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Article

SPECIAL GUEST EDITOR SECTION

Reuse of Food Waste and Wastewater as a Source of Polyphenolic Compounds to Use as Food Additives

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Abstract

The problem of waste and byproducts generated from agro-industrial activities worldwide is an increasing concern in terms of environmental sustainability. In this ambit, the quantity of food wastes—produced in all steps of the whole food chain—is enormous, and it may be forecasted that food waste could amount to more than 120 billion tonnes by 2020. The reuse of food waste and wastewater as source of polyphenolic compounds could be an interesting discussion in this ambit. In fact, polyphenols obtained in this way might be used for food and non-food purposes by means of new, improved, and safe extraction methods. In light of the opportunity represented by the treatment of agro-industrial waste, different systems concerning the winemaking and olive oil production industries have also been discussed as describing approaches applicable to other sectors. More research is needed before considering recovery of phenolic compounds from wastewater as an economically convenient choice for the food sector.

Polyphenolic Compounds from Food Waste and Wastewater. State of Art

The agri-food industry is one of the largest manufacturing sectors in the world. The waste and byproducts obtained from agri-food transformation, as well as other waste typologies coming from other industrial sectors, are today causing notable concerns for the environment, animal and plant health, and sustainability. This amount of food waste, food processing waste and food loss is estimated to be about 1.3 billion tonnes/year (1). Generally, food waste is produced during all phases of the food life cycle from agricultural production activities to industrial processing, manufacturing and distributing steps. Without any prevention activity, or policy undertaken, food wastes are expected to rise up to about 126 billion tonnes by 2020 (2).

The remarkable amounts of biodegradable solid or liquid waste generated in the ambit of agri-food industries consist mainly of organic residues from processed raw materials. The food wastes generated from various processing industrial are characterized high variability and high volumetric capacity. These properties depend on the nature of produced waste, the production process, and the site of production. For this reason, dedicated predictions may be challenging in this ambit.

In particular, food processing wastewater has unique characteristics when compared with other wastewaters because of its chemical composition (proteins, carbohydrates, etc.), chemical-physical variables such as chemical and biochemical oxygen demand (COD and BOD, respectively), sensorial features, etc. The disposal of such a heterogeneous material may be challenging enough in a general ambit of environmental sustainability (3, 4).

Improper disposal practices of such wastes can result in environmental problems like toxicity to aquatic life, pollution of surface and ground waters, altered soil quality, phytotoxicity, colored natural waters, and odor. For this reason, worldwide legislation requirements for handling of the waste and its disposal has become increasingly restrictive over the last decade (5).

Against the backdrop of the problems associated with the large-scale generation of food waste and its subsequent disposal, the European Commission encourages the recycle of food wastes as raw materials for new products in the ambit of circular economy and environmentally-friend technologies until 2020 (6). Concepts such as long-lasting design, maintenance, repair, reuse, remanufacturing, refurbishing, and recycling (seven basic actions) are strongly needed (7). In other words, a circular

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economy promotes resource minimization and the adoption of cleaner technologies (8).

Several methodologies have been proposed for the management of these wastes including decantation, separation, dissolved air flotation, de-emulsification, centrifugation, coagulation, flocculation, adsorption, advanced oxidation processes, bioreactors, ozonation, enzymatic treatments, and coupled processes, i.e., electrocoagulation (9–11), all of which aim to reduce organic matter from downstream processing wastes.

The efficiency, complexity, and cost-effectiveness of these methods may vary significantly. Combined physicochemical and biological systems seem to guarantee high efficiency in terms of pollution control. However, the great amount of sludge produced remains a significant problem in the management of agro-food wastewater.

Due to such enormous amounts of food-related materials that are discharged worldwide, technologies that include recycling, recovery and sustainability of high-value added ingredients inside food chain are promising (12). Nowadays, known solutions to handle the overwhelming problem of food waste management and related safe disposal have paved the way to various valorization strategies and techniques so as to generate useful end products. The main strategies for the valorization of food wastes concern (13):

(a) The biotechnological transformation into chemicals or biofuels.
(b) The conversion of food waste to generate biofuels such as bio-gas, bio-alcohol, bio-hydrogen, bio-char and bio-diesel.
(c) The extensive use as fertilizers or animal feeds.

Among the various valorization technologies correlated to energy conversion, biological fermentation processes have been implemented because of their simplicity and cheapness, although some inherent disadvantages (such as longer processing times) have to be taken into account.

Fermentation of food waste into bio-ethanol is a viable waste management option, but its application on large scale can only be possible after detailed techno-economic analyses. Hydrothermal carbonization is another attractive option for converting such waste into useful products such as hydro-char, hydro-oil, and other energy-rich compounds. It has several advantages with respect to environmental, energy, economical and health aspects. However, the thermal processes of incineration, pyrolysis, and gasification have failed from an energetic point of view on account of the high moisture content, lower heating values, and greater heterogeneity inherent in the food wastes as well in increasing greenhouse gas (GHG) emissions (14). An advantage offered by agri-food wastes is that they are almost entirely of biological origin; consequently, they can then represent a biological and readily available resource.

In fact, this heterogeneous material is particularly rich in secondary metabolites, which are the same as those present in the primary foods before transformation. These are widely recognized as endowed with numerous biological activities, and used as a model to build, for example, new pharmaceutical devices. Among these, a peculiar class of bio-organic compounds – polyphenols – albeit less represented in the natural world, possess peculiar and specific biological roles, in particular defensive and protective functions for organisms with self-synthetic abilities, and also for humans when consumed in the diet.

The exploitation of byproducts from fruit and vegetable processing industries as a source of functional compounds for their application in foods is a promising field requiring interdisciplinary research by food technologists, food chemists, nutritionists, and toxicologists. During the past few decades, several attempts have been broadly made to develop feasible methods and different ways utilizing vegetable and fruit wastes in therapeutic purposes (15). The preparation of certain valuable products can be achieved by means of the valorization of agro-industrial wastes by recovery of high-value components such as polysaccharides, proteins, fibers, phytochemicals, and flavor compounds, that are used as functional ingredients and nutraceuticals (16). In this respect, bioactive constituents such as phenolic compounds can be recovered as well as pharmaceuticals and cosmetics (17, 18). Limited resources and increasing interest in the use of bioactive compounds play an important role in the development of sustainable waste management practices. In contrast to de-polluting approaches, the recovery of bioactive compounds from agro-food wastes is one of the most important challenges for sustainable industrial processes due to their potential uses such as ingredients in foodstuffs, pharmaceutics, and cosmetic formulations.

Among these compounds, polyphenols (PP), secondary metabolites ubiquitously distributed in all higher plants, have been recently most researched (19). The importance of PP has been reported in different ambits and production areas (food, pharmaceutical articles, etc.) because of claimed properties and dedicated actions against pathogenic agents, ultraviolet light, and other eco-menaces (20–22). The chemical classification of PP may be challenging enough. Eleven sub-classes at least may be identified representing more than 8000 aromatic structures (derived from tyrosine or *L*-phenylalanine), with a recognized identification of about one flavonoid per two phenolic molecules (23–26). Notable protective activities have been ascribed to PP: these molecules are recognized as antioxidants, free radical scavengers, and metal chelators. The ability to reduce and inhibit different types of enzymes (telomerase, lipoxygenase, and cyclooxygenase) has been considered (19, 27).

Phenolic compounds are considered as the most important natural antioxidants (28, 29). Polyphenols have gained great importance as phytochemical compounds due to several associated health benefits concerning lifestyle diseases as well as oxidative stress (18). They also contribute to the prevention of several types of human diseases, such as cardiovascular disease, cancer, osteoporosis, diabetes, and neurodegenerative diseases. In particular, some phenolic compounds (e.g., flavan-3-ol; flavonol, tannin, and neolignan) have demonstrated specific health protective effects such as antimicrobial activity against viruses, bacteria, and fungi (19, 26, 30, 31). For example, epidemiological studies have shown the positive correlation between the Mediterranean diet and the low incidence of cardiovascular diseases and certain kinds of cancer (breast, prostate, and skin cancer, intestine). Mediterranean diet's healthy effects can be related to the consumption of extra-virgin olive oil, which is rich in polyphenolic compounds, antioxidant substances (31).

Their role has been clearly recognized by the European Food Safety Authority (EFSA), which allows the following health claim: "*olive oil polyphenols contribute to the protection of blood lipids from oxidative stress*". It has also been reported that a great variability exists in terms of concentration of phenolic compounds in virgin olive oils found on the retail market (32). Several review papers report on phenolic compounds in olive oil and their health benefits, thus the reader can refer to them for further details on the nutritional properties of these compounds (33, 34).

The use of olive oil wastewater (OMW) extracts in foods is a new trend in the food sector to formulate new products with positive effects on consumers health. In addition, the recovery of natural phenolic compounds is of great interest due to their importance in terms of antioxidant effect to better preserve the quality and shelf life of food (35). Consequently, the production of functional foods from OMW extracts constitutes a viable alternative to transform this agro-industrial waste stream into a useful and relevant ingredient (36, 37).

Agro-Food Wastewater as Source of Polyphenolic Compounds

Worldwide legislation requirements for the handling of waste and its disposal have become increasingly restrictive over the last decade (14). Limited resources and increasing interest in the use of bioactive compounds play an important role in the development of sustainable waste management practices. Wastewater reuse potential, in different industries, depends on waste volume, concentration and characteristics, best available treatment technologies, operation and maintenance costs, and finally, availability of raw water and effluent standards (3). The recovery of bioactive compounds from agro-food waste is one of the most important challenges for sustainable industrial processes. Wastewater from winemaking and oil mills can represent, due to their high polyphenolic content, interesting sources for the extraction and reuse of biocomponents such polyphenolic compounds (13, 14, 38).

Food-related PP are also a matter of interest for consumers when speaking of health promotion, reduction of human diseases and related risks, and anti-pathogenic actions. The reason is basically correlated with the possibility to use of antioxidants of natural origin, such as polyphenols compounds, to replace synthetic food additives (39, 40).

However, PP are reported to be effective on condition that bioavailability, stability, and bioactivity are preserved. This aspect includes chemical, physical, and biological conditions (41–43). In addition, the comparison of numerous studies concerning the correlation between different treatments and demonstrated PP amount may be challenging enough because of contradictive results (14, 26, 44, 45).

The use of encapsulated PP instead of free compounds is the subject of numerous studies. Also, microencapsulated products are widely used in the food, pharmaceutical, and cosmetic industries but also in various other domains: personal care, agricultural products, veterinary medicine, industrial chemicals, biotechnology, biomedical, and sensor industries (43, 46). Certain byproducts from the orange juice industry can be used to obtain high dietary fiber powders for carrier-applications when speaking of encapsulation of phenolic extracts from pomegranate peels. In this way, two food waste types that are beneficial to health were combined into one multipurpose functional food.

Various methods are used for encapsulation of phenolics: spray drying, spray cooling/chilling, extrusion, fluidized bed coating, coacervation, liposome entrapment, inclusion complexation, centrifugal suspension separation, lyophilization, co-crystallization, emulsion, etc.

The selection of encapsulation method depends upon specific application and parameters, such as required particle size, physicochemical properties of the core and coating materials, release mechanisms, process cost, etc. In addition, many phenolic compounds show limited water solubility and have an unpleasant taste, which must be masked before their incorporation in foodstuffs or oral medicines. Therefore, the supplementation of phenolic compounds requires the formulation of a finished protecting product able to maintain structural phenolic integrity until consumption (43).

Microencapsulation is one of the techniques used for enhancing shelf life and stability of phenolics (42). It is defined as a process in which tiny particles or droplets are surrounded by a coating or embedded in a homogeneous or heterogeneous matrix, to produce small capsules with many useful properties. Despite their limited post-recovery applications, many studies have addressed the potential reuse of phenolic compounds recovered from agri-food wastewater.

Recently, phenolic compounds (tocopherol mixtures, and α-tocopherol) recovered from olive mill wastewater have been tested against ascorbic acid for the prevention in oil oxidation. Galanakis et al. (20) reported that, the oxidation of both extra virgin and a refined olive oil was reduced by olive phenolics, even if the ascorbic acid resulted more efficient than olive polyphenols. In addition, some other phenolic-based compounds are also attractive for the production of foodstuffs: this is the case of betalains (47) and anthocyanins (48) which are considered as potential natural colorants for food and pharmaceutical or cosmetic uses. Galanakis et al. evaluated the addition of olive phenols to bakery products to enhance the oxidative stability of these products, and prolonging related shelf life (21). A concentration of 200 mg PP/kg resulted in the most efficient antimicrobial formulation in bread making, with the extension of the preservation of rusks from 6 to 12 weeks.

These results suggest the potential use of olive PP as antioxidant and antimicrobial agents in food products that are sensitive to oxidative deterioration during cooking. Esposto et al. evaluated the effect of phenolic extracts recovered from a byproduct of the olive oil process on the quality of olive oil during deep frying at 180°C. Results indicated the capability of the extract in terms of the preservation of α-tocopherol content and reduction of undesirable volatile compounds in olive oil during the frying process (49). Similarly, Servili et al. used phenolic compounds extracted from olive mill wastewater to fortify milk beverages fermented with γ-amino butyric acid and autochthonous human gastrointestinal lactic acid bacteria (50).

A crude phenolic concentrate obtained by membrane treatment was also used with the aim of improving virgin olive oil phenolic content (34). Results obtained with four different olive cultivars showed that the crude concentrate increased the phenolic content of olive oils without any alteration of the aroma profile. Furthermore, phenolic-based fractions recovered from olive mills by membrane separation have also been tested for their antioxidant activity (51).

Mohammadi et al. evaluated the antioxidant activity of olive leave extract (OLE) encapsulated by nanoemulsions in soybean oil. It was found that nanoencapsulated OLE was capable of controlling peroxide value better than unencapsulated OLE, whereas due to blocking phenolic compounds within dispersed emulsions droplets, thermal stability of encapsulated OLE was lower (52).

In addition, Urzu et al. added OLE and encapsulated OLE microparticles into starch-gluten fried matrices (53). Selani et al. and Garrido et al. studied the effect of addition of grape pomace extracts on raw and cooked chicken meat and pork burgers, respectively (54, 55). They reported that grape seed extracts were able to reduce rancid flavor development in various meat products, as also reported by Perumalla et al. (56). The antioxidant activity of seed extract was concentration-dependent between 0.02% and 0.1% (w/w). They also founded that addition of grape seed extract more than 1 mg/g resulted in a minor increase in the surface color of raw meat and retention in cooked meat, which may have a negative impact on consumer preference based on color without affecting the eating quality, as also reported by Ahn et al. (57). The application of phenolics from grape pomace extracts in dairy products was also studied: Silva et al. found that phenolics at a concentration of 0.5 mg/mL milk improved the antioxidant properties, increased gel strength, and decreased curd moisture content, whereas it did not affect physical attributes such as texture or firmness of the final cheese (58).

Spigno et al. showed the efficiency of encapsulated grape marc extract in improving the shelf life of hazelnut paste by inhibiting its oxidation (59). With reference to other possible uses of polyphenol compounds, Galanakis et al. suggested their application in sunscreen formulations to complement UV filter photoprotection of synthetic compounds for their activity as UV filters in a broader region of UV-B and UV-A spectra (22). In addition, the entrapment of olive phenols in silica particles, prior to their emulsification in cosmetics, improved their water resistance revealing the potential use of phenols from olive mill wastewater as UV booster in cosmetics (60).

Antibiotic resistance hence is a challenging subject from different viewpoints; the above-mentioned points have demonstrated that a multidisciplinary approach is needed in the ambit of public health and safety on the one hand, and in the food industry on the other.

With relation to food-related uses of wastewater from the food and beverage industry, two basic examples are described here - winemaking and olive oil production.

Wastewaters and Phenolics: The Winemaking Case Study

Grapes are one of the most traditional fruits in the world, and *Vitis vinifera* L. is the main species grown for the wine industry. This industry is a sector of great potential worldwide the global wine production in 2018 was 292 mhl (61). Wine production generates approximately 1.3 to 1.5 kg residues/L of produced wine, 75% of which is winery wastewater (62) that is generated in washing operations during grape harvesting, pressing, and first fermentation steps. Such solid wastes have practically no utility except for use as cattle feed or as a fertilizer; however, prolonged use as a fertilizer has resulted in germination problems due to toxicity associated with high levels of PP in waste matrixes (63). Consequently, the annual disposal of grape pomace worldwide represents a serious environmental and economic problem. However, both fruit and wine production leads to wasted mass such as grape pomace, grape seeds, and grape skin: all of these wastes contain important phenolics (64). As a result, these residues can represent an inexpensive source of beneficial phytochemicals, which could be successfully used in the food, cosmetic, and pharmaceutical industries (65). Moreover, grapes are one the world's largest fruit crops with most production in France, Spain, and Portugal. With concern regarding the large-scale generation of grape pomace wastes from wineries of such countries, the growing research interest in the effective extraction or recovery of PP is evident (14).

The interest on grape pomace as a potential source for polyphenol recovery depends on the fact that this waste byproduct has a significant proportion of PP due to the partial maceration during the winemaking process (66). Only a small part of these compounds is transferred from grape to wine, while large quantities remain in the pomace, the byproduct consisting of pressed grape leftovers (e.g., seeds, skin, stems). The primary phenolic compounds present in grape byproducts are (+)-catechin, (-)-epicatechin, quercetin, myricetin, rutin, kaempferol, gallic acid, ellagic acid, syringic acid, caffeic acid, and *trans*-resveratrol (67). The most well-known polyphenol present in grape wastes, with good commercialization prospects, is resveratrol. Also, grape pomace contains grape pigments such as anthocyanidins that structurally are derivatives of malvidin, peonidin, and cyanidin (68). These phenolic compounds could be exploited as natural colorants in food matrixes (69, 70).

Grape seeds contain also phenolic acids, ellagitannins, flavones, flavan-3-ols, anthocyanins, stilbenes, and resveratrol. Polyphenols can be extracted by means of the use of traditional organic solvents, although alternative systems based on enzymes or improved liquid extraction methods are reported (66, 70–73). Extraction efficiency also depends on the distribution and nature of the PP in the grape pomace matrix (74). During the winemaking process, skins and seeds from grapes are kept in contact with the fermenting wine for several days so as to increase the phenolic content of produced wines. However, grape residues in the form of pomace still contain high polyphenol levels, especially retained in the skin matrix. Although a majority of PP are bound via hydrophobic or hydrogen bonds to cell wall polysaccharides, there are also PP which are found in the cell cytoplasm, inside the cellular vacuoles or associated with the nucleus (74). Thus, non-cellular PP can be recovered by traditional solvent-solid extractions, and more tightly-bound cellular PP may be extracted by more advanced techniques (74). Conventional solvent extraction is the most widespread technique used in laboratories and on an industrial scale to extract bioactive compounds from plant matrixes. Nowadays, safe and simple extraction protocols without organic solvents and chemical additives have received significant attention.

Díaz-Reinoso et al. evaluated the performance of different ultrafiltration (UF) and nanofiltration (NF) membranes with molecular weight cut-off (MWCO) in 2009 (75). UF and NF processes are able to separate specific compounds through a sieving mechanism based on MWCO (76–79). Using UF, Galanakis et al. (2015) have separated polyphenols from pectins containing waste. Up to 99% retention of the phenols was achieved for some polar phenolics, such as o-diphenols and hydroxycinnamic acids (78).

Recently, some authors (73, 80, 81) have also investigated the effect of supercritical carbon dioxide extraction (pressure and temperature), and the effect of solvent type as some of the parameters evaluated to identify optimal, low-cost, environmentally-friendly phenolic extraction from stems separated from grape pomace. The best results were obtained by working at high pressure (400 bar) and low temperature (35°C), with an increase of extraction yields of 60% for stems of white grape varieties, using 5%, v/v ethanol as a co-solvent. The intensification on total PP extracted from the stems of white and red grape varieties can be seven times higher after HVED-assisted extraction (81, 82). Also, the effect of various organic (methanol, ethyl acetate) and inorganic (aqueous potassium hydroxide) solvents was also investigated (73) proving that both extracts, ethyl acetate ones and those treated with supercritical carbon dioxide at various extraction pressures, were enriched in phenolic compounds. In addition, the use of pulsed electric field (PEF) for the intensification of extraction has been reported in this ambit (83, 84).

Should PEF technology be applied to grapes, the extraction of PP would increase even at moderate temperature ($T = 25°C$) during vinification and fermentation (85). Both higher temperatures and HVED improved the extraction of PP. Heat is generally assumed to damage grape cell membranes, which results in an increased extraction of PP (86). However, Cacace et al. reported that PP are thermodegradable: consequently, 60°C was chosen as the upper and critical limit with reference to extraction (87). Finally, Boussetta et al. reported that HVED application is useful for reducing extraction times and temperature (81).

Wastewater and Phenolics: The Olive Mill Case Study

Around 6×10^6 m^3 OMW are produced yearly worldwide: 98% of OMW is produced in the Mediterranean basin. About 2.5 L of waste are released into the environment for each liter of olive oil produced, with a total estimated amount of:

(a) 1.4 million m^3 of OMW produced every year in Italy alone.
(b) Over 30 million m^3 produced in the Mediterranean area (88, 89).

These effluents are well known for their significant negative impact on the environment because of their high organic load, including a significant amount of phytotoxic and antibacterial phenolic substances with resistance to biological degradation (90). Some European regulations allow spreading OMW onto agricultural fields, but this procedure has many limitations. In particular, it is not very suitable in regions where wet conditions following the harvest period do not require irrigation. Moreover, this practice is only allowed when there are no visible field slopes for obvious reasons of surface run-off (32). It was reported that over 90% of phenols in olives are transferred to the aqueous phase, i.e., OMW, during the pressing of the drupes.

The concentration of total phenolic compounds in OMW can be up to 10 g/L (33). Olive oil wastewater phenolics are likely to possess great variability depending on the olive variety, location and maturity level, as well as technological factors applied for virgin olive oil extraction (91). Due to the high concentration of phenolics, the olive mill byproducts could be conveniently converted into a valuable source of antioxidant compounds. Recovered antioxidants can be added to a variety of foods to preserve their quality and develop new functionality, e.g., improve nutritional properties or better resistance to lipid oxidation (20–22).

Several conventional (solvent, heat, grinding) and non-conventional methodologies (ultrasound, microwave, sub- and super-critical fluid extractions, pressurized liquid extraction, and gas-assisted mechanical expression) have been investigated for recovery purposes (92–94). Several studies reported on the recovery of phenolic compounds from OMW, as briefly described in the following sections, to give an overview on the most appropriate uses in the food industry. Solvent extraction methods include liquid-liquid or solid-liquid extraction and solid phase extraction (SPE) (37, 95). Lafka et al. recovered phenolic compounds from OMW using liquid extraction by means of several solvents, including supercritical carbon dioxide (CO_2) extraction. Obtained results showed that ethanol was the most appropriate solvent for the extraction of phenolic compounds from OMW. Supercritical CO_2 was confirmed to be an efficient solvent for recovering phenolic compounds with relatively high antioxidant activity from OMW (96).

In addition, Khoufi et al. reported that a liquid–liquid extraction method applied after aerobic or anaerobic digestion can allow a recovery above 90% of phenolic compounds using ethyl acetate (96).

According to different researchers, pressurized liquid extraction has been also applied for the recovery of phenolic compounds from OMW (97–99). Generally, this method is faster than traditional extraction techniques using a reduced volume of solvents.

Bouaziz et al. successfully used a mixture of ethanol and water (70:30, v/v) to extract PP from olive leaves. These results are therefore promising in terms of using food-grade solvents and then use of OMW phenols as food ingredients (99, 100). In addition, SPE is a relatively new procedure applied with excellent results for the extraction of natural PP (101, 102).

An integrated physicochemical biotechnological approach was described for the recovery of PP from OMW with a procedure of extraction in solid phase. Recovery yields were reported to be >60% (103, 104) in relation to similar procedures, reporting also that reversed phase solid phase extraction (RP-SPE) allowed the recovery of approximately 1 g of purified hydroxytyrosol per liter of OMW. These methods are generally effective for analytical purposes; however, they are quite expensive on the large scale, e.g., for applicative industrial uses, and one of main disadvantages is solvent residues in extracts. Several examples of successful application of membrane filtration for the separation and recovery of OMW phenolics can be found. A sequential combination of rough filtration, microfiltration (MF), UF, NF, and reverse osmosis (RO), was implemented by Villanova et al. for preparing tyrosol and/or hydroxytyrosol from olive mill wastewater (105). The process allows recovering, after concentration, at least 1 g/L of hydroxytyrosol and 0.6 g/L of tyrosol. These components can then be isolated with purity higher than 98% by inverted phase chromatography on a preparatory column. The process also allows the recovery of at least 70% of the water volume as to the starting total volume of the raw effluent, with a quality within the legal limits (lower than 100 mg O_2/L COD), which allows for its agricultural or civil reuse. In 2010, Galanakis et al. clarified olive mill wastewater by using four different UF membranes in the range of 2–100 kDa (37), showing that a 25 kDa membrane was highly efficient for the removal of heavier fractions ascribed to hydroxycinnamic acids and flavonols. By using this membrane, almost all of the initial phenolic compounds were separated and recovered in the permeate stream (10% retention).

Servili et al. applied an industrial plan based on an initial enzymatic pretreatment, followed by a three-phase membrane system (MF, UF, RO) for the recovery of OMW PP (34). The membrane treatment produced a crude phenolic concentrate with final volumes between 20 and 25% of the original OMW. Cassano et al. evaluated the efficiency of the OMW fractionation by means of a membrane integrated system for the recovery of low molecular weight (MW) phenolics. In particular, the process included an integrated membrane process based on the use of UF and NF membranes. Suspended solids were completely removed in the first UF step, whereas most of the organic compounds were removed in the following UF step. Phenolic compounds were recovered in the permeate stream of both UF processes (51).

Similarly, D'Antuono et al. applied membrane filtration to recover OMW phenolic compounds from two Italian and three Greek olive cultivars (106). Zagklis et al. characterized fractions obtained by a membrane process after using NF. Recovered phenolics were further treated with adsorption/desorption resins (107).

Garcia-Castello et al. analyzed the potential of an integrated membrane system for the recovery of phenolics from OMW through a combination of MF and NF membranes followed by a concentration step performed by using osmotic distillation (OD). In their study (108), almost all of the initial phenolics were recovered (319 mg/L) in the permeate of the NF step after a preliminary MF of raw wastewater devoted to the removal of suspended solids.

A concentrated solution containing about 0.5 g/L of low MW PP, with hydroxytyrosol representing 56% of the total, was obtained by treating the NF permeate by OD. Bazzarelli et al.

processed OMW through an integrated membrane process (MF and NF) and relatively new membrane operations such as OD and membrane emulsification (ME). In particular, the NF permeate (with phenolic content of 12.5 g/L) was concentrated by the OD unit, obtaining a phenolic-enriched OD retentate (87.5 g/L). A final ME step was used to encapsulate the final recovered product (encapsulation efficiency 90%). The MF process had a negligible rejection of rather phenolic compounds (about 6.8%) assuring their recovery in the permeate stream (109).

According to Arvaniti et al., UF provides a "clean" solution appropriate to feed next treatment processes based on the use of NF or RO membranes in the recovery of phenolic compounds from OMW. Ultrafiltration alone cannot isolate individual phenolic fractions; however, further purification with NF and/or RO membranes cannot be achieved without UF (110).

Standardized fractions enriched in phenolic compounds were obtained from *Olea europaea* L. tissues (leaves and pitted olive pulp) and *Cynara scolymus* L. byproducts (leaves and stems) through an environmentally-friendly process based on water extraction and membrane separation technology (111). In the investigated approach, a preliminary MF step was carried out with tubular titanium oxide (TiO_2)-made ceramic membranes to remove suspended solids improving the performance of the following steps performed with spiral-wound membrane modules in polyethersulfone (PES).

The control of fouling is one of the problems that still slows down large-scale membrane applications with respect to OMW management. Properly tailored pretreatment processes and the use of critical and threshold flux theories are important factors to ensure the cost-effectiveness of the membrane treatment when transferred to the industrial scale (112–114).

Fine modulo

Conclusions

The enormous quantity of food waste, food processing waste and food loss, can be effectively re-used for food and non-food purposes. The use of OMW extract in foods is a new trend in the food sector with the aim of formulating new products with a positive effect on consumer health. In this ambit, the usefulness of natural phenolic compounds is of great interest; consequently, many attempts and studies have been considered with the aim of obtaining two results: the safe treatment of wastewater from the food production chain, and the production of functional foods from OMW extracts. Because of the opportunity represented by the treatment of agro-industrial waste, different systems concerning winemaking and olive oil production industries have been also discussed as simple examples. More research is needed before considering recovery of phenolic compounds from wastewater as an economically convenient choice for the food sector. In fact, the importance of similar compounds is now recognized when speaking of hygienic issues and different therapies for human illnesses (114–119), and other arguments (120–133).

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Conflicts of Interest

The author declare that She has no conflicts of interest toward this work, and the views and opinions expressed in the text are those of the author, and not necessarily express those of the publisher.

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