CHAPTER

Food industry by-products valorization and new ingredients: Cases of study

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1. Introduction: waste and wastewaters from food industry

Along the time, different institutions have promoted and attempted to define what can be considered as food waste. In this sense, in Europe as well as in the United States, it was established a similar waste hierarchy for the waste treatment: (1) reduction; (2) reuse; (3) material recovery; (4) energy recovery; and (5) waste disposal (Directive, 2008/98/EC; USEPA, 2001).

In 2009, the Food and Agriculture Organization (FAO) of the United Nations published a report that estimated the world population will reach in 2050 around 9.1 billion and the main increment of population will take place in developing countries (FAO, 2009). Besides, the FAO also performed a study published in 2011 that resulted in the conclusion that one-third of food produced all over the world gets lost during the food chain from farm (agricultural production) to fork (consumption in households) (FAO, 2011; Carciochi et al., 2017). According to the FAO (2011), if food lost occurs during the production, postharvest, and processing stages, then it is called food losses. On the other hand, when food lost is in the retail or consumption stages, it is named food wastes.

In 2012, the ISEKI Food Association established the term "food waste recovery" with the meaning of "extraction of valuable compounds from waste by-products" (Galanakis, 2018; ISEKI, 2012).

The European Commission, in 2014, redefined the concept "food waste" as the "loss of food, including nonedible parts, from the food supply chain, that are not sent for animal feed, redistribution …" (Galanakis, 2018). Considering the above discussion, here and after, this meaning for food waste will be used.

Due to the general characteristics of food wastes (organic load, high moisture, and microbial activity, among others), they generate different problems on the

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environment such as CO_2 emissions, toxicity in surface and ground waters, changes in soil quality, etc. (Nayak and Bhushan, 2019; FAO, 2011).

Reduction of food waste is one of the goals of the United Nations to achieve a more sustainable world by 2030 (United Nations, 2015). According to this idea, the European Union has proposed different strategies to promote a reduction in waste production, facilitating the food donation and improving labeling. On the other hand, to reduce the amounts of wastes, it is possible also to focus the efforts on the recovery of substances and compounds included in waste streams from food industry (Galanakis, 2018).

The environmentally sustainable food production has to reconsider wastes as by-products that can be transformed to provide valuable compounds (antioxidants, fiber, and fuels, among others) and then be used as new products or raw materials in the food industry or even applied in other sectors such as pharmaceutical, polymer, and energy industries (Carciochi et al., 2017).

In this sense, the term biorefinery has been appeared as the whole strategy focused on a green economy, considering all steps in the food chain production, for the achievement of a zero-waste concept (Fava et al., 2015; Nayak and Bhushan, 2019; Carciochi et al., 2017). In this context, biocompounds are considered as biological compounds that can be used to increase the nutrient content in human diets and are extracted mainly from plant materials such as fungi, legumes, cereals, grains, vegetables, and fruits (Radrigán et al., 2017; Quintin et al., 2019).

In addition, due to the increasing consumers' request on natural ingredients derived from natural sources with almost no content in toxins, pesticides, etc., and the growing demand on nutraceutical products, some food industries are including natural food products, as wild products, as a new source of healthy food resources.

2. Food ingredients obtaining

The extraction, recovery, concentration, and purification operations of valuable ingredients (antioxidants, vitamins, proteins, sugars, colorants, etc.) from food waste and from nonwidely exploited harvest such as wild fruits become a key step to guarantee both environmental and economic sustainability of the whole obtaining process. Furthermore, whatever are the steps considered in the ingredients obtaining, it is necessary to maintain the stability of the desired healthy properties of these ingredients.

All of these operations are based mainly on the application of a combination of chemical, thermal, and physical processes on food or their wastes matrices (Nayak and Bhushan, 2019). There are three fundamental issues to consider: (1) the concentration of the ingredients to be extracted is usually low; consequently, the cost of the extraction and concentration can be high; (2) in general, target ingredients are thermolabile and this limit the use of some technologies (Galanakis, 2018; Carciochi et al., 2017); and (3) it is necessary to evaluate the toxicity and amount of residues obtained by using some extraction operations.

Available technologies used for the extraction and recovery of biocompounds can be classified by conventional and nonconventional technologies. Conventional technologies are considered as classic and traditional operations (solvent extraction, ultrafiltration [UF], microfiltration [MF], nanofiltration [NF], adsorption, centrifugation, alcohol precipitation, electrodialysis, thermal and/or vacuum concentration, among others), whereas nonconventional are those technologies that are more recently studied and, in some cases, implemented in industry. Some examples are ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), liquid membranes, low-temperature plasma treatment, colloidal gas aphrons, enzyme-assisted extraction, nanotechnology, and pulsed electric field (Galanakis, 2012; Carciochi et al., 2017; Radrigán et al., 2017; Nayak and Bhushan, 2019; Renard, 2018).

Some of these technologies and examples of application are summarized in Table 5.1 according to their widespread use in food industry or their promising applicability and focused on the environmental and health sustainability.

3. Successful study cases

3.1 Wild vegetables as a source of value-added compounds

One of the main interests in food by-products rises from different axis like recovery and exploitation of natural alternative resources, pollution reduction, environmental awareness, and many others that joint in conjunction to accomplish food security (Galanakis, 2012). Worldwide malnutrition and food inequity are important subjects that have led researchers to investigate alternatives products to address these problematics.

Unbalanced diets and food scarcity are the main problems that converge in undernourished populations; therefore, for a diet to be balanced, it must be composed of a wide range of food groups that ensure the delivery of nutrients that the body requires (Roberfroid, 2011). Even though fruits and vegetables are relatively accessible for some social groups, in certain regions of the world, some factors such as adverse environmental conditions, overpopulation, social-political problems, and poverty affect the access to agroindustrial crops and the consequent famine and malnutrition, on those regions. However, although a large percentage of our diet relays on technified agriculture, wild fruits, vegetables, and spices are still being used in some social groups as a source of nutrition and folk medicine, due to their high content in phytochemicals (Bvenura & Sivakumar, 2017).

In this sense, the interest for alternative vegetables, which present benefits and unique qualities, has grown in recent years (Martins and Ferreira, 2017), and therefore, they are described in this section. In this section, some vegetables rich in phenolic compounds have been selected.

Table 5.1 Characteristics and extraction examples of different conventional and nonconventional extracting operations.

	Characteristics	Extraction examples
Conventional treatments		
Solvent extraction	Characterized by the use of organic solvents, mainly ethanol and high time and energy consumption. Solvents have to be considered as GRAS (generally recognized as safe) by USFDA (1–2).	Pectin from orange peel and grapefruit peel (3–4). Phenolics from pomegranate wastes (5).
Membrane technology	Used for the treatment of liquid streams from food industry. Based on a tangential filtration in which a semipermeable membrane is used as a separation agent (6). Membrane processes most used are microfiltration, ultrafiltration, nanofiltration, and reverse osmosis. Other membrane operations are membrane distillation and osmotic distillation.	Phenolics from olive mill wastewaters and artichoke processing wastewaters (7–9). Betalains and colorants from different sources (10).
Nonconventional treatments		
Ultrasound-assisted extraction (UAE)	Alternative to conventional extraction technologies (11–12). Based on the use of frequencies between 20 kHz and 100 MHz (13–14) that generate the cavitation effect and facilitate the extraction of ingredients from the solid matrix (15). UAE promotes a reduction of extracting time and solvent amounts and increases the quality and concentration of biocompounds in extracts.	Anthocyanins from carrot pomace (16). Flavonoids from grapefruit peels and kiwifruit juice (17–18). Carbohydrates from herb, wood, algae, and mushrooms (15).
Microwave-assisted extraction (MAE)	Based on the use of high frequencies (300 MHz–300 GHz). Reduces the extraction time since it promotes a quicker heating, decreases thermal gradients, and minimizes the need of high amounts of extracting solvents (13, 19).	Polyphenols from pomegranate peels and grape marcs (20–21). Lycopene from tomato peels (22). Pectins from orange and mango peels (23–24).

Table 5.1 Characteristics and extraction examples of different conventional and nonconventional extracting operations.—*cont'd*

	Characteristics	Extraction examples
Supercritical fluid extraction	Based on the use of supercritical fluid (SF) characterized by its dissolving properties of a liquid and a viscosity and diffusivity close to vapors. SF reduces energy consumption and organic solvents resulting in a higher quality of extracts (11, 13, 25–28). The most SF used is CO_2.	Antioxidants and polyphenols from apple pomace, agroindustrial soybean wastes and hazelnut, coffee, and grape wastes (29–30).
Pulsed electric field	Based on the application of high-voltage pulses to a product using electrodes. High-voltage pulses provoke cell membrane rupture, a decreasing of the extraction time, and an increment of the permeability of cell membranes (11, 13, 25–27)	Proteins from waste chicken meat (32). Pigments as carotenoids from tomatoes (33).
Enzyme-assisted extraction (EAE)	The EAE is getting more importance since it is considered a green extraction method due to the organic and natural nature of enzymes. It allows the rupture of cell membranes and favors the recovery of biocompounds (13, 25–27).	Proteins and antioxidants from sesame bran (34).

1. Cvjetko Bubalo et al. (2018); 2. USFDA (2016); 3. Georgiev et al. (2012); 4. Bagherian et al. (2011); 5. Saffarzadeh-Matin and Khosrowshahi (2017); 6. Galanakis et al. (2016); 7. Garcia-Castello et al., 2010; 8. Conidi et al. (2014); 9. Conidi et al. (2015); 10. Raghavarao et al. (2014); 11. Galanakis (2012); 12. Paniwnyk et al. (2017); 13. Radrigán et al., 2017; 14. Lavilla and Bendicho (2017); 15. Herrera and Luque de Castro (2005); 16. Agcam, Akyıldız and Balasubramaniam (2017); 17. Garcia-Castello et al., 2015; 18. Wang et al. (2019); 19. Martín and Navarrete (2018); 20. Kaderides et al. (2019); 21. Garrido et al. (2019); 22. Eh and Teoh (2012); 23. Maran et al. (2013); 24. Maran et al. (2015); 25. Carciochi et al., 2017; 26. Renard, 2018; 27. Quintin et al., 2019; 28. Galanakis (2018); 29. Ferrentino et al. (2018); 30. Alvarez et al. (2019); 31. Manna et al. (2015); 32. Ghosh et al. (2019); 33. Bot el al., 2018; 34. Görgüç et al. (2019).

Besides the plants mentioned in Table 5.2, there are many other wild vegetables with a great potential of study due to their bioactive profile. Scarcity of precise identification of these compounds has only shown in some documents general screening of the phenolic profiling, but they have not been studied in depth, leaving a great potential for the research of recovery and exploitation of more natural phytochemicals that could be employed as new functional ingredients (Lin and Lin, 1997; El-Kader et al., 2006).

Table 5.2 Selected potential wild vegetables rich in bioactive compounds.

Scientific name	Common name	Uses	Distribution	Principal bioactive compounds	References
Amaranthus spinosus	Quelite/phak khom	Food, beverage	America, asia	Amaranthine, quercetin, kaempferol, rutin	1,2,3
Apium nodiflorum (L) Lag.	Erba canella/ rabaças	Food, ethnomedicine	Italy	Germacrene D, allo-ocimene, limonene	4,5
Calendula spp.	Calendula/ margarita	Food, cosmetics, ethnomedicine	Mediterranean	Caffeic acid, quercetin	6,7
Cichorium intybus L.	Belgian endive/ chicory	Food, ethnomedicine	Europe*	Chicoric acid, caffeoylquinic acid, quercetin	8,9,10
Foeniculum vulgare Mill.	Fennel	Food, ethnomedicine	Spread around the world	Caffeoylquinic acid, quercetin, kaempferol, isorhamnetin	11,12,13
Portulaca oleracea L.	Verdolaga/ purslane	Food, ethnomedicine	Spread around the world	Kaempferol, apigenin, luteolin, myricetin	14
Carduus tenuiflorus Curtis	Sheep thistle	Ethnomedicine	Spread around the world	n/f	15
Beta macrocarpa Guss.	Acelga de fruto grande	Ethnomedicine	Mediterranean	n/f	16

*1. Balakrishnan et al. (2011); 2. Hilou et al. (2006); 3. Peter and Gandhi (2017); 4. Maxia et al. (2012); 5. Carvalho et al. (2011); 6. Faustino et al. (2018); 7. Guijarro-Real et al. (2019); 8. Abbas et al. (2014); 9. Gallucci et al. (2017); 10. Tardugno et al. (2018); 11. Caleja et al. (2014); 12. Levorato et al. (2018); 13. Qurishi et al. (2012); 14. Farkhondeh and Samarghandian (2019); 15. Enrique de Briano, Acciaresi and Briano (2013); 16. Iamonico (2019); *Majoritarian Italian usage.*

3.2 Citric industry: traditional processing and new environmental and integrated processes for the recovery of food ingredients

The citrus processing industry plays an important role in the agroindustrial sector. According to data published by the FAO (2016), citrus represents the third most important fruit crop with a production of about 124,246,000 tons.

In contrast with other types of fruit, citrus fruits can be consumed mostly fresh or pressed to produce juice. Furthermore, citrus fruits can be processed to obtain other food products such as dehydrated citrus products such as jams, jellies, or marmalades.

Citrus by-products are considered to be an economic and renewable source of valuable compounds such as minerals, dietary fibers, oils, lipids, pectins, and bioactive compounds, such as polyphenols and carotenoids.

To obtain them, it is possible to use conventional techniques, but recently, it has been developed green or clean techniques based on reduced use of energy, short extraction time, decrease of solvent consumption, overall enhancement of extraction rate, enhancement of the quality extracts, improvement of aqueous extraction processes, and improved extraction of heat-sensitive compounds (Vilkhu et al., 2008).

Different studies have analyzed the use of green technologies for the recovery of bioactive compounds from citrus by-products. MAE, UAE, and membrane processes are among the most innovative techniques developed recently (Conidi et al., 2018; Khan et al., 2010).

An optimized MAE method for the extraction of polyphenol compounds from citrus mandarin juice peels has been reported by Hayat et al. (2009), and higher extraction yields of phenolic acids and antioxidant capacity of the extracts were obtained, if compared with traditional technology.

Garcia-Castello et al. (2015) compared conventional extraction and UAE of flavonoids from grapefruit peels, and the experimental results showed better performance of UAE in terms of extraction yields (on average total phenolic content 50% and total antioxidant activity [TAA] 66% higher), at lower temperature and extraction time, when compared with conventional solvent extraction.

Membrane processes have been also successfully investigated for the recovery of biological active compounds from citrus by-products, thus transforming these by-products to source material for high added-value compounds.

Conidi et al. (2012) investigated the use of spiral-wound NF membranes with different molecular weight cutoff (MWCO) (from 250 to 1000 Da) and polymeric material (polyamide, polypiperazine amide, and polyethersulfone) for the separation and concentration of phenolic compounds from red range press liquors.

In a previous work, the same authors studied the concentration of polyphenols from bergamot juice, a by-product of the essential oil production, using tight UF and NF membranes (Conidi et al., 2011).

As a green extraction technique, an innovative process on orange peel wastes was developed by Boukroufa et al. (2015). The process involved the use of MAE and

UAE in an integrated system, for the recovery of essential oil, polyphenols, and pectin from orange peel.

In a similar approach, Fidalgo et al. (2016) studied the extraction of pectin and D-limonene from waste orange and lemon peel by an innovative eco-friendly process using water as dispersing medium and microwaves as energy source. The investigated process permitted to obtain high yields of extracted selected compounds, with high quality and environmental viability.

A sustainable process based on the integration of UF, NF, and osmotic distillation (OD) membrane processes for the recovery and concentration of flavonoids from orange press liquor was investigated by Cassano et al. (2014). The press liquor was previously clarified by a UF membrane module in hollow fiber configuration and concentrated from 10 to 32°Brix by using an NF membrane in spiral-wound configuration. The NF retentate was finally concentrated by OD up to 47°Brix, working in conditions of low mechanical and thermal damage. The concentrated extracts, produced at low temperatures without thermal damage to the compounds of interest, offered interesting perspectives for the use of these products as natural colorants and/or for nutraceutical applications.

A schematic of integrated green processes for the recovery of valuable compounds from citrus by-products is illustrated in Fig. 5.1.

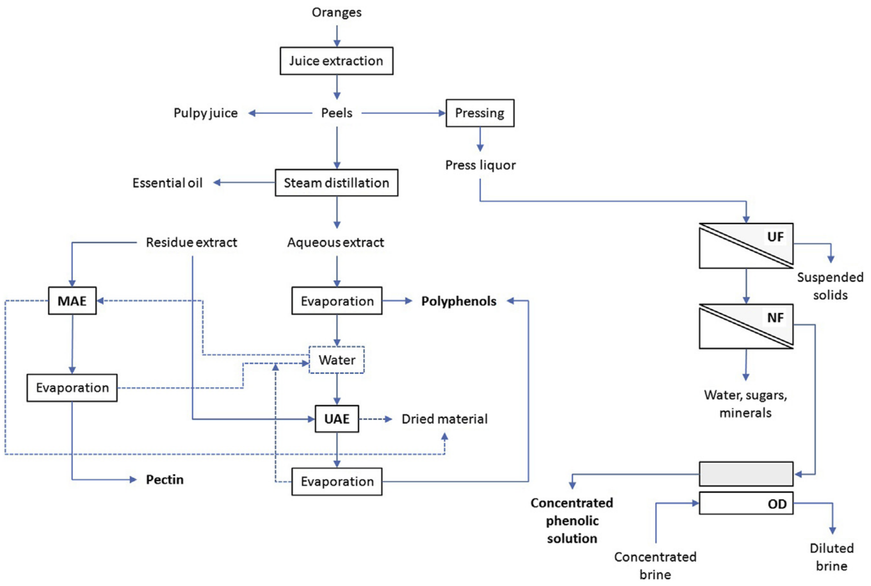

FIGURE 5.1

Integrated green processes for the recovery of valuable compounds from citrus by-products (*MAE*, Microwave-assisted extraction; *UAE*, Ultrasound-assisted extraction; *UF*, Ultrafiltration; *NF*, Nanofiltration; *OD*, Osmotic distillation).

Adapted from Boukroufa et al., 2015 and Cassano et al., 2014.

3.3 Olive oil industry: traditional processing and new environmental and integrated processes for the recovery of food ingredients

Olive oil production is one of the most traditional agricultural industries with a great economic importance in most of the Mediterranean countries.

The by-products obtained are a solid fraction (called olive pomace), and a liquid fraction called olive mill wastewater (OMW). This liquid stream is the mix between olive vegetation water and the possible underprocess added water.

The organic fraction of OMWs contains sugars, tannins, polyphenols, polyalcohols, pectins, lipids, proteins, and organic acids (Cassano et al., 2011). OMWs are considered highly polluting effluents due to their high organic load and require a complex management and disposal system due to their negative effects on the environmental (such as antimicrobial, ecotoxic, and phytotoxic properties) (Paraskeva et al., 2007).

Different management options have been proposed for the treatment of olive wastes, but independently by the efficiency of those physical, chemical, and biological treatments, the disposal of great amounts of sludge produced remains a significant problem in the OMW treatment.

According to hierarchy of the EU and EPA aims, aforementioned in the Section 1 of this chapter, it is increasing attention to the valorization of these streams through the recovery and/or the biotransformation of their organic matter as the recovery of antioxidant substances and phenols, which can be exploited in pharmaceutical, food, and cosmetical applications (Kalogerakis et al., 2013). For instance, their introduction in foodstuffs could decrease the use of synthetic antioxidants, such as butylate hydroxyanisole, butylated hydroxytoluene (BTH), and tert-butylhydroquinone. Due to their potential negative effects on health, these synthetic antioxidants are restricted to maximum concentrations in foodstuffs (Araújo et al., 2015).

The efficient recovery of phenolic compounds has been extensively studied and optimized to maximize their reintroduction in the food chain and contribute to a higher valorization and better management of OMWs (Kalogerakis et al., 2013).

Excellent biological properties in terms of antioxidant, free radical scavenging, antimicrobial, and anticarcinogenic activities of the polyphenols of OMWs have been reviewed by different authors (Obied et al., 2005; Araújo et al., 2015). The most important biophenols include benzoic acid and hydroxycinnamic acid derivatives and, in larger amounts, tyrosol, hydroxytyrosol, and oleuropein.

The efficiency of a green approach for the recovery of phenols from OMWs based on the use of UAE was studied by Klen and Vodopivec (2011). Results showed higher recoveries at both levels of individual and total phenol yields in comparison with conventional technologies (filtration, solid-phase, and liquid-liquid extraction).

Membrane processes have been also successfully used as "green processes" for the recovery of water, organic compounds, and polyphenols from OMWs. These processes, mostly in a sequential form or combined with other separation technologies, successfully meet the requirement for the recovery, purification, and concentration of antioxidants from OMWs suitable for food or pharmaceutical formulations (Conidi et al., 2018).

Garcia-Castello et al. (2010) investigated the recovery and concentration of OMW phenolic compounds from OMWs using an integrated membrane system based on conventional pressure-driven processes (MF and NF) and OD. Results showed that about 78% of the initial phenolic compound content was recovered in the microfiltered permeate solution. The MF permeate was then submitted to NF, after which a concentrated solution containing approximately 0.5 g/L phenolic compounds, with hydroxytyrosol representing 56% of the total, was obtained by processing the NF permeate with OD.

A combination of UF and NF processes for the recovery of biologically active compounds and water from OMWs was investigated by Cassano et al. (2013). In this approach, raw wastewaters were firstly clarified with a UF hollow fiber membrane module (HFS, Toray) to remove suspended solids and colloidal substances. Afterward, the UF permeate was processed by a UF unit equipped with a flat-sheet membrane. The produced permeate stream was finally concentrated by using an NF unit equipped with a spiral-wound NF membrane. UF membranes allowed to recover most part of phenolic compounds in the permeate streams due to their low rejection toward these components (in the range of 26%–31%, respectively). On the other hand, a rejection higher than 93% toward total polyphenols was measured by the NF90 membrane. Free low-molecular-weight polyphenols, including caffeic acid, p-coumaric, catechol, tyrosol, procatechuic acid, and hydroxyl-tyrosol, were completely retained, in accordance with the estimated MWCO (200 Da) of the membrane and the MW of target compounds (138–284 g/mol). The NF retentate, enriched in polyphenols, was considered suitable for food, cosmetic, and pharmaceutical formulations, whereas the purified aqueous fraction (NF permeate) could be reused in the olive oil extraction or for membrane cleaning.

Conidi et al. (2014) evaluated the performance of a multiphase biocatalytic membrane reactor in the conversion of a UF permeate containing oleuropein into oleuropein aglycon catalyzed by β-glucosidase immobilized in a polymeric membrane. The maximum oleuropein conversion reached was about 45.7%, and the reaction rate was about 2×10^{-4} mmol/min cm^3.

An integrated membrane system based on a sequential combination of pressure-driven membrane operations and innovative membrane systems has been recently implemented by Bazzarelli et al. (2016). OMWs were firstly acidified with sulfuric acid to achieve a complete removal of suspended solids and then clarified by MF. The MF permeate was treated by NF to obtain water from the permeate side and a concentrated polyphenolic solution from the retentate side. The NF retentate was dewatered by OD by using a polypropylene hollow fiber membrane contactor (Liqui-Cell Extra-Flow 2.5 × 8″, Membrana) and calcium chloride dihydrate at 60 w/w% as stripping solution. The OD retentate was finally encapsulated in a water-in-oil emulsion by membrane emulsification. According to the process mass balance, about 1.5 kg of phenolic compounds (85% of the initial phenolic content) and 800 L of purified water can be recovered from 1 m^3 of OMW (Fig. 5.2).

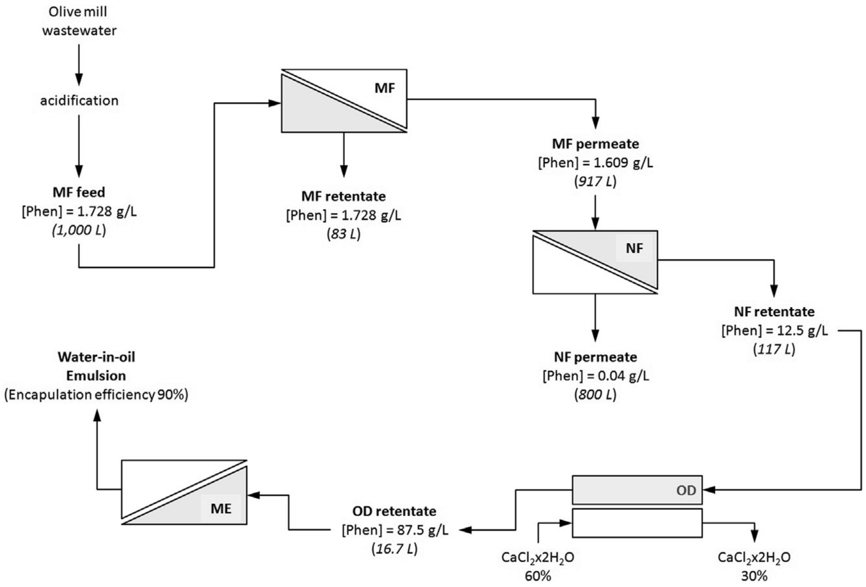

FIGURE 5.2

Integrated membrane process used for the fractionation of olive mill wastewaters (*MF*, Microfiltration; *NF*, Nanofiltration; *OD*, Osmotic distillation; *ME*, Membrane emulsification).

Adapted from Bazzarelli, F., Piacentini, E., Poerio, T., Mazzei, R., Cassano, A. & Giorno, L. (2016). Advances in membrane operations for water purification and biophenols recovery/valorization from OMWWs. Journal of Membrane Science, 497, 402–409. https://doi.org/10.1016/j.memsci.2015.09.049.

3.4 Cactus pear industry

3.4.1 Characterization and evaluation of new ingredients from fruits and their wastes

Within the Cactaceae family, *Opuntia* genus has strong cultural and economic significance throughout the Americas and some arid environments regions in the world as crops for their alimentary and forage products. World scientific community has constantly increased their publications, with 249 records including the words "*Opuntia* fruit" in their titles, until January 2019 (WOS, 2019). Scientific records mainly come from Italy, Mexico, United States, and Morocco with more than 50% of the total publications. Among these, the variety *ficus-indica* (OFI), commonly known as prickly pear or cactus pear, is widely studied, although some authors have also researched on some of the 300 wild more uncommon species (spp.) (Morales-Martinez et al., 2018; Morales et al., 2014; Melgar et al., 2017).

Opuntia spp. fruits vary substantially on their morphological and chemical attributes, for instance, the percentage of peel fluctuates from 25% in OFI to nearly 65% in *Opuntia engelmannii* variety (Melgar et al., 2017). Since only the edible part of the fruit is used in the food chains, a huge amount of waste could be transformed into a potentially useful by-product, providing high added-value compounds with food, cosmetic, and pharmaceutical applications (Galanakis, 2012).

Table 5.3 presents a wide variety of bioactive compounds recovered from *Opuntia* spp. by-products. Constituents such as pectins, phenolic acids, flavonoids, carotenoids, betalains, tocopherols, and phytosterols have been previously described (Amaya-Cruz et al., 2019; Morales-Martínez et al., 2018; Morales et al., 2014; Melgar et al., 2017). These compounds could provide ample possibilities of use of this by-product as a source of new ingredients with added healthy properties (Valdés and León, 2017).

Despite the importance of phenolic and nonphenolic compounds within *Opuntia* spp. by-products, this section focuses on their inner natural pigments. Betalains are water-soluble nitrogen-containing compounds present in a restricted number of families of the plant order of Caryophyllales. Besides anthocyanins, carotenoids, and chlorophylls, betalains complement the natural widespread molecules used for coloring purposes (Delgado-Vargas and Paredes-Lopez, 2003). These compounds are situated in the vacuoles of the plant and their basic structure consisting of a moiety of betalamic acid and, depending on the residue, could be classified as red/purple betacyanins and yellow-orange betaxanthins (when cyclo-Dopa [cyclo-3-(3,4-dihydroxyphenylalanine)] and hydroxycinnamic acid derivatives or sugars, and amines or amino acids residues also bind to betalamic acid, respectively) (Herbach et al., 2006).

The ever-increasing demand for new natural ingredients has brought the development of a plethora of pigment extraction systems and their description in the scientific literature. Sample preparation is considered a crucial part of this process and frequently, eco-friendly solvents and technologies, which contemplate recovery, recycling, and sustainability, are the most commonly used (Płotka-Wasylka et al., 2017). Otherwise, generation of hazardous toxic waste could result contradictory to the concept of by-products recovery. For instance, due to the polarity of betalains and morphological properties of the by-products, solid-liquid extractions are the most commonly used (Barba et al., 2017), with aqueous-based solvents used mainly as the extracting phases, and also often mixed with other volatile organic compounds applied as organic solvents (Amaya-Cruz et al., 2019; Koubaa et al., 2016; Mena et al., 2018; Morales et al., 2015; Melgar et al., 2017). Extraction techniques and characterization of the complex nonhomogeneous extracts are also varied and differed among the scientific community. In Table 5.4, frequently used solvents and techniques are listed along with recurrent final detection techniques.

Besides betalains' ability of pigmentation, their antioxidant ability and capacity to absorb free radical makes this natural colorant an excellent alternative to the artificial counterpart, due to their potential benefits against chronical diseases such as cardiovascular, inflammatory, diabetes, cancer, oxidative stress, and other diseases associated with aging (Amaya-Cruz et al., 2019; Garcia-Cruz et al., 2017; Herbach et al., 2006; Sawicki et al., 2016; Stintzing et al., 2005).

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.

Family	Compound name	Variety	Author
Hydroxybenzoic acids	2-Hydroxybenzoic acid	OFI	Amaya-Cruz et al. (2019)
	2,3-Dihydroxybenzoic acid	OFI	Amaya-Cruz et al. (2019)
	2,4-Dihydroxybenzoic acid	OFI	Amaya-Cruz et al. (2019)
	3-Hydroxybenzoic acid	OFI	Amaya-Cruz et al. (2019)
	3,5-Dihydroxybenzoic acid	OFI	Amaya-Cruz et al. (2019)
	4-Hydroxybenzoic acid	OFI; OM; OMT	Amaya-Cruz et al. (2019), Ndhlala et al. (2007), Guzmán-Maldonado et al. (2010)
	4-Hydroxybenzoic acid 4-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Benzoic acid	OFI	Amaya-Cruz et al. (2019)
	Ellagic acid	OFI	Amaya-Cruz et al. (2019)
	Ellagic acid acetyl-arabinoside	OFI	Amaya-Cruz et al. (2019)
	Ellagic acid arabinoside	OFI	Amaya-Cruz et al. (2019)
	Eucomic acid	OFI	Melgar et al. (2017), Chougui et al. (2015)
	Gallic acid	OMT	Guzmán-Maldonado et al. (2010)
	Gallic acid 3-*O*-gallate	OFI	Amaya-Cruz et al. (2019)
	Isovanillic acid	OFI	Amaya-Cruz et al. (2019)
	Piscidic acid	OFI	Melgar et al. (2017), Chougui et al. (2015)
	Protocatechuic acid	OFI; OM	Amaya-Cruz et al. (2019), Ndhlala et al. (2007), Mena et al. (2018)
	Protocatechuic acid 4-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Syringic acid	OFI	Amaya-Cruz et al. (2019)
	Vanillic acid	OFI; OMT	Amaya-Cruz et al. (2019), Guzmán-Maldonado et al. (2010)

Continued

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
Hydroxycinnamic acids	3-*O*-Caffeoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	3-*p*-Coumaroylquinic acid	OFI	Amaya-Cruz et al. (2019)
	3,4-*O*-Dicaffeoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	4-*O*-Caffeoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	4-*p*-Coumaroylquinic acid	OFI	Amaya-Cruz et al. (2019)
	4-Sinapoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	5-*O*-Caffeoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	5-*p*-Coumaroylquinic acid	OFI	Amaya-Cruz et al. (2019)
	5-Sinapoylquinic acid	OFI	Amaya-Cruz et al. (2019)
	Caffeic acid	OFI; OM	Amaya-Cruz et al. (2019), Ndhlala et al. (2007)
	Caffeic acid 4-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Caffeic acid hexoside	OMT	Morales et al. (2015)
	Caffeoyl tartaric acid	OFI	Amaya-Cruz et al. (2019)
	Cinnamic acid	OFI	Amaya-Cruz et al. (2019)
	Dihydrosinapic acid hexoside	OFI	Mena et al. (2018)
	Ferulic acid	OFI; OM	Amaya-Cruz et al. (2019), Ndhlala et al. (2007), Mena et al. (2018)
	Ferulic acid 4-*O*-glucoside	OFI; OMT	Amaya-Cruz et al. (2019), Morales et al. (2015)
	Isoferulic acid	OFI	Amaya-Cruz et al. (2019)
	m-Coumaric acid	OFI	Amaya-Cruz et al. (2019)
	o-Coumaric acid	OFI	Amaya-Cruz et al. (2019)
	p-Coumaric acid	OFI; OM	Amaya-Cruz et al. (2019); Ndhlala et al. (2007)
	p-Coumaric acid 4-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Sinapic acid	OFI	Amaya-Cruz et al. (2019)
	Sinapic acid hexoside	OFI	Mena et al. (2018)

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
Flavan-3-ols	(+)-Catechin	OMT	Guzmán-Maldonado et al. (2010)
	(−)-Epicatechin	OMT	Guzmán-Maldonado et al. (2010)
	(−)-Epicatechin 3-*O*-glucose	OFI	Amaya-Cruz et al. (2019)
Flavonols	Isorhamnetin	OFI; OL	Amaya-Cruz et al. (2019), Kuti (2004), Moussa-ayoub et al. (2011), Chougui et al. (2015)
	Isorhamnetin 3-*O*-glucoside	OFI; OSC; OMT	Amaya-Cruz et al. (2019), Moussa-ayoub et al. (2011), Melgar et al. (2017), Yeddes et al. (2013), Morales et al. (2015)
	Isorhamnetin 3-*O*-rutinoside	OFI; OMC; OSC; OMT	Moussa-Ayoub et al. (2011); Yeddes et al. (2013); Morales et al. (2015)
	Isorhamnetin 4′-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Isorhamentin-*O*-(deoxyhexosyl-hexoside)	OFI; OE; OMT	Melgar et al. (2017); Morales et al. (2015)
	Isorhamentin-*O*-(deoxyhexosyl-pentosyl-hexoside)	OFI	Melgar et al. (2017)
	Isorhamnetin-*O*-(di-deoxyhexosyl-hexoside)	OFI; OMT	Melgar et al. (2017); Morales et al. (2015)
	Isorhamentin-*O*-(pentosyl-hexoside)	OFI	Melgar et al. (2017)
	Kaempferide	OFI	Amaya-Cruz et al. (2019)
	Kaempferol	OFI; OL, OSP	Amaya-Cruz et al. (2019); Kuti (2004)
	Kaempferol 3-*O*-(2″-rhamnosyl-galactoside) 7-O-rhamnoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-(6″-acetyl-galactoside) 7-O-rhamnoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-acetyl-glucoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)

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Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
	Kaempferol 3-*O*-glucosyl-rhamnosyl-galactoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-glucosyl-rhamnosyl-glucoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-glucuronide	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-rhamnoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3-*O*-rutinoside	OFI	Amaya-Cruz et al. (2019); Melgar et al. (2017); Mena et al. (2018)
	Kaempferol 3-*O*-xylosyl-glucoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 3,7,4′-*O*-triglucoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol 7-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Kaempferol-*O*-(di-deoxyhexosyl-hexoside)-hexoside	OMT	Morales et al. (2015)
	Myricetin	OFI	Amaya-Cruz et al. (2019)
	Myricetin 3-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Myricetin 3-*O*-rhamnoside	OFI	Amaya-Cruz et al. (2019)
	Myricetin hexoside	OFI	Mena et al. (2018)
	Quercetin	OFI; OL, OSP, OSC	Amaya-Cruz et al. (2019), Kuti (2004)
	Quercetin 3-*O*-arabinoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-glucoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-glucosyl-rhamnosyl-glucoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-glucosyl-xyloside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-rhamnosyl-galactoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-rutinoside	OFI; OSC	Melgar et al. (2017), Yeddes et al. (2013), Mena et al. (2018)

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
	Quercetin 3-*O*-xyloside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3-*O*-xylosyl-rutinoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 3,4′-*O*-diglucoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin 7,4′-*O*-diglucoside	OFI	Amaya-Cruz et al. (2019)
	Quercetin-*O*-(di-deoxyhexosyl-hexoside)	OMT	Morales et al. (2015)
Flavanones	Hesperidin	OFI	Amaya-Cruz et al. (2019)
	Naringenin	OFI	Amaya-Cruz et al. (2019)
	Naringenin-hexoside	OFI	Mena et al. (2018)
	Naringin	OFI	Mena et al. (2018)
Betacyanins	15-Decarboxy-betanin	OFI	Amaya-Cruz et al. (2019)
	17-Decarboxy-betanin	OFI	Amaya-Cruz et al. (2019)
	17-Decarboxy-neobetanin	OFI	Amaya-Cruz et al. (2019)
	2-Decarboxy-betanin	OFI; OMT	Amaya-Cruz et al. (2019), Morales et al. (2015)
	2-Decarboxy-isobetanin	OMT	Morales et al. (2015)
	2-Decarboxy-neobetanin	OFI	Amaya-Cruz et al. (2019)
	2,15,17-Tridecarboxy-betanin	OFI	Amaya-Cruz et al. (2019)
	2,15,17-Tridecarboxy-neobetanin	OFI	Amaya-Cruz et al. (2019)
	Betalamic acid	OFI	Amaya-Cruz et al. (2019)
	Betanidin	OFI; OE; OSC	Amaya-Cruz et al. (2019), Melgar et al. (2017), Yeddes et al. (2013)
	Betanidin-5-*O*-β-sophoroside	OE	Melgar et al. (2017)
	Betanin	OFI: OMC; OE; OSC; OMT	Amaya-Cruz et al. (2019), Moussa-ayoub et al. (2011), Melgar et al. (2017), Yeddes et al. (2013), Morales et al. (2015)

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Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
	Gomphrenin I	OE	Melgar et al. (2017)
	Gomphrenin II	OFI	Amaya-Cruz et al. (2019)
	Gomphrenin III	OFI	Amaya-Cruz et al. (2019)
	Hylocerenin	OFI	Amaya-Cruz et al. (2019)
	Isobetanin	OFI; OE; OSC; OMT	Amaya-Cruz et al. (2019), Melgar et al. (2017), Yeddes et al. (2013), Morales et al. (2015)
	Isophyllocactin	OFI	Amaya-Cruz et al. (2019)
	Neobetanin	OFI	Amaya-Cruz et al. (2019)
	Phyllocactin	OFI	Amaya-Cruz et al. (2019)
Betaxanthins	3-Methoxy-tyramine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	5-Hydroxynorvaline-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Alanine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Arginine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Aspartic acid-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Dopa-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Dopamine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Glutamine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Histidine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Indicaxanthin	OFI	Melgar et al. (2017)
	Isoleucine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Lysine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Methionine sulfoxide-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Methionine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Phenylalanine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Proline-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Proline-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Serine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Threonine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Tryptophan-betaxanthin	OFI	Amaya-Cruz et al. (2019)

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
	Tyramine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Tyrosine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	Valine-betaxanthin	OFI	Amaya-Cruz et al. (2019)
	γ-Aminobutyric acid-betaxanthin	OFI	Amaya-Cruz et al. (2019)
Carotenoids	Antheraxanthin	OFI	Amaya-Cruz et al. (2019), Cano et al. (2017)
	Astaxanthin	OFI	Amaya-Cruz et al. (2019)
	Bixin	OFI	Amaya-Cruz et al. (2019)
	Canthaxanthin	OFI	Amaya-Cruz et al. (2019)
	Flavoxanthin	OFI	Amaya-Cruz et al. (2019)
	Lutein	OFI	Cano et al. (2017)
	Lycopene	OFI	Cano et al. (2017)
	Neoxanthin	OFI	Amaya-Cruz et al. (2019), Cano et al. (2017)
	Neurosporene	OFI	Amaya-Cruz et al. (2019)
	Norbixin	OFI	Amaya-Cruz et al. (2019)
	Physalien	OFI	Amaya-Cruz et al. (2019)
	Phytofluene	OFI	Amaya-Cruz et al. (2019)
	Violaxanthin	OFI	Amaya-Cruz et al. (2019), Cano et al. (2017)
	Zeaxanthin	OFI	Cano et al. (2017)
	α-Carotene	OFI	Amaya-Cruz et al. (2019)
	α-Cryptoxanthin	OFI	Amaya-Cruz et al. (2019), Cano et al. (2017)
	β-Apo-8′-Carotenal	OFI	Amaya-Cruz et al. (2019)
	β-Carotene	OFI	Amaya-Cruz et al. (2019), Cano et al. (2017)
	β-Cryptoxanthin	OFI	Cano et al. (2017)
	ζ-Carotene	OFI	Amaya-Cruz et al. (2019)
Phytosterols	Campesteryl β-d-glucoside	OFI	Amaya-Cruz et al. (2019)
	Fucosterol	OFI	Amaya-Cruz et al. (2019)
	Lanosterol	OFI	Amaya-Cruz et al. (2019)
	Sitosteryl β-d-glucoside	OFI	Amaya-Cruz et al. (2019)
	Stigmasteryl β-d-glucoside	OFI	Amaya-Cruz et al. (2019)
Chlorophylls	Chlorophyll *a*	OFI	Cano et al. (2017)
	Chlorophyll *b*	OFI	Cano et al. (2017)

Continued

Table 5.3 Bioactive compounds cited in *Opuntia* spp. peels.—*cont'd*

Family	Compound name	Variety	Author
Lignin	Guaiacyl(8-*O*-4) syringyl(8—8) guaiacyl-hexoside	OFI	Mena et al. (2018)
	Guaiacyl(t8-*O*-4) guaiacyl-hexoside	OFI	Mena et al. (2018)
	Secoisolariciresinol-hexoside	OFI	Mena et al. (2018)
	Syringaresinol	OFI	Mena et al. (2018)
	Syringyl(t8-*O*-4) guaiacyl	OFI	Mena et al. (2018)

OE, Opuntia engelmannii; *OFI*, Opuntia ficus-indica; *OL*, Opuntia lindheimeri; *OM*, Opuntia megacantha; *OMC*, Opuntia macrorhiza; *OMT*, Opuntia matudae; *OSC*, Opuntia stricta; *OSP*, Opuntia streptacantha.

3.4.2 Industrial processing: integrated processes for the recovery of bioactive compounds

Regarding phenolic composition, Aguirre et al. (2013) investigated the extraction of polyphenols from *Opuntia ficus-indica* skin by using a reflux system. The optimum conditions for the extraction of polyphenols were 45% of ethanol, 80°C, and 2 h of extraction. Higher temperatures led to an increase of the extraction of polyphenols but a decrease of the antioxidant potential.

The potential of an integrated membrane process for producing cactus pear juice concentrate, with high nutritional value, has been investigated by Cassano et al. (2007). The process included an initial clarification of the depectinized juice by UF. The clarified juice was then concentrated by OD from 11 °Brix of total soluble solids (TSS) up to 61 °Brix. The content of Vitamin C, glutamic acid, citric acid, and betalains was very well preserved during the process independently by the TSS content. The evaluation of the TAA in the concentrated samples confirmed the validity of the process in preserving bioactive compounds of the fresh juice.

Therefore, the OD retentate was considered suitable for nutraceutical applications (i.e., nutritional supplements) or as coloring foodstuff due to its high concentration of betalains.

Cassano et al. (2010) evaluated also the effect of polyvinylidenfluoride flat-sheet MF and UF membranes (0.20 μm as pore size and 200 kDa as MWCO, respectively) on the physicochemical composition of the cactus pear juice. The clarified juice showed physicochemical and nutritional properties similar to those of the fresh juice except for the absence of suspended solids and betalains, which were retained by the selected membranes. Accordingly, the retentate fraction can be used as a raw material to extract betalains or directly as functional food.

More recently, an integrated membrane-based process aimed to purify, fractionate, and concentrate betacyanins in cactus pear juice has been investigated by

Table 5.4 Extraction systems and final detection analysis using *Opuntia* spp. by-products.

Technology of extraction	Final detection technique	Solvent	Author
Homogenization	HPLC-DAD	Methanol:water (50/50)	Kuti (2004)
	HPLC-DAD-MS	Tetrahydrofuran	Cano et al. (2017)
Maceration	UPLC-QTOF-MS^e	Methanol:water (50/50) Acetone:water (70/30)	Amaya-Cruz et al. (2019)
	HPLC-UV-Vis	Methanol:water (50/50)	Ndhlala et al. (2007)
	HPLC-DAD	Methanol:water (70/30)	Guzmán-Maldonado et al. (2010)
	UPLC-DAD-ESI/MS^n	Ethanol:water (80/20)	Melgar et al. (2017)
	HPLC-QTRAP-MS	Methanol:water (80/20)	Morales et al. (2015)
	Spectrophotometry	Methanol:water (66/34)	Diaz-Vela et al. (2013)
	UPLC-DAD-ESI/MS^n	Ethanol:water (70/30)	Chougui et al. (2015)
Microwave-assisted extraction	Scanning electron microscope	Water	Han et al. (2016)
Pulsed electric field	HPLC-DAD	Water	Koubaa et al. (2016)
Reflux extraction	Spectrophotometry	Ethanol:water	Aguirre-Joya et al. (2013)
Supercritical fluids extraction	UPLC-QTOF-MS	Carbon dioxide	Koubaa et al. (2016)
Ultrasound-assisted extraction	HPLC-DAD	Methanol:water (80/20)	Moussa-Ayoub et al. (2011)
	HPLC-DAD	Methanol:water (70/30)	Moussa-ayoub et al. (2011)
	RP-HPLC-ESI-MS	Methanol:acetic acid (99/1)	Yeddes et al. (2013)
	UHPLC-ESI-MS	Methanol:water (80/20)	Mena et al. (2018)

HPLC, *High-performance liquid chromatography;* UHPLC, *Ultrahigh-performance liquid chromatography;* RP, *Reverse phase;* UPLC, *Ultra-performance liquid chromatography;* DAD, *Diode array detector;* ESI, *Electrospray ionization;* MS, *Mass spectrometry;* UV-Vis, *Ultraviolet visible spectra;* QTOF, *Quadrupole time of flight;* QTRAP, *Triple quadrupole linear ion trap.*

Tamba et al. (2019). The proposed design is based on the MF of depectinized juice to promote the removal of insoluble solids, followed by UF or NF treatment for solute separation. By selecting the appropriate UF or NF membrane and operating pressure, the retention of solutes can be modulated to promote the concentration of all the solutes or, alternatively, the purification of betacyanins with respect to the total dry matter.

4. Conclusions

Next generations will have to focus on different issues: (1) population increment that leads to a higher food needs; (2) healthy authorities and consumers require each time higher food standards in terms of nutritional values and safety of food products; and (3) solve environmental problems related to waste generation along the food chain.

This chapter emphasizes in the need of the use of green and clean technologies for the waste treatment and for the recovery and obtaining of biocompounds from food wastes. Besides, some case studies have been described as successful source of biocomponents that can be recovered from various vegetable sources.

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