatic pressure	Total phenolic content (30%)	Prasad et al. (2009a, 2009b, 2009c)
Flavonoids	Grape by-products	High hydrostatic pressure	Total anthocyanin content (14%)	Corrales et al. (2008)
Phenolic compounds	Grape skins	Pulsed electric fields	Total polyphenol content (20%)	Boussetta et al. (2009)
Flavonoids	Grape by-products	Pulsed electric fields	Total anthocyanin content (20%)	Corrales et al. (2008)
Phenolic compounds	Pomegranate peels	Pressurized water	Total phenolic content (81.7%)	Çam and Hışıl (2010)
Flavonoids	Red grape pomace	Pressurized solvent	Total anthocyanin content (45%)	Monrad et al. (2010a)
Phenolic compounds	Apple pomace	Pressurized solvent	Total phenolic content (—)	Wijngaard and Brunton (2009)
Phenolic compounds	Blackberry pomace	Pressurized solvent	Total phenolic content (6.33%)	Paula et al. (2014)
Flavonoids	Grape pomace	Pressurized solvent	Total anthocyanin content (50%)	Srinivas et al. (2011)
Phenolic compounds	Cherry pomace	Pressurized solvent	Total phenolic content (—)	Adil et al. (2008)
Volatiles	Cantaloupe seeds	Supercritical fluid extraction	Essential oils (—)	Ismail et al. (2010)
Flavonoids	Orange pomace	Supercritical fluid extraction	Limonene (1.16%) Palmitic acid (26%) Oleic acid (88%)	Benelli et al. (2010)
Phenolic compounds	Apple peels	Supercritical fluid extraction	Glycosides-derivatives (50%) Quercetin glycosides (78%)	Massias et al. (2015)
Flavones	*Citrus depressa* Hayata peels	Supercritical fluid extraction	Nobiletin and tangeretin (107%)	Lee et al. (2010)
Essential oils	Apricot kernel	Supercritical fluid extraction	Oil (32%)	Özkal et al. (2005a)

(*continued on next page*)

Table 2. (*Continued*)

Compound	Source	Applied technology	Recovery yield	Reference
Phenolic compounds	Cherry cull	Supercritical fluid extraction	Total phenolic content (28.8%)	Serra et al. (2010)
Essential oils	Peach almond	Supercritical fluid extraction	Almond oil (24%)	Mezzomo et al. (2009)
Essential oils	Peach seeds	Supercritical fluid extraction	Seed oil (70%)	Sánchez-Vicente et al. (2009)
Essential oils	Pomegranate seeds	Supercritical fluid extraction	Seed oil (16%)	Liu et al. (2009a, 2009b, 2009c)
Essential oils	Opuntia dillenii seeds	Supercritical fluid extraction	Seed oil (6.65%)	Liu et al., 2009a, 2009b, 2009c
Carotenoids	Apricot bagasse	Supercritical fluid extraction	β-carotene (6%)	Döker et al. (2004)
Carotenoids	Apricot pomace	Supercritical fluid extraction	β-carotene (0.8%)	Şanal et al. (2004)
Aroma compounds	Grape skins	Supercritical fluid extraction	Glycosides (100%)	Palma et al. (2000)
Essential oils	Grape seeds	Supercritical fluid extraction	Seed oil (11.5%)	Passos et al. (2010)
Flavonoids	Pomelo peels	Supercritical fluid extraction	Total flavonoids compounds (2.37%)	He et al. (2012a, 2012b)
Essential oils	Citrus sphaerocarpa Tanaka peel	Supercritical fluid extraction	Oils (1.55%)	Suetsugu et al. (2013)
Flavanones	Grapefruit seeds	Supercritical fluid extraction	Limonoids (0.6%) Naringin (0.2%)	Yu et al. (2007)
Flavonoids	Citrus unshiu peels	Supercritical fluid extraction	Terpenes compounds (2.5%)	Lee et al. (2001)
Phenolic compounds	Apple and peach pomace	Supercritical fluid extraction	Total phenolic content (—)	Adil et al. (2007)
Phenolic compounds	Grape seeds	Supercritical fluid extraction	Total phenolic content (—)	Murga et al. (2000)
Essential oils	Passiflora seeds	Supercritical fluid extraction	Seed oil (25.83%)	Liu et al. (2009a, 2009b, 2009c)
Essential oils	Peach seeds	Supercritical fluid extraction	Seed oil (35%) β-sitosterol	Ekinci and Gürü (2014)
Essential oils	*Hippophae rhamnoides* seeds	Supercritical fluid extraction	Seed oil (>90%)	Yin et al. (2005)
Phenolic compounds	Blueberry, cranberry, and raspberry pomaces	Supercritical fluid extraction	Raspberry solubles yield (5.20%) Cranberry solubles yield (3.89%) Blueberry solubles yield (1.4%)	Laroze et al. (2010)
Essential oils	Cherry seeds	Supercritical fluid extraction	Seed oil (8%)	Oneto et al. (2001)
Carotenoids	Seabuckthorn seeds	Supercritical fluid extraction	Tocopherol (77.2%) Carotene (75.5%)	Kagliwal et al. (2011)
Bioactive compounds	Banana peels	Supercritical fluid extraction	Total extracted solids (6.5%)	Comim et al. (2010)
Phenolic compounds	Cherry pomace	Subcritical fluid extraction	Total phenolic content (—)	Adil et al. (2008)
Essential oils Phenolic compounds	Citrus peel	Supercritical fluid extraction	Oil (90%) Total phenolic content (50%)	Omar et al. (2013)

2.5) to recover flavonoids from freeze-dried Sunbelt (*Vitis labrusca* L.) grape pomace (Srinivas et al., 2011). A response surface methodology identified an optimal temperature of 85.4°C to extract the maximum amount of anthocyanins from grape pomace and 124°C for flavonoids. The results also showed that the addition of organic acid to the process did not significantly influence the extraction yield. The authors stated that the promising results obtained on an analytical scale have the potential to push the technology to be industrially scaled-up and designed.

To test the effect of ethanol/ water ratio on the extraction efficiency, a dried red grape pomace was treated for the recovery of anthocyanins using subcritical solvents with 10, 30, 50, and 70% ethanol in water (v/v). During the treatment, the pressure was kept constant at 6.8 MPa while the temperature was ranged from 40 to 140°C. Solvents containing 70 and 50% ethanol in water extracted more total anthocyanins (463 and 455 mg/100 g of dry matter, respectively) than conventional solvent extraction (methanol/ water/formic acid (60:37:3 v/v/v)). The total amounts of anthocyanins extracted at 100°C (450 mg/100 g of dry matter), 80°C (436 mg/100 g of dry matter), and 120°C (411 mg/100 g of dry matter) were higher than at the other temperatures. Solvents containing 70 and 50% ethanol in water extracted similar amounts of anthocyanins indicating that ethanol/water ratio was not a key process parameter for ratio up to 50% (Monrad et al., 2010a, 2010b).

As concern published results on the valorization of food wastes using water as pressurized liquid, the technology has been successfully applied to the waste derived from the production of citrus juice, such as peel (Cheigh et al., 2012; Tanaka et al., 2012; Wang et al., 2014) and flavedo (Ueno et al., 2008) to extract dietary fibers.

From citrus *junos* peel, Tanaka et al. (2012) processed the residues obtained from SFE extraction to separate pectin and hemicellulose with subcritical water. They used a semi-continuous apparatus working in the temperature range of 160–320°C, and water flow rates of 2.1, 3.5, and 7.0 mL/min under a pressure of 20 MPa. About 78% of the pectin was recovered from the fraction processed at 160°C while most of the cellulose (about 80%) was separated from hemicellulose performing the process at 200°C. The characteristics of the recovered cellulose were also analyzed using scanning electron microscopy (SEM), attenuated total reflectance Fourier transmission infrared (ATR-FTIR), and thermogravimetric-differential thermal analyses (TG-DTA) reporting the high crystallinity and low impurity of the material extracted with subcritical water. With the same apparatus, pectin was extracted from the flavedo of citrus *junos* at the optimal temperature of 160°C reaching a yield of about 80% (Ueno et al., 2008).

Subcritical water was also used to extract pectin from both citrus peel and apple pomace achieving maximum yields of 22% and 17%, respectively. The yield was significantly affected by the extraction temperature and composition of the raw material. The analyzed extracted pectin showed a high antioxidative and anti-tumor activity although no information was given about the mechanism involved in their antioxidant and anti-tumor bioactivities (Wang et al., 2014).

Interesting results were published recovering hesperidin and narirutin flavanones from citrus *unshiu* reaching maximum yields of hesperidin (72 ± 5 mg/g) and narirutin (11.7 ±

0.8 mg/g) performing the subcritical water process at 160°C, 10 MPa for only 10 min. These yields accounted for approximately 99% of the total amount of these flavanones in the original material. To assess the potential of the technology, conventional extraction methods were compared with subcritical water in terms of yields. The hesperidin yield by subcritical water was more than 1.9, 3.2, and 34.2-fold higher than those obtained by the extraction methods using ethanol (70%), methanol (70%), or hot water, respectively. The narirutin yield was more than 1.2, 1.5, and 3.7-fold higher (Cheigh et al., 2012).

The technology has been also successfully applied for the recovery of phenolic compounds from winery waste (Ju and Howard, 2005; García-Marino et al., 2006; Aliakbarian et al., 2012). In the study of Aliakbarian et al. (2012), the process parameters were optimized (140°C and 11.6 MPa) achieving 31.69 mg of gallic acid equivalent (GAE) per gram and 15.28 mg_{CE}/g of total polyphenols and flavonoids, respectively. Subcritical water extraction process was also compared to conventional extraction with organic solvent and aqueous-based system at atmospheric pressure resulting more efficient for recovery of antioxidants (Aliakbarian et al., 2012). Winery wastes, in particular grape seeds, were treated to recover catechins and proanthocyanidins by subcritical water process in different assays. The results showed that subcritical water achieved higher recoveries when the material was submitted to three sequential extractions at 50, 100, and 150°C; although selective extractions of compounds with different degrees of polymerization were achieved using one-step extraction at different temperatures. As an example, better recoveries for flavanol dimers and trimers, showing higher antioxidant activity, were obtained using a single extraction at 150°C. In addition, gallic acid, with antioxidant characteristics similar to that of catechin and epicatechin monomers, was obtained in greater quantities by a single extraction at 150°C (García-Marino et al., 2006).

Excellent results in terms of extraction of phenolics were achieved applying subcritical water process on dried red grape skin (Ju and Howard, 2005). The obtained values demonstrated that subcritical water extracts had comparable or higher levels of anthocyanins and antioxidant capacities values than extracts obtained using conventional hot water or 60% methanol. Subcritical water at 100 to 110°C appears to be an excellent alternative to organic solvents to extract anthocyanins and other phenolics from dried red grape skin and possibly other grape processing byproducts.

He et al. (2012a, 2012b) showed subcritical water extraction potential in the recovery of phenolic compounds from pomegranate seeds performing the process at 6 MPa, 220°C, 1:40 solid to water ratio for 30 min and obtaining the highest total phenolic compounds content (4854.7 mg/100 g). Also in this study, a comparison was carried out between subcritical water and methanol or ethanol conventional leaching. Total phenolic content and antioxidant capacities, obtained with subcritical water at 160°C, achieved similar results when methanol or acetone leaching were used. In particular, the extraction time was significantly reduced, changing from 2 h with the conventional leaching to 30 min with the subcritical water extraction. Furthermore, one should also consider that the environmental friendly criteria of subcritical water are much higher than that of organic solvents extraction. On the same fruit, pressurized water was also applied for the extraction of polyphenols from peels (Çam and Hışıl, 2010). The process was optimized in terms of particle size, temperature, and time. Such factors affected significantly the extraction yield. Extraction of polyphenols from pomegranate peels could be achieved effectively by applying the following combinations: temperature of 40°C, time of 5 min, and particle as small as possible, but not smaller than 65 μm. The resulting extraction yield was comparable with the one achieved with the conventional methanol extraction, but with the advantages that the processing times were lower. In addition, no final purification step was necessary.

Supercritical fluid extraction

Supercritical fluid extraction (SFE) is based on the use of a fluid at pressures and temperatures beyond its critical point, in order to achieve significant physical changes that will modify its capabilities as solvent. Although the first experimental works dealing with supercritical fluid extraction started back to the 19th century, the interest of this technique as a potential alternative to conventional solvent-based extraction techniques increased only recently. The most widely used solvent to perform SFE has been carbon dioxide (CO_2) thanks to its great versatility. In supercritical phase (temperature of 31.1°C and pressure of 7.4 MPa), CO_2 properties can be tuned in order to provide extracts with desirable compositions (selectivity enhancements). At the same time, it ensures an innocuous separation process both to human health and to the environment, with no degradation of heat sensitive compounds and the absence of toxic solvents residue in the solutes after the process. In addition to its physical characteristics, CO_2 is safe, food grade, and widely available at a relatively low cost and high purity. According to Wijngaard et al. (2012), SFE extraction techniques are gaining popularity due to its ability to increase target molecule specificity and reduce waste solvent production. However, SFE has some characteristics that limit its application. As it is mainly suitable to extract nonpolar substances, thus, SFE has low capacity to recovery compounds from wastes rich in water. To overcome this limitation, a co-solvent is usually added such as water or ethanol. The effect of the co-solvent is to expand the range of polar compounds and intermediate polarity that can be extracted.

Table 2 reports most of the studies published on the SFE applied to fruit wastes. The technology was mainly applied to recover phenolic compounds and fatty acids. The implicit goal of these works was the search for substances with some biological activity to be used in therapeutic medicaments, cosmetics, or food ingredients. Fatty acids from fruit wastes have been recovered by SFE at temperatures between 40 and 80°C and pressures from 10 and 60 MPa. The main reasons to use these parameter ranges are the thermal protection of thermally sensitive compounds and their solubility in CO_2. Low temperatures (just above the critical point of CO_2) and high pressures increase CO_2 density and consequently the solubility of nonpolar compounds in CO_2. Interesting compounds have been extracted from kernel cantaloupe (Ismail et al., 2010) and apricot (Özkal et al., 2005a), peach almond (Mezzomo et al., 2009; Sánchez-Vicente et al., 2009), pomegranate seeds (Liu et al.,

2009a, 2009b, 2009c), *Opuntia dillenii Haw.* seeds (Liu et al., 2009a, 2009b, 2009c), grape seeds (Passos et al., 2009, 2010), passiflora seeds (Liu et al., 2009a, 2009b, 2009c), and Kabosu (*Citrus sphaerocarpa* Tanaka) peels (Suetsugu et al., 2013). In most of these studies, SFE optimal process condition was identified in terms of pressure, temperature, particle size, solvent flow rate and co-solvent concentration to get extracts with the highest yields and with the required physical and chemical properties. Liu et al. (2009a, 2009b, 2009c) applied the response surface methodology to optimize SFE process parameters for the recovery of oil from the passion fruit seeds. The optimal process parameters were: temperature of 56°C, pressure of 26 MPa, and extraction time of 4 h. Under these conditions, the oil yield was 25.8% and the product showed a golden orange color with physical and chemical properties up to the required standard for edible oil (content of unsaturated fatty acid up to 89.4% and linoleic acid over 72%). A similar study was performed on pomegranate seeds where SFE process parameters were optimized and maximum oil yield was predicted with response surface methodology to be 156.3 g/kg dry basis at 37.9 MPa, 47°C, and 21.3 L/h of CO_2 flow rate. In addition, the authors compared the fatty acid composition and the tocopherols' content of SFE pomegranate seed oil with those obtained by the Soxhlet method. Minor differences were found in fatty acid composition of the oils extracted with the two methods while the content of total tocopherols was about 14% higher in SFE extracts (Liu et al., 2009a, 2009b, 2009c).

SFE achieved an oil extraction yield of 6.65% from *Opuntia dillenii Haw*. seeds at 47 MPa, 47°C, 2.79 h, and a CO_2 flow rate of 10 kg/h. The chemical composition of the seed oil measured by GC–MS reported that the main fatty acids were linolenic acid (66.6%), palmitic acid (19.8%), stearic acid (9.0%), and linoleic acid (2.7%). The antioxidant activity of seed oil was assessed by means of 2,2-diphenyl-1-pic-rylhydrazyl (DPPH) radical-scavenging assay and β-carotene bleaching test. Both methods showed results comparable to the references ascorbic acid and butylated hydroxytoluene (BHT) (Liu et al., 2009a, 2009b, 2009c).

Two papers demonstrated the feasibility of the process to extract peach oil with a yield up to 24% depending on the processing parameters (Mezzomo et al., 2009; Sánchez-Vicente et al., 2009). In one of the two studies, it was demonstrated that SFE (with ethanol as co-solvent) was able to reach extraction yield 70% higher than the Soxhlet extraction in liquid hexane. Furthermore, GC and HPLC analyses revealed no differences in fatty acids and tocopherols content between the two extracts (Sánchez-Vicente et al., 2009). The second study focused on the possibility to scale up SFE process parameters such as extraction pressure, CO_2 flow rate, and particle size. The kinetic study revealed that these parameters influenced the mass transfer rate, constant extraction yield period, and global yield, and that the process was mainly driven by the convection as the dominant mass transfer mechanism, while the diffusion was the limiting factor (Mezzomo et al., 2009).

An interesting study was performed on apricot kernel for the extraction of oil where, besides the investigation of the effects of particle size, solvent flow rate, extraction pressure and temperature, and co-solvent concentration on oil yield, the mass transfer coefficients were also determined applying the model of broken and intact cells. Two extraction periods were distinguished: in the first one, the oil was easily extracted from the surface with a fast extraction. In the second one, the oil was slowly extracted from the intact oil cells of kernel structure. For both periods, the authors were able to evaluate the mass transfer coefficients changing between 0.7 and 3.7 min^{-1}, and between 0.00009 and 0.0005 min^{-1}, respectively (Özkal et al., 2005a). In addition, the same authors published a second paper in which the response surface methodology was applied to determine the effects of solvent flow rate (2, 3, and 4 g/min), pressure (30, 37.5, and 45 MPa), temperature (40, 50, and 60°C), and co-solvent concentration (0, 1.5, and 3 wt % ethanol) on the oil yield. The results indicated that all parameters had a significant effect on oil yield. In detail, the maximum yield value (0.26 g/g kernel) was obtained performing the extraction for 15 min on apricot kernel particles with a diameter of 0.85 mm and with a 4 g/min solvent flow rate containing 3 wt % ethanol at 45 MPa and 60°C. The SFE extracts also showed no differences in fatty acid composition if compared to the one derived from a conventional extraction performed with hexane (Özkal et al., 2005b).

Interesting results were also published in the study of Suetsugu et al. (2013) where oil was extracted from *Citrus sphaerocarpa* Tanaka peel by SFE. A maximum yield of 1.55% (by weight of wet sample) was obtained at 80°C and 20 MPa, which was over 13 times higher than that of the conventional cold-press method. The extracts, analyzed by GC/MS, were comprised of 49 compounds including several non-polar and weakly polar hydrocarbons such as terpenoid, free fatty acid, and coumarin with lower content of monoterpenes and higher content of oxygenated compounds, sesquiterpenes, which strongly contribute to the aromatic characteristics of the extracts.

Compared to the studies cited so far, lower temperatures (from 40 to 50°C) were applied to extract oil from grape seeds at 18, 20, and 22 MPa. The results demonstrated that the extraction rate increased with increasing the pressure and decreasing the temperature, due to the influence of both variables on the oil solubility. The triacylglycerides content and the fatty acids profile of the oil were roughly unaffected by the operating conditions, while the antioxidant activity (assessed by the DPPH• spectrophotometric method) showed to increase with pressure and noticeably with temperature (Passos et al., 2010).

The second class of extracts obtained by SFE is phenolic compounds. The process showed promising results in the recovery of phenolics from apple peels (Massias et al., 2015) and pomace (Adil et al., 2007), cherry pomace (Serra et al., 2010), peach pomace (Adil et al., 2007), and grape seeds (Murga et al., 2000).

Polyphenols were extracted at 57 MPa and 55°C from apple and 51 MPa and 52°C from peach pomace with an extraction time of 40 min and adding 20% ethanol. Total phenolic content of the extracts were 0.47 and 0.26 mg gallic acid equiv./g sample and the antiradical efficiency were 3.30 and 1.5 mg DPPH•/mg sample for apple and peach pomaces, respectively (Adil et al., 2007). More recently, SFE was applied for the recovery of phenolic compounds from apple peels at 25 MPa and 50°C using CO_2 and ethanol (96%) in 75:25 molar ratio. The process was

able to extract all the principal phenolics known to be present in the apple peels, including the polar sugar-based quercetin derivatives and phloridzin. Furthermore, it was observed that the increase of the matrix loading did not increase the extracted amounts in the same ratio, showing a poor efficiency that was related to the fluid saturation or high bed packing. The maximum phenolics yield, achieved in this study, was equal to 120 mg from 15 g of peels with only 1.1 kg of extracting fluid. Making a comparison between SFE and the conventional methanol/acetone/water or ethanol extracts, a higher contribution (in the range of 75–80 wt % instead of 50%) of quercetin derivatives to the phenolic pool was found (Massias et al., 2015).

As observed so far, most of the cited studies involved the use of a co-solvent to extract polyphenol from fruit pomace. In the study of Serra et al. (2010) performed on cherry pomace, it was clearly observed that SFE carried out without a co-solvent brought to low yields. In addition, the extracts showed low phenolic content and antioxidant activity. On the contrary, the extracts obtained with CO_2 and ethanol as co-solvent (90:10, v/v) exhibited the highest antioxidant activity (181.4 ± 23.7 mol TEAC/g).

Similar results were observed in the study of Murga et al. (2000) where it was demonstrated that SFE extraction yield of phenolics and tannins (namely, some low polymerized proanthocyanidins and some low molecular weight phenolic compounds) from grape seeds increased with the pressure and the amount of alcohols (i.e., ethanol and methanol) used as co-solvent.

A study performed on orange pomaces (*Citrus sinensis* L. Osbeck) compared SFE with low pressure methods such as ultrasounds and Soxhlet in terms of process yield, extract composition, and biological activity (Benelli et al., 2010). The low pressure techniques (ultrasounds and Soxhlet) presented the highest process yield when using ethanol and water as solvent. SFE extracts achieved lower yields but better results for the antioxidant activity measured by DPPH assays, total phenolic content, and β-carotene bleaching method. The results also demonstrated that when a co-solvent (ethanol) was used with CO_2, the extraction yields, the antioxidant activity by DPPH assays, and the total phenolic content increased.

Flavonoids have been also demonstrated to be extracted by SFE from different sources such as *Citrus depressa* Hayata peels (Lee et al., 2010), pomelo peels (He et al., 2012a, 2012b), and grapefruit seeds (Yu et al., 2007). The results achieved on pomelo peels treated by SFE showed interesting higher scavenging activity on hydroxyl, 1,1-diphenyl-2-picrylhydrazyl (DPPH) and 2,27prime;-azino-bis(3-ethylbenzthiazoline-6-sulphonic acid) (ABTS) radicals than those obtained by conventional solvent extraction (He et al., 2012a, 2012b).

Highest yields were obtained in the recovery of limonoids (6.3 mg/g seeds) such as limonin and limonin-17-b-D-glucopyranoside, and flavonoid naringin (0.2 mg/g seeds) from grapefruit seeds where SFE was able to extract the low polar compounds and the presence of ethanol as a co-solvent induced the extraction of the high polar compounds (Yu et al., 2007).

In a subsequent study on *Citrus depressa* Hayata peels, it was demonstrated that the yields of extraction of nobiletin and tangeretin were significantly affected by the co-solvent ratio (Lee et al., 2010). Results showed that the yield of nobiletin and tangeretin increased up to 9.1% when water and ethanol (85%) were used as modifier in supercritical carbon dioxide. Overall, the nobiletin and tangeretin yield of SFE was 7% higher than that of the conventional solid–liquid extraction. The purity of SFE extracts (35.3%) was about 7.2 times as high as that obtained by conventional solid–liquid extraction (4.9%).

Only two studies assessed the feasibility of SFE in the extraction of carotenoids from waste of fruit processing. Both dealt with the application of the process for the recovery of β-carotene from freeze-dried and grinded (75–600 μm) apricot pomace but only one reported experimental data assessing SFE extraction ability (Şanal et al., 2004). The second one intended to investigate the effect of the main process parameters such as pressure, temperature, CO_2 flow rate, and particle size on the extraction yields of β-carotene (Döker et al., 2004). The experimental results reported that the highest extraction yield of β-carotene (88 μg/g dry pomace) was achieved when 4 mL of 2,2-dimethoxypropane were added to 1 g of pomace at 40.5 MPa, 55°C, 1 mL CO_2/min flow rate, and performing the process for 90 min. By the comparison of SFE with the traditional solvent extraction (petroleum ether/methanol (1:1, v/v)), the authors pointed out that β-carotene was extracted by SFE in a shorter time (two-fold) with a nontoxic solvent, thus, leading to a cheaper process (31-fold lower than those estimated with the traditional solvent extraction plant).

Only one study showed the possibility to apply SFE for the recovery of glycosides from grape seeds. However, due to the glycoside polarity, the use of methanol as co-solvent was needed (Palma et al., 2000). The authors reported that SFE showed the same reproducibility and the same recovery as the conventional methods but with 25% shorter time.

Membrane separation

Membrane technology is based on a thin physical barrier through which materials can either pass (the permeate) or retained (the retentate) due to a driving force that can be a difference of pressure, concentration, temperature, and/or electrical potential. The separation performance of a membrane is influenced by inherent characteristics, such as its chemical composition, or by process variables such as temperature, pressure, feed flow. In addition, interactions between components in the feed flow and the membrane surface should also be considered (Salehi, 2013). Membrane processes are generally classified according to the dimensions of the molecule to be separated (Arvanitoyannis et al., 2006): microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO). MF is a membrane process that involves the use of membranes with a pore size of 0.2–2 μm that can selectively separate particles with molecular weights higher than 200 kDa. The process involves pressures lower than 0.2 MPa and can separate molecules between 0.025 and 10 μm. Thanks to these characteristics, it is primarily used to separate particles and bacteria from other smaller solutes (Hua et al., 2007). UF process involves the use of membranes with a molecular weight cut off in the range of 1–300 kDa and a pore size of 0.01 μm. UF uses pressures greater than 1 MPa. It is used to separate colloids like proteins from small molecules like sugars and salts. As concern NF

membranes, they have pores in the range of 0.5–1 nm and are able to concentrate, fractionate, or purify aqueous solutions of organic solutes with molecular weight between 100 and 1000 Da and mixture of monovalent/multivalent salts working with pressures between 1 and 4 MPa (Salehi, 2013). RO membranes are characterized by a molecular weight cut off about 100 Da with pressures 5–10 times higher than those used in UF (between 4 and 10 MPa) and concentrate particles with molar masses below 350 Da.

Pressure, temperature, viscosity, density of the feed fluid, and the tangential velocity are the main physical operational parameters affecting the permeate flow rate. The utilization of membrane technology for the purification and concentration of bioactive compounds from wastes of fruit processing has been successfully employed with possible applications in food colorants, food supplements, pharmaceutical applications, and cosmetic products (Crespo and Brazinha, 2010; Conidi et al., 2014). In particular, several advantages may be considered comparing membrane technology to the others in terms of absence of phase transition, mild process conditions, low energy requirements, separation efficiency, and easy scale up (Castro-mu et al., 2015). However, it is necessary to state that one constrain of the technology is the membranes fouling, which implies a lack of long durability of the membrane during the process.

Table 2 reports the main results of the technology applied for the recovery of bioactive compounds from fruit wastes. Recently, research papers have been published demonstrating UF efficiency for recovering from macromolecules to micromolecules with low molecular weight from different food wastewaters such as dietary fibers, proteins, sugars, phenolic compounds, and antioxidants components but also NF efficiency to recover, fractionate, and concentrate different kind of bioactive compounds with low molecular weight (Castro-Munoz et al., 2015).

Galanakis et al. (2013) developed an UF-based process for the recovery and fractionation of different phenolic compounds from winery sludge derived from red grapes. Three membranes types (1, 20, and 100 kDa) were employed showing different results in terms of compounds recovery: the 20 kDa membrane retained high percentage (60%) of polar solutes (phenolic and sugars), the 100 kDa membrane allowed the separation of polar solutes from pectin and hydrolyzed derivate, the 1 kDa shows a higher selectivity successfully separating hydroxycinnamic acid derivate from anthocyanins and flavonols in the diluted and concentrated extract, respectively.

Nawaz et al. (2006) showed the efficiency of UF technology in concentrating polyphenols extracted from grape seeds using a conventional ethanol–water mixture. With UF concentration step, they demonstrated that the procedure provided high extraction rates, high extraction selectivity, short extraction time, and significant labor savings. The best conditions for the extraction were a solid to liquid ratio of 0.2 g/mL, according to mass transfer calculations and absorbance readings, performing a double extraction with a membrane with a small pore size (0.22 μm). At these conditions, they obtained the maximum recover of polyphenols (11.4% of the total seeds weight) from the grape seed.

Significant results reported NF efficiency in fractionating aqueous extracts from distilled fermented grape pomace using several membranes such as Nanomax 95 membrane (250 Da), Nanomax 50 (350 Da), DL 2540 (150–300 Da), GE 2540 (1000 Da), and the Inside Ceram (1000 Da). Among the above, the last membrane had the higher rejection of total solids (90%) and total phenols (90%) compared to the others (Díaz-Reinoso et al., 2009). The authors also showed that the retentates obtained in the tested membranes were characterized by total soluble solids content ranging from 0.22 to 1.41 g/100 g, total phenolics ranging from 0.17 to 1.09 mg GAE/mL, total sugars ranging from 0.27 to 2.09 mg/mL, and antioxidant activity from 3.42 to 22.5 mmol Trolox. Moreover, the same authors also demonstrated the possibility to use membranes processing as prior step for the recovery of antioxidant compounds (Díaz-Reinoso et al., 2010).

Studies were also published showing the potential of UF technology for anthocyanin fractionation (Kalbasi and Cisneros-Zevallos, 2007) from grape as well as by NF-UF-microfiltration for producing proanthocyanic fractions (Santamaría et al., 2002).

More recently, Conidi et al. (2011) investigated the possibility to separate and concentrate polyphenols in the bergamot juice, in order to develop a natural product enriched in polyphenols suitable for nutraceutical applications. The initial clarification of the depectinized bergamot juice was performed by UF. Subsequently, the clarified juice was submitted to different UF and NF processes in order to evaluate the effect of the process on the rejection of the membranes toward sugars, organic acids, and polyphenols. They demonstrated that the UF step of the depectinized juice produced a clarified juice with physicochemical and nutritional properties similar to those of the treated juice, except for the absence of suspended solids. Furthermore, an NF membrane (450 Da) induced the best separation of polyphenols and sugars with a clear permeate enriched in sugar and organic acids and a retentate rich of phenolic compounds. The results of the study indicated a clear tendency in considering the integrated UF/NF treatment as a source of bioactive compounds, especially in terms of polyphenols, with potential applications for the valorization of the product.

Membrane technology has been also applied for the recovering of antioxidant components from orange press liquors (Ruby Figueroa et al., 2011). In this study, the authors used a response surface methodology to optimize the process parameters of an UF membrane achieving a maximum polyphenol rejection of 28.5% and antioxidant activity of 32.28 mM Trolox at 0.02 MPa, 20°C, and feed flow rate of 254 L/h. An additional research showing the potential of membrane technology was published evaluating the separation and concentration of phenolic compounds from a press liquor obtained from orange peels testing four different spiral wound NF membranes (250, 300, 400, and 1000 Da) (Conidi et al., 2012). The results indicated a reduction of the average rejection toward sugars increasing the nominal molecular weight cut-off of the membranes, and a higher rejection of anthocyanins (80%) and flavonoids (70%).

Microwaves

Microwaves assisted extraction has gained great popularity to recover low molecular weight organic compounds or small

molecules from food matrices (Kaufmann and Christen, 2002). It is a relatively new application able to extract soluble solids from a wide range of materials using microwave energy. It has been recognized as a green technology because it reduces the use of organic solvents. Alupului et al. (2012) described the extraction mechanisms of the process based on three steps: (1) the separation of solutes from the active sites of the sample matrix under increased temperature and pressure; (2) the diffusion of the solvent across sample matrix; and (3) the release of solutes from sample matrix to solvent.

The principle on which the technology has been developed is based on the ability of microwaves—the electromagnetic radiation with a wavelength from 0.001 m to 1 m—to pass through the medium with the adsorption of the energy and its conversion into thermal energy. Due to the heating effect, the moisture inside the cells evaporates producing a high pressure build up. This modifies the physical properties of the biological tissues, improving the porosity of the biological matrix (Zhang et al., 2011). Thanks to this effect, the technology provides a better penetration of the extracting solvent through the matrix and an improved yield of the desired compounds. Compared to the classical heating methods based on the conduction mechanism, microwaves assisted extraction has several advantages like higher heating rate, faster control of the process, higher purity of the final product, shorter extraction time, and lower power needed to run the process (Mosquera et al., 2013).

Microwaves assisted extraction of natural bioactive compounds from fruit wastes can be affected by several factors such as: power, frequency, processing time, sample moisture content and particle size, extraction temperature, pressure and extraction cycles, type of solvent, and ratio of solid sample to liquid solvent (Mandal et al., 2007). Among these factors, the solvent used in the process seems to be the most critical, also considering its features in terms of solubility, dielectric constant, and dissipation factors (the efficiency with which a solvent heat up under microwaves). It has been demonstrated that polar solvents with high dielectric constant as water are preferred to nonpolar solvents (Wang and Weller, 2006). It has been also observed that the dissipation factor is important in determining the process efficiency. In particular, it has been published a high recovery in phenolic compounds using solvents such as ethanol or methanol, which have a higher dissipation factor, compared to water although the higher dielectric constant (Gil-Chávez et al., 2013). A high solvent dissipation factor makes the process more efficient in terms of heating up the moisture inside the matrix thus generating the pressure hold up that leads to the rupture of the cell walls and the extraction of valuable bioactive compounds. It is also important to take into account that, besides the several advantages of microwaves, there are, at least, two important limitations that hinder the development of this technology at industrial scale: first, the possibility to recover nonpolar compounds; second, the possibility to prevent the modification of the chemical structure and the bioactivity alteration of some target compounds (Gil-Chávez et al., 2013).

Significant studies have been published on the recovery of valuable compounds from the waste of fruit processing (Table 2). Two of the most important papers widely described the effect of microwave energy and treatment time on the extraction of phenolic compounds from citrus mandarin peel and pomace (Hayat et al., 2010a, 2010b). The results showed that an increase of microwave energy and time significantly increased the content of free phenolics in the extracts, while decreased the bound phenolic content. This indicated that the treatment had cleaved and liberated some of the bound phenolics from the tissue matrix thus allowing them to be available in the free form in the extracts. The studies also showed that the process has a negative effect on some compounds. In particular, an increase of the microwave energy and a longer treatment time resulted in the degradation of some flavonol compounds.

Microwaves assisted extraction has been also applied to sea buckthorn juice by-product for the recovery of flavonoids. Its efficiency has been compared to conventional solvent extraction methods (Périno-Issartier et al., 2011). The authors stated that microwaves technique could be considered as a green extraction method with several advantages like shorter extraction time (15 min), cleaner feature (no solvent or water used), and extraction of valuable flavonoids (isorhamnetin, isorhamnetin 3-O-glucoside, isorhamnetin 3-O-rutinoside, and quercetin 3-O-glucoside) at optimized power (400 W). In addition, the microwaves extracts showed much higher phenolic contents (1147 mg gallic acid equivalents (GAE) per gram) with a greater antioxidant activity in comparison with the conventional solvent extraction method (741 mg GAE/g).

Microwaves assisted extraction was also applied for pectin extraction from dried apple pomace. The response surface methodology was applied to optimize the effect of extraction processing parameters on the yield of pectin (Wang et al., 2007). The authors demonstrated that the technology significantly reduced the extraction time achieving a pectin yield of 0.32 g from 2 g of dried apple pomace.

Significant results were also achieved in the recovery of pectin from waste of *Citrullus Lanatanus* fruit rinds (Maran et al., 2014). Microwaves extraction was performed at a power ranging from 160 to 480 W, irradiation time from 60 to 180 s, pH from 1 to 2, and solid–liquid ratio from 1:10 to 1:30 g/mL. The results showed that all the process variables have significant effect on the extraction yield of pectin leading to the highest value of 25.79% with a microwave power of 477 W, an irradiation time of 128 s, a pH of 1.52, and a solid–liquid ration of 1:20.3 g/mL.

The same authors tested the efficiency of the technology also for the recovery of pectin from dried orange peels. They found the optimal process conditions of microwave power of 422 W, irradiation time of 169 s, pH of 1.4, and solid–liquid ratio of 1:16.9 g/mL at which the maximum pectin yield of 19.24% was achieved (Maran et al., 2013).

Li et al. (2011) developed a microwaves assisted extraction method for the recovery of polyphenols from grape seeds of *Vitis vinifera* cultivars of Cabernet Sauvignon, Shiraz, Sauvignon Blanc, and Chardonnay. Optimal process conditions were identified in ethanol concentration of 47.2%, solid–liquid ratio of 45.3:1 g/mL, and processing time of 4.6 min inducing about 92% of total polyphenols extraction.

In a subsequent study, the technology has been also applied for the recovery of pectin from navel orange peel, reaching an extraction yield of 18.13% and a higher intrinsic viscosity and viscosity-average-molecular weight of 0.36 L/g and 1.23×10^5

Da. The values were compared to the one of a commercial pectin (0.22 L/g intrinsic viscosity and 0.66×10^5 Da viscosity-average-molecular weight) resulting in a product with better rheological features (Guo et al., 2012).

Microwave assisted extraction was applied as prior stage before the chromatographic determination of anthocyanins in grape extracts. Among the processing parameters, the solvent was the most important variable. When methanol (40%) in water was used as extraction solvent the process was able to extract the compounds of interest in 5 min at 100°C (Liazid et al., 2011).

Hong et al. (2001) also applied the treatment to optimize the extraction of phenolic compounds from grape seeds. The microwave power (300–150 W) and the time of extraction (20–200 s) were varied to optimize the process. The results showed that neither the time nor the power had a significant effect on the overall yield (average of 13.5%) and on the polyphenol content (392 mg TAE/g of crude extract) of the extracts. However, when the solvent polarity was changed by the addition of 10% water, the yield increased to 15.2% and the polyphenol content increased to 429 mg TAE/g of crude extract.

Ultrasounds

Ultrasounds have been considered an emerging potential technology. That thanks to its capacity in accelerating heat and mass transfer, it can be successfully used in the extraction field. The efficiency of the process is related to the phenomenon called acoustic cavitation. Sound waves (frequencies higher than 20 kHz) travel in the matter causing a series of expansion and compression cycles. The expansion pulls molecules apart while the compression pushes them together. When the ultrasound waves reach a sufficient intensity, bubbles are formed in the liquid. Once formed, bubbles are able to absorb the energy from the sound waves, to grow during the expansion cycles, and be recompressed during the compression cycle. When they are no more able to absorb this energy, they collapse leading to shock waves of extreme conditions of pressure and temperature (around 100 MPa and 5000 K). The implosion of cavitation bubbles, close to a solid boundary, involves high-speed jets of liquid that can hit the surface of the food matrix. The cavitation phenomenon induces a greater penetration of the solvent into the cellular material, thus improving the mass transfer, disrupting biological cell walls, and facilitating the release of compounds (Chemat et al., 2011). Ultrasound frequency have a great effect on the extraction yield and kinetics, however the technology can have also a considerable disadvantage because the presence of a dispersed phase can attenuate the ultrasound waves due to the differences in compressibility, heat capacity, and thermal diffusion between the droplets of the dispersed phase and the continuous primary phase.

The technology has the potential to be developed and scaled up with good payback on capital investment (generally less than 1 year) due to the availability of high amplitude/power units for large commercial operations, improved energy efficiency of the equipment, ease of installation, competitive energy costs, and low maintenance costs (Patist and Bates, 2008).

The main published results on ultrasound assisted extraction for the recovery of bioactive compounds from fruit wastes are reported in Table 2. It can be observed that the technology has not only been used to extract bioactive compounds such as antioxidants and tocopherols, but also essential oils, steroids, and lipids. As concern the extraction of bioactive compounds, various studies have been published. Orange peels were treated with ultrasound to extract the flavanones hesperidin and naringin. The process conditions were optimized for the achievement of the highest extraction yield. A temperature of 40°C, an ethanol/water ratio of 4:1 (v/v), and a sonication power of 150 W were determined as optimal resulting in an enhanced extraction of 38% for naringin and 41% for hesperidin when compared to non-sonicated samples (Khan et al., 2010).

Extraction of all-*trans*-β-carotene from orange peels was also performed by ultrasound. Several processing factors were optimized including the materials particle size (< 0.074, 0.074–0.28, 0.28–0.36, 0.36–0.45, 0.45–2 mm), the extraction solvent (dichloromethane, ethanol, ethyl acetate, hexane, tetrahydrafuran), the solid/solvent ratio (1:30, 2:30, 3:30, 4:30, and 5:30), the temperature (−5, 5, 15, 25, 35, and 45°C), the extraction time (20, 40, 60, 80, 100, and 120 min), the electrical acoustic intensity (ranging from 0 to 1028.86 W/cm^2), the liquid height (2, 4, 6, 8, 10, and 12 cm, measured from the horn microtip to the tube bottom), and the duty cycle (0, 33.3%, 40%, 50%, 66.7%, and 100%) at a pulse width of 2 s (Sun et al., 2011). The results of the study indicate that each factor in the ultrasound treatment had a significant influence on the extraction yield of all-*trans*-β-carotene. Ultrasound significantly increased the extraction yield when the particles sizes were greater than 0.28 mm compared to a classical extraction. The solvent type influenced the stability and extraction yield of all-*trans*-β-carotene. Dichloromethane caused the degradation of the extract while the others induced no degradation with the ethanol having a high extraction yield similar to hexane and tetrahydrofuran. The optimal temperature for the extraction was 25°C with limited thermal degradation effects. Proper ultrasonic intensity and duty cycle were also necessary to optimize the extraction yield at the optimum conditions of particle size and solvent. The results indicated that low extraction efficiency and degradation during ultrasound-assisted extraction may occur if conditions are not optimized.

Ultrasound has been also applied to improve the extraction efficiency in terms of time needed and total polyphenol content from apple pomace. The study identified the optimal settings for the extraction as 0.142 W/g of ultrasonic power, 40.1°C of temperature, and 45 min for sonication duration. These conditions lead to an increase in the yield of catechin equivalents by more than 20% compared to the conventional maceration process. The authors concluded that the process for the recovery of polyphenol from apple pomace resulted in a relevant, rapid, and sustainable alternative to conventional procedures (such as reflux or maceration) in terms of time, energy, and enhanced final yield (Virot et al., 2010). An attempt to scale up the previous process on a 30 L extraction tank, equipped with a quadruple output of ultrasounds at 25 kHz and 4×200 W, yielded the same results (20% higher yield than the conventional maceration).

The effectiveness of the ultrasound assisted extraction was also assessed for the recovery of phenolic compounds from coconut shell powder (Rodrigues et al., 2008). Also in this

study, an optimization of the process parameters such as the temperature, solution to solid ratio, pH and extraction time was performed using surface response methodology in order to achieve the highest extraction yield. The results showed that the process yielded 22.44 mg of phenolic compounds per gram of coconut shell performing the ultrasound extraction at the optimum operating condition of 30°C, solution to solid ratio of 50, treatment time of 15 min, and pH of 6.5.

The study carried out by Chen et al. (2011) demonstrated the applicability of the technology on the extraction of polysaccharides from litchi seeds, which is a fruit with a noticeable resonance in traditional Chinese medicine thanks to their broad spectra of therapeutic and health properties. The optimal process conditions for the polysaccharides extraction were determined by the response surface methodology as follows: 15 mL/g of solvent to material ratio, 45 min of ultrasonic time, and 222 W of power. The structural and compositional analyses were also performed detecting arabinose, fructose, galactose, glucose, and mannose in different concentration depending on the extracts. The results on the composition indicated that they were neutral-type polysaccharides with defined glycosidic linkages and the calculation of the relative molar percentages was also addressed by the authors.

The ultrasound assisted extraction was also optimized to obtain the maximum recovery of phenolic compounds, antioxidant activities, and anthocyanins from grape seeds (Ghafoor et al., 2009). Also for this matrix, process variables had significant effects on the extraction of functional components with the process time being highly significant for the recovery of phenolics and antioxidants. The optimal conditions included 53.15% ethanol, 56.03°C temperature, and 29.03 min time to achieve the maximum total phenolic compounds (5.44 mg GAE/100 mL); 53.1% ethanol, 61°C temperature, and 31 min time to achieve the maximum antioxidant activity (12.31 mg/mL); and 52.4% ethanol, 55.13°C temperature, and 29.5 min time to achieve the maximum total anthocyanins (2.28 mg/mL).

Besides the optimization of the process conditions, the results published so far on this technology appeared to be incomplete regarding the stability of the extracted compounds. No information have been collected so far regarding this aspect although studies performed on ultrasound assisted extraction from plant wastes showed that phenolic compounds were less degraded (Dobiáš et al., 2010) while carotenoids were strongly affected by the process conditions usually applied (Zhao et al., 2006).

High hydrostatic pressure

The application of high hydrostatic pressure (HHP) processing in food technology started with the demonstration that the shelf life of milk and other food products could be extended by pressure treatment. HHP in food processing uses a pressure range of 100–1000 MPa. Thus, special equipments are needed to generate and endure such high pressures. A typical HHP system consists of a high pressure vessel, its head closure, a pressure generation system, and a temperature control device. The heart of the HHP system is the pressure vessel and its wall thickness that determines the maximum working pressure. Depending on the internal diameter of the vessel, the maximum working pressure is in the range of 400–600 MPa. In case of higher pressures, pre-stressed vessel designs as multilayer vessels or wire-wound vessels are used.

The most common and widely application of HHP has been developed for microbial inactivation and preservation of foodstuff. More recently, the potential of the technology has been also confirmed for the extraction of valuable compounds from food wastes. Studies showed that many structural changes occurred in foods during the application of such high pressures, such as cellular deformation, cellular membrane damage, and protein denaturation. The damage of the fruit cellular structure can improve the mass transfer rate, enhance the solvent permeability increase diffusion, reduce the processing time, and, as a result, achieve high extraction yields (Shouqin et al., 2004).

Although the promising results achieved in the commercialization of HHP technology at industrial scale, studies reporting its efficacy in the extraction of bioactive compounds from wastes of fruit processing are not so numerous (Table 2). The three studies we could recover where all published in 2009. They documented the application of the technology on litchi (Prasad et al., 2009a, 2009b, 2009c) and longan (Prasad et al., 2009a) fruit pericarp and on grape skins (Corrales et al., 2009).

As concern HHP processing of fruits pericarp, two studies optimized the conditions to achieve the highest yields and recovery of total phenolic compounds. In the study performed on longan fruit pericarp, the influence of different solvents, solvent concentration (25−100% v/v), and solid-to-liquid ratio (1:25–1:100 w/v) were individuated. Also, HPP was carried out at various pressures (200−500 MPa), durations (2.5−30 min), and temperatures (30−70°C). The results showed a higher extraction yield of total phenolic compounds (20%) compared to the conventional extraction (12%). The application of the treatment on litchi fruit pericarp leads to the same positive results. After having optimized the solvent type, the ethanol concentration (35–95 v/ v), the material to solvent ratio (1:25–1:100 w/ v), the acidic medium, the extraction pressure (200–500 MPa), the time (2.5–30 min), and temperature (30–90°C), the results lead to higher extraction yield compared to those achieved performing an ultrasonic extraction and a conventional extraction, although no significant differences were detected in terms of total phenolic content and antioxidant activity (Prasad et al., 2009a, 2009b, 2009c).

HHP performed on red grape skins for the recovery of anthocyanins was assessed testing different process parameters such as pressure (200, 400, 600 MPa), ethanol concentration (20–100%), time (30–90 min), and temperature (20–70°C) in order to achieve the highest extraction yield. Extracts obtained at an ethanol concentration of 50%, 70°C, and 600 MPa possessed the highest antioxidant capacity and the extraction yields were three-fold greater than conventional extractions. The antioxidant capacity of the extracts was not directly correlated with the highest amount of anthocyanins, which were optimally extracted using 100% ethanol, 50°C, and 600 MPa achieving extraction yields about 23% higher than those obtained under conventional conditions. Anthocyanin recovery with HHP was selective and increased according to the glucoside moiety linked to the flavylium nucleus following this extraction sequence

malvidin > peonidin > petunidin > delphinidin > cyanidin (Corrales et al., 2009).

Overall, the results achieved by the studies assessed HHP efficacy as extraction technique, besides the pasteurization and sterilization processes. However, more researches are needed based on the matrix characteristics, solvent choice, liquid–solid ratio, temperature, pressure, and time.

Pulsed electric fields

The application of pulsed electric fields (PEF) as extraction technique for the recovery of bioactive compounds has not been well studied so far. The technology involves the application of an external electrical field for few microseconds to the food placed between two electrodes. The exposure of a biological cell (plant, animal, and microbial) to high intensity fields (kV/cm) in form of very short pulses (μs to ms) induces the formation of temporary or permanent pores on the cell. This phenomenon, named *electroporation*, causes the permeabilization of cell membrane, that is, an increase of its permeability, and if the intensity of the treatment is sufficiently high, cell membrane disintegration occurs. The degree of the achieved permeabilization and thus the treatment intensity depends on several process parameters such as the electric field strength, and the number, duration, and shape of pulses.

This type of process might be beneficial for the development of quality retaining preservation and extraction processes in the food industry. However, process safety, cost-effectiveness, and consumer benefits of pulsed electric field treatment have to be confirmed.

The efficiency of the treatment has been mainly demonstrated as an alternative to traditional preservation techniques thanks to its ability to inactivate vegetative cells and spores (Heinz et al., 2001). Furthermore, the use of PEF as a pre-treatment stage before pressing and in combination with mechanical operations was investigated in order to enhance the yield of juice extraction (López et al., 2008). As concern the application of PEF as an extraction technique, just two studies have been found both applied to wastes derived from wine production (Table 2).

Corrales et al. (2008) studied PEF application (3 kV/cm) combined with a heat treatment at 70°C to extract anthocyanins. The technology showed that after 1 h of treatment, the total phenolic content of the extract was 50% higher than in the control samples performed with a water bath incubated at a temperature of 70°C for 1 h. Furthermore, the antioxidant activity of the extracts was four-fold higher. In addition, the extraction of individual anthocyanins was also studied showing that anthocyanin monoglucosides were selectively extracted by PEF.

An electrically assisted extraction was also performed on Chardonnay grape skins for polyphenol recovery (Boussetta et al., 2009). The highest level of polyphenol concentration (21.4 ± 0.8 μmol of gallic acid equivalent (GAE)/g of dry matter) was achieved after 180 min of PEF treatment.

In conclusion, the study performed so far are not too much exhaustive to justify the application of the technology for the recovery of bioactive compounds from the fruit wastes. Further studies are needed to test PEF efficiency on other waste sources and different parts of them such as seeds and stems.

Combined processes

The experience accomplished on the use of innovative extraction technologies has achieved a status where it is possible to explore the feasibility to combine them in more complex process and further increasing the extraction yield. This section addresses some of these possibilities. To our knowledge, most of the studies published so far involve the combination of SFE with other techniques such as ultrasounds, enzymes, or high pressure solvents. The possibility to combine an enzymatic pre-treatment before SFE has gaining the attention of the researchers thanks to the enzymes' ability to degrade cell walls enlarging the broken/intact cells ratio and consequently increasing the extraction yield.

Passos et al. (2009) demonstrated the potential to combine an enzymatic pre-treatment before SFE for the recovery of oil from grape seeds. The pre-treatment was carried out with cell wall degrading enzyme cocktail of cellulase, protease, xylanase, and pectinase. The choice upon the type of enzymes used was based on the knowledge that the oil removal can be favored upon partial hydrolysis of the plant cell walls by means of appropriate enzymes. The results showed the efficacy of the enzymatic pre-treatment that increased the extraction yield by 43.5% while the maximum yield of the grape seeds SFE treated was 11.5%.

Interesting works have been found in literature illustrating the possibility to integrate SFE and ultrasound assisted extraction. The combination of SFE and ultrasounds has been promising in the extraction of essential oils and antioxidants from passion fruit seeds (Barrales et al., 2015) and blackberry bagasse (Pasquel Reátegui et al., 2014). In the work of Barrales et al. (2015), the application of the ultrasound power of 160 W favored the oil extraction from passion fruit seeds, since the SFE global yield improvement achieved 29% (at 40°C and 16 MPa). The obtained oil was rich in polyunsaturated fatty acids (about 67%), tocopherol, and tocotrienol (between 60 and 90 mg/100g oil) with high DPPH radical scavenging activity (between 1.8 and 2.6 mg TE/g oil), which showed correlation with the tocopherol and tocotrienol total content.

Pasquel Reátegui et al. (2014) observed an increase of the extraction rate when ultrasound was combined with SFE for the recovery of antioxidants from blackberry bagasse. The extracts showed high antioxidant activity and phenolic contents with an increase of anthocyanins using water as co-solvent. They also analyzed the blackberry bagasse undergoing SFE with and without ultrasounds by scanning electron microscopy observing that ultrasound disturbed the cell walls, enhancing the release of the extractable compounds.

Paula et al. (2014) compared SFE assisted by ultrasound (SFE-US) with pressurized solvent extraction (PLE) for the recovery of phenolic compounds from blackberry residues. During SFE-US three variables were optimized: temperature (40, 50, and 60°C), pressure (15, 20, and 25 MPa), and ultrasound power (0, 200, and 400 W) while CO_2 flow rate (2.77×10^{-4} kg/s) and extraction time (120 min) were kept constant. In PLE, four different solvents (water, acidified water at pH 2.5, ethanol, and ethanol + water 50% v/v) and three temperatures (60, 80, and 100°C) were used while pressure (7.5 MPa), solvent to feed ratio (18.0 kg/kg residue), and extraction time (30 min)

were kept constant. The optimized conditions for SFE-US were 60°C, 15 MPa, and 200 W, and for PLE were 100°C, ethanol + water 50% v/v. The two processes held to comparable extraction yield estimated equal to 6.35% for SFE-US and 6.33% for PLE. In addition, the following results were also achieved for both techniques, respectively: total phenolics equal to 0.091 and 7.36 mg gallic acid equivalent/g residue; monomeric anthocyanins equal to 0 and 1.02 mg cianidin-3-glicoside equivalent/g residue; antioxidant activity equal to 0.54 and 76.03 μmol trolox equivalent/g residue (DPPH) and 1.39 and 68.28 μmol trolox equivalent/g residue (ABTS).

Recently, a growing interest has been shown on coupling membrane separation techniques to SFE process due to the advantages of both methods. The resulted combined process is based on the simple ideas that the extracts obtained by SFE, even with high yields, have low selectivity especially for solutes with similar molecular weights. The membrane works as a molecular sieve capable of fractionating the extract in terms of molecular weight or steric conformation.

In SFE, the separation of extracts is usually achieved by depressurization or cooling with the solvent going to the gaseous phase and the subsequent collection of the extracts. However, the cost of the solvent recompression to liquid or supercritical phase is high thus coupling SFE with membranes allows keeping CO_2 at high pressures with significant energetic and economical savings (Viganó et al., 2015). Furthermore, the physical properties of CO_2 in supercritical phase allows to operate with a high permeate flux due to the low viscosity (10 times lower than water and diffusivity 10 to 100 times higher).

Spricigo et al. (2001) tested a reverse osmosis membrane made of cellulose acetate in the separation of nutmeg essential oil extracted with SFE. The membrane provided a mean retention of 96.4% of the oil, not being significantly affected by the temperature, transmembrane pressure, and concentration of oil in the feed. At pressure of 12 MPa, higher permeate fluxes were obtained with a transmembrane pressure of 4 MPa and no losses on the retention of essential oil. The flux of CO_2 was linearly proportional to the applied transmembrane pressure, being reduced with the increase of oil concentration in the feed. The membrane presented high permeability to CO_2, and an adequate resistance to the severe pressure conditions applied.

On the same side, Sarmento et al. (2004) evaluated two thin-film reverse osmosis membranes and a cellulose triacetate/diacetate blend membrane in terms of permeability of supercritical CO_2 and retention of lemongrass, orange, and nutmeg essential oils at 12 MPa and 40°C. The oil retention and permeability of CO_2 were evaluated as function of feed concentration and transmembrane pressure. The flux of CO_2 was linearly dependent on the applied transmembrane pressure, but was not affected by the oil concentration in the feed. The thin-film membrane had the highest essential oil retention (up to 90%) but the lowest CO_2 flux (8.75 kg/h m^2) when subjected to a pressure gradient of 1 MPa. The other tested membranes presented good permeability to CO_2 and resistance to the severe pressures applied.

Following this trend, Carlson et al. (2005) suggested the possibility to use the reverse osmosis membrane separation and nanofiltration membrane as alternative to avoid the intense CO_2 depressurization step, which is necessary for the recovering of limonene.

The low-film reverse osmosis membrane proved to be the best choice for the separation of supercritical CO_2 and limonene mixture. A limonene retention factor as high as 0.94 was achieved for a high feed concentration of limonene (30%) but low fluxes of CO_2. In addition, almost 70% of the solvent (permeate CO_2) was recycled with only 0.5 MPa of pressurization lost (from 11.5 to 12 MPa) while the other 30% of the solvent (retentate CO_2) needed to be pressurized from 5 to 12 MPa with an occurred phase change and more energy required to reach the extraction pressure. On the contrary, the polyamide reverse osmosis membrane showed no limonene retention and the nanofiltration membranes presented a low limonene retention factor of 0.3 and a good permeate CO_2 flux.

Future perspectives and final remarks

The ever-growing demand for the extraction of fruit bioactive compounds encourages continuous search for convenient, non-thermal, and *GRAS* (generally recognized as safe) status extraction methods. The selection of the most sustainable extraction technique depends on the source and the kind of bioactive compound to be extracted. As an example, SFE is the best technique used for apolar compounds such as carotenoids and fatty compounds, while PLE and ultrasounds are more suited for more polar compounds, such as polyphenols. Although several papers have been published so far, demonstrating that some of these technologies can be efficiently applied in this field, their scale up, and industrial implementation is still at their infancy and required extensive and deeper studies. To our knowledge, SFE and HHP are mature techniques already used at the industrial scale but their applications for the recovery of bioactive compounds from fruit wastes are not yet developed.

For this reason, the understanding of every aspect of these non-conventional extraction processes is vital as most of these technologies are based on different mechanism. Accordingly, the extraction yield can vary as the result of different factors. The engineering project of the extraction systems with the possibility of a scale up requires knowledge about thermodynamic constraints as solubility, selectivity, kinetic parameters, and mass transfer. For this reason, more researchers are required in order to develop mathematical models taking into account all the factors mentioning above and help in the comprehension of the processes mechanisms.

Besides the technologies aspects, it should be also taken into account the product nature and in particular, to consider that bioactive compounds are usually a minor part of the fruit by-product. The exhausted materials derived as by-product from the extraction process may also have a commercial application. In particular, some matrices collected after the extraction consist of carbohydrates that may also have a commercial application. Therefore, at industrial scale sustainable solutions should take into account the possible treatment of all usable streams present in the process. For example, from apple pomace it is possible to extract polyphenols choosing the right extraction technique and subsequently treat the remained exhausted pomace for pectin recovery.

The possibility to combine technologies such as PEF, ultrasound, and microwave assisted extraction with pressurized fluid extraction techniques to enhance extraction efficiencies is another field with increasing potentials but not yet completely exploited.

Following all the above considerations, future investigations should be also performed in order to conduct integral studies, which include not only the development of recovery protocols, but also findings on specific applications and preservation assays in order to secure an industrial exploitation and sustainability of the final product. Encapsulation is the key recovery stage that needs further investigation, as it is able to improve functionality and extent the shelf-life of the extracted products.

Finally, it will be very important and necessary to raise awareness among the consumers on the advantages derived from the application of these technologies in order to create a new class of functional foods that will be able to replace the common synthetic pharmaceuticals compounds by the natural nutraceuticals ones.

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