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A Narrative Review of Extraction and Purification Methods for Polyphenolic Compounds Derived from Olive Oil Mill Wastewater

Ali Ammarlo [a, b] *, Arash Ghaitaranpour [a], Adel Mirza Alizadeh [c], Nesa Azimzadeh [c]

a. Department of Food Science and Technology, Faculty of Agriculture, Urmia University, Urmia, Iran.
b. Food and Drug Deputy, Zanjan University of Medical Sciences, Zanjan, Iran.
c. Department of Food Safety and Hygiene, School of Public Health, Zanjan University of Medical Sciences, Zanjan, Iran.

***Corresponding author:** Department of Food Science and Technology, Faculty of Agriculture, Urmia University, Urmia, Iran. Postal Code: 5756151818. E-mail: A.Ammarlo@urmia.ac.ir

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Olive oil production represents a major economic activity in Iran. The standard industrial extraction process involves three key steps: (i) crushing of olives to rupture cellular structures and release oil, (ii) malaxation to enhance oil yield through gentle mixing of the olive paste, and (iii) separation of oil from the aqueous and solid residues. In the three-phase centrifugation process, the addition of hot water facilitates oil separation but simultaneously generates large volumes of olive mill wastewater (OMWW) and solid waste compounds, particularly phenolic substances, making it one of the most environmentally problematic effluents in the agro-industrial sector. Despite these challenges, OMWW is also a rich source of bioactive compounds, including phenolics, tocopherols, and carotenoids, which exhibit antioxidant properties. The valorization of food industry wastes and by-products, therefore, serves a dual purpose: it ensures the effective utilization of waste streams while simultaneously promoting public health. This approach contributes significantly to the development of a sustainable food chain from both environmental and economic perspectives. Polyphenolic compounds recovered from OMWW show great potential for applications in the food, pharmaceutical, and cosmetic industries, thereby reframing OMWW from an environmental burden to a valuable resource. The aim of this review is to investigate various methodologies for the extraction and purification of polyphenolic compounds from wastes and by-products generated during the olive oil production process.

1. Introduction

In most Mediterranean countries, the production of olive oil is a significant economic sector and one of the oldest agricultural industries. Due to its superior nutritional qualities, olive oil has become more popular since 2005, and during the last ten years, its production has increased by about 40% globally (Dermeche et al., 2013). Three operational steps are typically involved in the extraction of olive oil: (I) crushing of olives to rupture cellular structures and release oil; (II) blending, in which the olive paste is gently mixed to maximize oil yield; and (III) separation of oil from the remaining waste. The final separation step can be carried out using different methods, most notably centrifugal extraction systems, which are classified into two main types: (1) the traditional discontinuous pressing process and (2) the two-phase or three-phase centrifugation systems (Figure 1) (Klen & Vodopivec, 2012).

Hot water is added during the centrifugation stage of the three-phase continuous process, which results in a higher olive oil wastewater (OOWW) production, typically ranging between 80 and 120 L per 100 kg of olives. The process is referred to as “three-phase” because, at the end of continuous centrifugation, three different phases are obtained: an oily phase (olive oil), an aqueous phase (OOWW), and a solid residue. Despite its high water

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consumption, the three-phase method is still used in certain countries, such as Portugal, Greece, and Italy, particularly in facilities where rapid production is prioritized (Dermeche et al., 2013). However, the two-phase system, which requires less energy and water, has largely replaced the three-phase process in major olive oil-producing countries such as Spain, where 90% of olive mills are two-phase operations. Developed in the 1990s, the two-phase system eliminates the addition of water, thereby reducing effluent volumes and generating two outputs: an oily phase (olive oil) and a semi-solid phase (wet pomace), commonly referred to as two-phase olive oil waste (Paulo & Santos, 2021). In general, two main types of residues are produced during olive oil extraction, regardless of the method used: (i) a liquid effluent, commonly known as "black water" generated in both the three-phase (OMWW) and two-phase (TPOOW) systems, and is known as and (ii) a solid residue, typically in the form of olive oil pomace. Due to its acidic pH, high organic load, and phytotoxic properties, including phenolic compounds, olive oil production wastewater is among the most hazardous wastewaters generated by the food and agriculture industries. It poses significant risks to aquatic ecosystems, soil, microorganisms, and air quality. From an environmental perspective, the magnitude of the problem is substantial; the volume of OMWW produced annually, approximately 10 million cubic meters, is equivalent to the domestic wastewater generated by about 20 million people (McNamara et al., 2008).

Consequently, the management and valorization of OMWW are considered a crucial environmental concern. Although numerous treatment methods have been explored to reduce its toxicity, recent research has increasingly shifted toward recovery and valorization approaches. OMWW can be regarded as a source of bioactive substances that can be extracted and utilized as natural antioxidants in the food, pharmaceutical, and cosmetic industries. These compounds include phenolics, tocopherols, and carotenoids (Dermeche et al., 2013).

Depending on the olive variety, climate, cultivation practices, and oil extraction method, OMWW's composition varies both qualitatively and quantitatively. Olive mill wastewater (OMW) contains a high concentration of mineral salts and organic compounds, including sugar, tannin, pectin, and phenolic substances (Carrara et al., 2021).

This article reviews the extraction techniques that have been investigated for the recovery of polyphenolic compounds from olive oil mill wastewater, focusing on the differences in these methods with respect to their processes, efficiency, and applicability .This review aims to investigate various methodologies for the recovery of polyphenolic compounds from wastes and by-products generated during the olive oil production process.

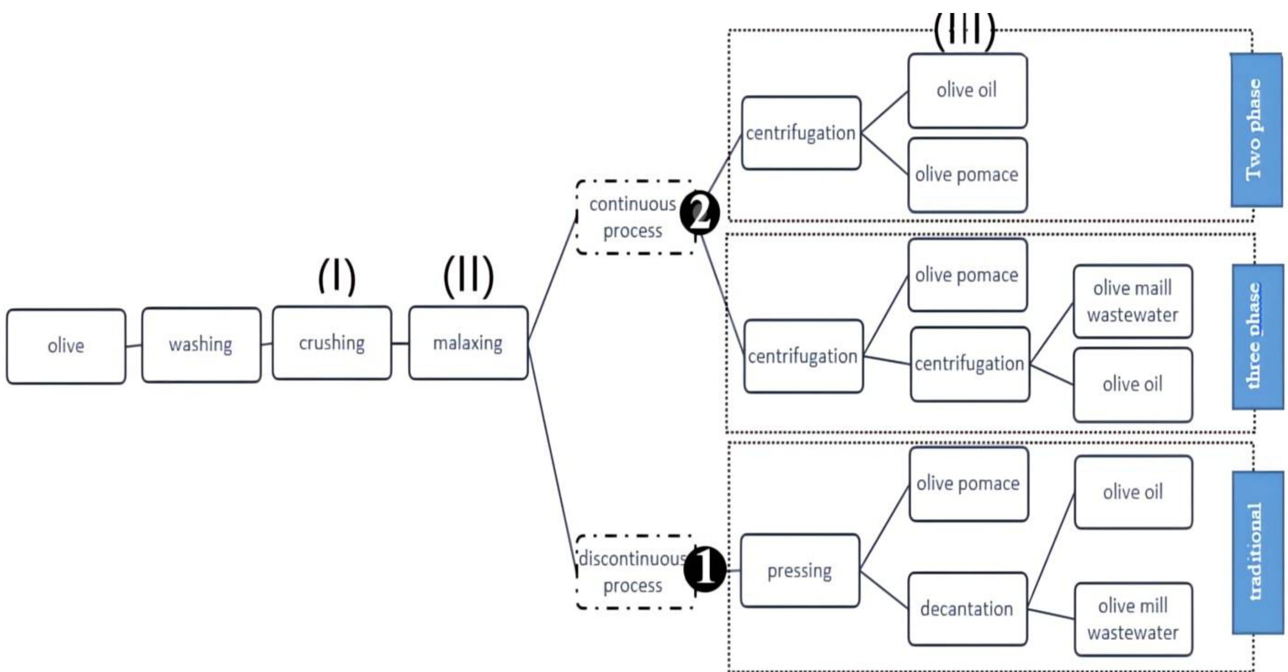

Figure 1. Diagram of the olive oil production process in different ways

1.1 Features of the wastewater from the olive oil mill

OMW has a pH of three to five, making it an acidic liquid. The most popular technique for obtaining antioxidant crude extracts from wastewater from olive mills is solvent extraction. The most effective organic solvent is ethyl acetate, or EtOAc (Kalogerakis et al., 2013). OMWW is essentially a complex mixture composed of vegetable juice, soft tissues of the olive oil fruit (e.g., mucilage and pectin), and process water, which is centrifuged during oil extraction. It is characterized by a dark, almost black coloration and a distinctive, strong odor. Due to the content of organic acids, namely malic and citric acids, the pH of OMWW typically ranges from 2.0 to 6.0 (Table 1). The effluent almost demonstrates a high electrical conductivity of 16.79 mS/cm, attributed to the presence of phenolic acids and fatty acids. The high conductivity can be explained by the high content of organic compounds in the form of salts. Dry matter (6-17%) and organic matter (4-16%) make up the majority of wastewater. In addition, it contains tannins, polyalcohols, minerals, pectins, lipids, and reducing sugars, primarily glucose (90%) and fructose (10%). Because phenols are highly hydrophilic and come into contact with oil during the extraction stage, OMWW has a higher concentration of potassium and significant levels of nitrogen, phosphorus, calcium, magnesium, and iron when compared to other organic wastes (Foti et al., 2021).

Table 1. Physico-chemical characterization of the raw OMWW collected from an olive oil processing plant adapted from (Pinto et al., 2025)

Physico-Chemical	OMWW Sample
PH	6.5 ± 0.10
EC (mS cm-1)	0.7 ± 0.003
COD (mg L-1)	1504 ± 350
BOD5 (mg L-1)	35 ± 3
TSS (mg L-1)	222 ± 0.6
N t (mg L-1)	439 ± 7
P t (mg L-1)	84.3 ± 4.2
DO (%)	5.6 ± 0.8
Alkalinity (CaCO3 mg L-1)	0
Turbidity (NTU)	417 ± 5
N-NH 4 (mg L-1)	234 ± 1
N-NO3 (mg L-1)	2.5 ± 0.1
N-NO2 (mg L-1)	0.018 ± 0.00
Phenols (mg GAE. L-1)	56 ± 6
Fecal Coliforms (Nº/100 mL)	93 ± 2
E. coli (Nº/100 mL)	9.1 ± 0.2
Parasite eggs (Nº/L)	9.1 ± 0.1

1.2 Features of microbiology

The ripeness stage of the fruit and the olive variety have the greatest influence on the microbial flora in OMWW, although additional factors also play a role. The microbial density of OMWW is highly variable, typically ranging from 105-106 CFU/mL (CFU: Colony Forming Unit), and consists primarily of molds, bacteria, and yeasts. The yeast community is mainly represented by species belonging to the genera *Pichia, Candida,* and *Saccharomyces* (Bleve et al., 2011). More than one hundred fungi have been identified in OMWW, predominantly from the genera *Acremonium, Alternaria, Aspergillus, Bionectria, Byssochlamys, Chalara, Cerrena, Fusarium, Lasiodiplodia, Lecythophora, Paecilomyces, Phenicilliaecom, Phenicilliae,* and *Scopulariopsis*. Furthermore, several studies have reported that the culturable bacterial community is relatively limited, including *Firmicutes*, *Actinobacteria*, *Alphaproteobacteria*, *Betaproteobacteria*, and *Gammaproteobacteria* (Ntougias et al., 2013).

1.3 Bioactive properties

Considering the Food and Drug Administration (FDA) in the United States has designated olive pulp extract as GRAS (Generally Recognized as Safe (GRN No. 459), adding phenol from OMWW to food is an intriguing way to both increases the nutritional value of the food and prolong its shelf life., and as an antioxidant with a final concentration of up to 3 g/kg, it can be found in cooked products, beverages, cereals and sauces, condiments and snacks (Figure 2) (Foti et al., 2021). Figure 2 Schematic representation of the application of polyphenolic compounds recovered from olive oil mill wastewater in food products, including dairy matrices such as milk, where they contribute to improving oxidative stability, inhibiting microbial growth, and extending product shelf life.

1.4 Phenolic substances

Consuming olive oil has been linked in numerous studies to a number of health benefits, such as lowered risk factors for coronary heart disease, prevention of atherosclerosis and other chronic diseases, cancer, chronic inflammation, stroke, and other illnesses. Partially responsible for these advantageous health effects are phenolic compounds (Servili et al., 2013). The most prevalent phenolic compounds (PCs) in olive factory wastewater are oleeuropein, hydroxytyrosol (HTyr), and tyrosol, with HTyr being the most active. Therefore, theoretically, oleuropein′s enzymatic hydrolysis or degradation causes the molecule to cleave, resulting in the formation of HTyr and tyrosol byproducts (Çelik et al., 2021). OMWW contains phenolic compounds with potent biological activity. The European Food Safety Authority acknowledges hydroxytyrosol as a blood lipid oxidation protector. Its impact on disease prevention has been demonstrated in a number of studies carried out in animal models or in lab settings (Achmon & Fishman, 2015). In vitro, hydroxytyrosol has also been demonstrated to have antimicrobial effects against both gram-positive and gram-negative bacteria (Obied et al., 2007). Furthermore, bioavailability studies have demonstrated the efficiency of human absorption of hydroxytyrosol and olive oleuropein (De Bock et al., 2013), which supports their inclusion in the diet and consequently their extraction from OMWW, which can be a cheap and abundant source of phenols. Consequently, it has been demonstrated that the wastewater from olive mill is a potent source of antioxidant compounds (the ORAC (free radical scavenging ability) activity of olive hydroxytyrosol is 10-15 times higher than that of vitamin C, quercetin, or vitamin E). and The reason for this fact is that most of the phenolic compounds remain in the waste during the extraction of olive oil because OMWW is polar while olive oil is non-polar (Priego-Capote et al., 2004).

1.5 Other uses of olive oil industry waste

Because olive kernels have a high heating power (17.16 kJ/energy), they are traditionally used to produce heat or electricity. Furthermore, olive kernels are utilized as heavy metal ion biosorbents to create activated carbon, which can be utilized to eliminate pollutants, flavors, odors, and dyes. Similar to formaldehyde, olive kernels have also been utilized to make wood glues. Conversely, non-renewable resources are mostly used to make synthetic adhesives (e.g., oil and natural gas). Because fossil resources are becoming scarcer and more expensive, it is critical to find new ways to produce natural adhesives. In the meantime, formaldehyde is regarded as a priority pollutant by the US Environmental Protection Agency due to serious safety concerns about worker exposure in manufacturing facilities and its release into the environment. When comparing the usefulness of polyphenolic compounds derived from olive factory wastewater to other natural antioxidants, it is important to note that the primary causes of the restricted application of natural antioxidants are their high concentration requirements, disagreeable tastes and smells, and processing-related property loss. Because of the disadvantages mentioned above, synthetic chemical compounds with greater antioxidant activity, stability, and availability (propyl gallate (PG), butylated hydroxyanisole (BHA), and butylated hydroxytoluene (BHT) occasionally replace them (Peschel et al., 2006).

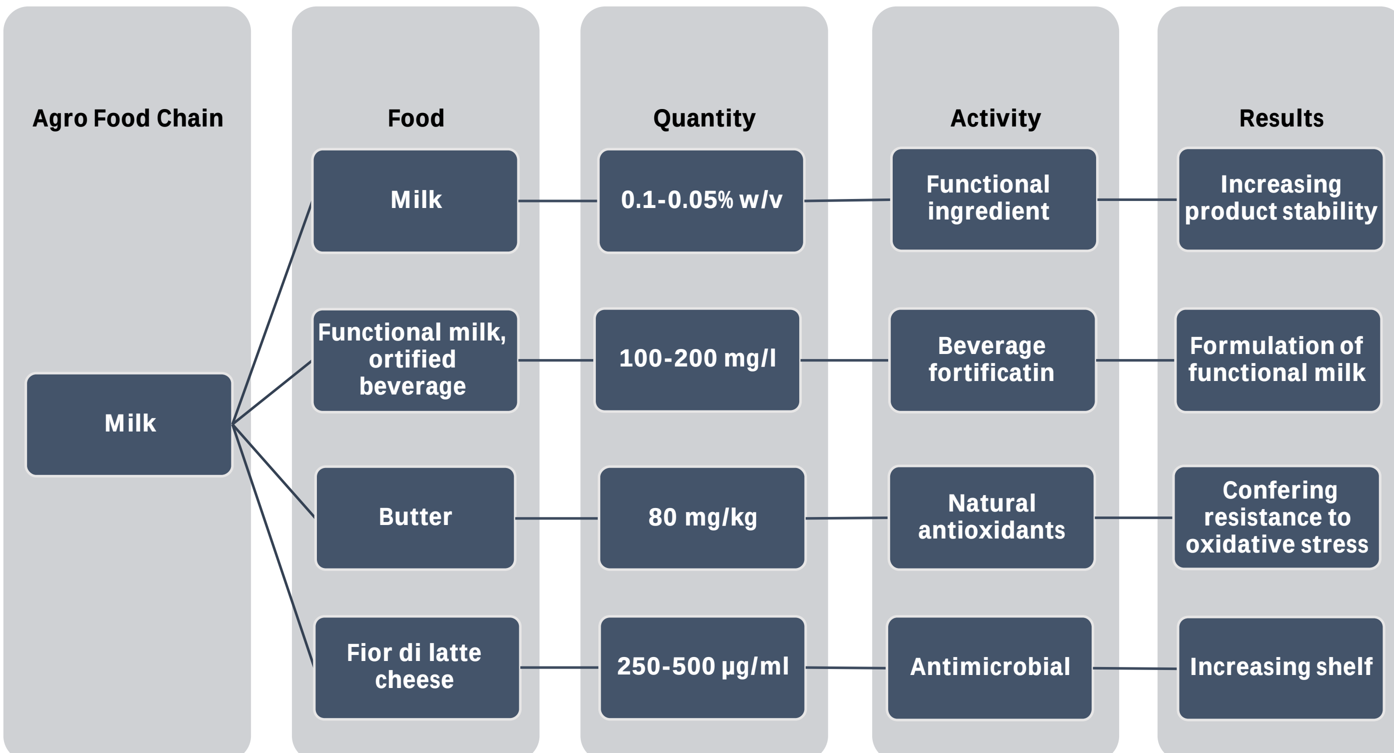

Figure 2. Uses of olive OMWW in the food chain (Foti et al., 2021)

1.6 Extraction of phenolic compounds

Depending on the kind of by-product (waste, waste water, olive leaf, etc.), different extraction methods are employed to extract phenolic compounds. When using extraction techniques to produce extracts high in phenolic compounds, there are three important factors to consider before extraction: (1) sample preparation techniques; (2) selecting the best solvent; and (3) selecting the best extraction technique. In order to extract phenolic compounds from wastewater, it is important to keep in mind that the methods for obtaining polyphenols from agricultural waste involve a number of related steps, such as the following: (1) Preparing the polyphenols; (2) Treatment of extracts produced by membrane technologies; (3) Selective extraction of polyphenol mixtures or other constituents; (4) The last steps in the process of lyophilization, or freeze-drying, are to create powder.

1.7 Sample Preparation

The sample preparation procedures are regarded as a crucial stage since they take into account the physicochemical modifications to the phenolic compounds′ chemical structure. In light of this, olive oil by-products (OOBP) samples ought to be freeze-dried and kept in conditions free of moisture or kept in a liquid nitrogen environment after collection. Because alkaline environments promote oxidation and polymerization reactions, an alternative method to the one described above would be to acidify the sample of olive oil by-products to a pH of 2-3. On the other hand, low pH can precipitate proteins, which increases the amount of free phenolic compounds in the extraction solvent. Due to the olive oil products (OOP′s) high-water content (roughly 60%), during sample preparation, it can be sent through a centrifugation process to separate the solid residues from the water. These waters are made up of the water in olive fruits and the water used to make olive oil. It should be noted that it is common to remove the lipid fraction of OOBP samples before extraction. Nevertheless, it is even possible to separate the lipid fraction after the extraction of phenolic compounds. In this case, a methanol/water solvent mixture is usually used. Non-polar substances like vitamins, fat-soluble pigments, triacylglycerols, and fatty acids may make it difficult to quantify the phenolic compounds in the extract that is produced. Because of this, n-hexane or petroleum ether is used as a solvent to extract non-polar compounds. The phenolic profile of a sample of fatty olive oil by-products may differ from the profile of phenolic compounds in the extract taken from a sample of non-fat olive oil factory by-products. Generally speaking, defatted samples exhibit higher concentrations of phenolic compounds. This is most likely because of the interaction between the phenolic compounds and the fats in the sample of olive oil byproducts (Obied et al., 2007). Because of this, it is advised to prepare a sample devoid of fat before extracting phenolic compounds.

1.8 Common methods of extraction

Polyphenols from olive oil and its byproducts can be recovered using a number of methods that have been suggested. From an industrial perspective, the most feasible method for removing bioactive compounds from plant-derived matrices is conventional solvent extraction, also known as solid-liquid extraction (SLE) or soaking (Montenegro-Landívar et al., 2021). Typically, the solid-liquid extraction method begins with homogenizing or soaking the OOBP sample. OOBP sample homogenization is typically accomplished by magnetic stirring, vortexing, or the use of high-performance liquid homogenizers like the Ultra-TurraxVR. Centrifugation or sedimentation comes next to homogenization. The analytes are then separated into two phases-a liquid and a solid-based on their polarity, or how well they bind to the additional organic solvent. The two phases are then separated, allowing for the recovery of the analytes. Selecting a solvent is an important first step. The intended end use, extraction efficiency, and analytes to be recovered should all be taken into consideration when choosing it. This is particularly crucial if the extract is going to be used in food applications. Generally recognized as safe (GRAS) solvents are used to extract phenolic compounds from OOBP. Because of their high selectivity for phenolic compounds, solvents like methanol, ethanol, and even hydroalcoholic mixtures are used in this field. Nonetheless, methanol is the most frequently utilized method for recovering phenolic compounds from byproducts of olive oil (Klen & Vodopivec, 2012).

On the other hand, EU Regulation 2009/32/EC caps its concentration in food matrices at 10 mg/kg (EC 2009). Ethanol is regarded as an extraction solvent in accordance with EU Regulation 2009/32/EC and good manufacturing practices. Therefore, when it comes to phenolic compound extraction from OOBP, ethanol is a safer substitute for methanol. However, keep in mind that ethanol is more lipophilic than methanol when selecting a solvent. As a result, analytes recovered using ethanol may be more structurally complex and lipophilic. Water and ethanol-based systems are typically the preferred systems for use in food, pharmaceutical, or cosmetic applications (Tapia-Quirós et al., 2022).

Ethanol (EtOH), which recovers approximately 95% of the phenolic compounds from OMWW, can be utilized for this purpose. In these circumstances, achieving high extraction performance requires a ratio of 1.5 (v/v) solvent to sample, which makes the process uneconomical. Furthermore, combining water and EtOH reduces the extractive strength′s selectivity. A different technique used in this field is called aqueous two-phase extraction (ATPE), which is based on the liquid-liquid phenomenon and can remove most proteins and other carbohydrates as well as other components of the olive factory effluent. Under some circumstances, the aqueous two-phase system (ATPE) experiences the desalination effect of inorganic salt additions, which results in the formation of two phases. Furthermore, this extraction system can recover mineral salt or ethanol, making it a cost-effective and environmentally friendly method.

In order to increase the effectiveness of later steps that concentrate on phenolic antioxidants, previous studies have used a multi-step process that starts with lowering the water content of OMWW in a pilot-scale mechanical steam recompression (MVR) evaporator. In this instance, there would be a reduction in the amount of solvent needed for extraction. This method involves separating the residual oil in OMWW, extracting phenolic antioxidants using ATPE based on EtOH/mineral salt under varying conditions, and then using nonionic polymer resins to maximize the purification of the polyphenolic compounds that are already present. This procedure, which consists of four steps, concentrates on phenolic compounds by employing the MVR evaporator to remove excess water. To remove any remaining oil in the OMWW, the olive mill wastewater concentrates (OMWC) is pretreated in a second step using hexane and acid (0.1% HCl). The next stage involves using ATPE to extract phenolic antioxidants from pretreated OMWCs. It is significant to note that there is evidence that OMWW contains two soluble and insoluble forms of olive biophenols, which may contribute to wastewater acidification. Hence, through the hydrolysis of complex soluble phenols (such as oleuropein′s hydrolysis to HTyr) as well as insoluble forms that bind polymeric organic matter to suspended particles, acidification of OMWW increases the extractable biophenolic content. Acidic environments can cause the precipitation of proteins and dietary fibers like pectin in the matrix, in addition to the release of free phenols. Thus, the extraction of extractable polyphenolic compounds can be made easier by the advantageous effect of acidification. Additionally, by preventing their fermentation, acidification can stabilize low molecular weight biophenols in plant juices.

Ammonium sulfate ($(NH_4)_2SO_4$) is employed as a desalination chemical in the ATPE method′s continuation to extract EtOH from the aqueous phase. Note that to improve the extraction efficiency, solvent extraction is assisted by ultrasound (Sonorex Super RK 510-Bandelin, 35 kHz) for 10 minutes, depending on the content of the wastewater particles. Next, the solvents are eliminated using a Heidolph Laborata 4000 rotary evaporator set to negative pressure and 40°C. It is evident, however, that SLE in combination with EtOH greatly enhances TPC extraction. Conversely, unlike the SLE system, EtOH in ATPE can concentrate and purify the OMWC matrices by splitting the system into two phases.

When compared to solvent use, ATPE also has an extra advantage: 20% less solvent is needed during ATPE to extract phenolic compounds per gram of concentrate than is needed during SLE with EtOAc. Thus, ATPE performs favorably overall when compared to other applied SLE methods.

1.9 Purification after conventional methods

Processes of absorption and purification hold significant importance in addition to solvent extraction in the recovery of phenolic antioxidants from OMWW. When it comes to extraction, polymeric adsorbents are more helpful than activated carbon, which might not be able to recover enough phenolic compounds because of their strong adsorption. Nonionic resins, specifically XAD16, typically perform better than other types of polymeric resins, although a variety of polymeric resins have been employed in research to selectively recover and purify phenolic compounds from OMWW. The majority of the unwanted fractions of OMWW are eliminated by the combined use of polymer resins, such as XAD16 and XAD7HP, in addition to the individual applications of these resins. The majority of the two-phase aqueous extracts in the studies were purified with Amberlite XAD16N resin. Washing the biophenols loaded onto the resin with multiple solvents in succession and generally at a low pH can improve the process's purification performance. But increasing the selectivity towards phenolic compounds by eliminating other fractions from OMWW is crucial for an effective recovery process, and this can be done by combining subsequent finishing steps (Çelik et al., 2021).

The separation of phenols from proteins and remaining carbohydrates in the extracts is made possible by a two-step resin washing procedure. XAD16N exhibits a quick adsorption tendency for TPC by ATPS extraction, achieving 76-78% adsorption in 15 minutes. Since it is known that the adsorption ratio significantly decreases as the initial concentration of phenol increases, the difference in adsorption ratios during the purification process may be related to the initial concentrations of phenol in the extracts loaded onto the resin. Therefore, after obtaining full absorption of TPC in the extracts on the resin, it is preferable to carry out sequential washing with water and acidic EtOH. It is crucial to bear in mind that, in the event of complete purification via the resin extraction procedure, accounting for TPC and HTyr recovery as well as the effectiveness of protein and carbohydrate removal, according to a previous study, washing the resin with ordinary water resulted in only a limited loss of TPC and HTyr adsorbed on XAD16 (Çelik et al., 2021).

Additionally, Frascari et al. (2019) employed one ion exchange resin (Amberlite IRA958 Cl) and four neutral resins (Amberlite XAD16N, FPX66, XAD761, and Optipore SD-2) in their investigation to determine the best adsorbent for phenolic compounds (PC) from Tunisian OMW. For phenolic removal, Amberlite XAD16N and Amberlite IRA958 are the most promising adsorbents. Due to lower industrial costs, Amberlite IRA958 is preferred even though Amberlite XAD16N demonstrated the highest adsorption capacity (81 mgPC/gdry resin).

1.10 Modern extraction technologies

The increasing need to employ more effective recovery techniques led to the development of new, unconventional techniques in addition to the traditional and widely used ones. By lowering extraction times, process temperatures, and solvent usage, these innovative techniques can increase extraction efficiency while consuming less energy. Ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), pressurized liquid extraction (PLE), enzyme-assisted extraction (EAE), ohmic heating (OH), and extraction using pulsed electric fields (PEF) are a few of these related technologies (Pappas et al., 2021). A study by Dermeche et al. (2013) focused on the extraction of high-value compounds, such as phenolic extracts, from OMWW using a variety of techniques, including enzymatic and chromatographic methods, solvent extraction, and membrane processes such as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis.

A number of factors should be taken into account in addition to the extraction yield, capital and operating costs, environmental concerns, exploitation time, and the encouragement of industrial-scale exploitation when choosing the best new extraction technique among the available techniques. Conventional extraction is widely recognized for its ease of application in industry, with the primary limitations being solvent consumption and extraction time. In this regard, modern technologies are far more appealing since they increase yield while drastically reducing solvent consumption and extraction time. When using contemporary techniques, it is even feasible in some circumstances to think about combining multiple techniques continuously or irregularly, such as PEF-UAE, UAE-SFE, UAE-MAE, or EAE-PLE (Belwal et al., 2020). Given that the transition from laboratory to industrial scale is a significant matter. Therefore, UAE or SFE would be the most advanced techniques.

1.10.1 Extraction with the help of ultrasound (UAE)

High-frequency waves (2 MHz) are used in ultrasound-assisted extraction to produce negative pressure, alter the medium's physicochemical characteristics, and facilitate the formation of cavitation bubbles. The samples' cell membranes breaking down is caused by this phenomenon, which also increases the solvent's and matrix's surface contact. Cavitation enhances heat and mass transfer through cell wall rupture, which is the reason for the growing use of this technique (Barba et al., 2016). The high extraction power of ultrasound is also a result of the oxidative energy of radicals produced during solvent sonolysis (hydroxyl and hydrogen peroxide for water) (Chemat, 2011).

The benefits of this technology for extracting natural compounds are numerous. More solvent penetration into cellular materials; shorter processing and shelf life; improved product yield and repeatability; lower solvent and emulsifier consumption; high processing power; decreased noise; extraction of heat-sensitive components; and substantial maintenance savings are just a few of the advantages it offers. Reduced processing energy needs, and ultimately, based on the aforementioned information, processing that is cheaper overall and more environmentally friendly (Roselló-Soto et al., 2015). Twenty phenolic compounds were recovered and identified in a study that tested the extraction of polyphenols from olive factory wastewater using ultrasound (20 kHz/450 W/5-10 minutes) (Priego-Capote et al., 2004).

However, because phenolic compounds are antioxidants, the radicals produced during the ultrasound treatment have the potential to degrade them; therefore, the extraction conditions need to be optimized to reduce this possibility. Thus, a number of circumstances were examined, and the following was determined to be its optimal state: 10% to 50% duty cycle, 2 mL/min extractor flow rate, and the radiation range. 3 mm is the probe position. 13-minute radiation time Volume and composition of the extractor: 6 mL, and the proportion of methanol to water is 75:25. Tyrosol, catechin, and tannic acid are the compounds present in higher concentrations, and this extraction method produced a high content of phenolic compounds (8680 mg/g) (Figure 3). The polarity of olive factory wastewater, as opposed to the non-polar nature of olive oil, which permits the majority of the phenolic compounds to remain in olive oil, is why the authors demonstrated that olive factory wastewater is a potent source of antioxidant compounds (Priego-Capote et al., 2004).

According to (Klen & Vodopivec, 2011) the effects of ultrasound (30 kHz / 100 W / room temperature / 5-20 minutes) on the recovery of total phenolic compounds from olive factory wastewater were assessed using liquid/liquid extraction, solid phase, and conventional filtration techniques. Stirring was contrasted. According to Klen & Vodopivec (2011), ultrasound under ideal conditions (30 kHz / 100 W / room temperature / 15 minutes) produced a higher yield of both specific and total polyphenols.

1.10.2 Supercritical Fluid Extraction

The application of supercritical fluid technology (SFT), a green technology that eliminates the need for organic solvents, is growing in popularity but is getting less and less attention because of environmental and health concerns. Supercritical fluids are created when the temperature and pressure of a liquid or gas are raised above the critical point. These fluids are excellent, clean solvents that are perfect for recovering valuable compounds from plant matrices because of their gas-like permeability and liquid-like solvent power. Carbon dioxide (CO_2) is the most widely used supercritical fluid because it is readily available, non-toxic and flammable, inexpensive (in comparison to liquid organic solvents), has tunable solvent properties, and is easy to control at critical temperature and pressure (31.1 °C and 7.38 MPa). Supercritical CO_2 (SCeCO_2) also has low surface tension, high molecular diffusion, and a relatively low viscosity. Mass transfer is enhanced by each of these characteristics. Furthermore, lowering the CO_2 pressure extracts the gas from the product and yields an extract free of solvents (Lafka et al., 2011). The selectivity of supercritical fluid extraction can be altered by varying the density, which in turn affects the pressure and temperature. The polarity can be changed by adding a co-solvent, such as water, ethanol, methanol, hexane, acetone, or chloroform. This can also alter the extraction's selectivity. An illustration of a pilot-scale SFE system with a solvent pump is shown in the figure below (Figure 4). This system enables the extraction of high-value compounds (like polyphenols) from plant matrices like olive waste and by-products.

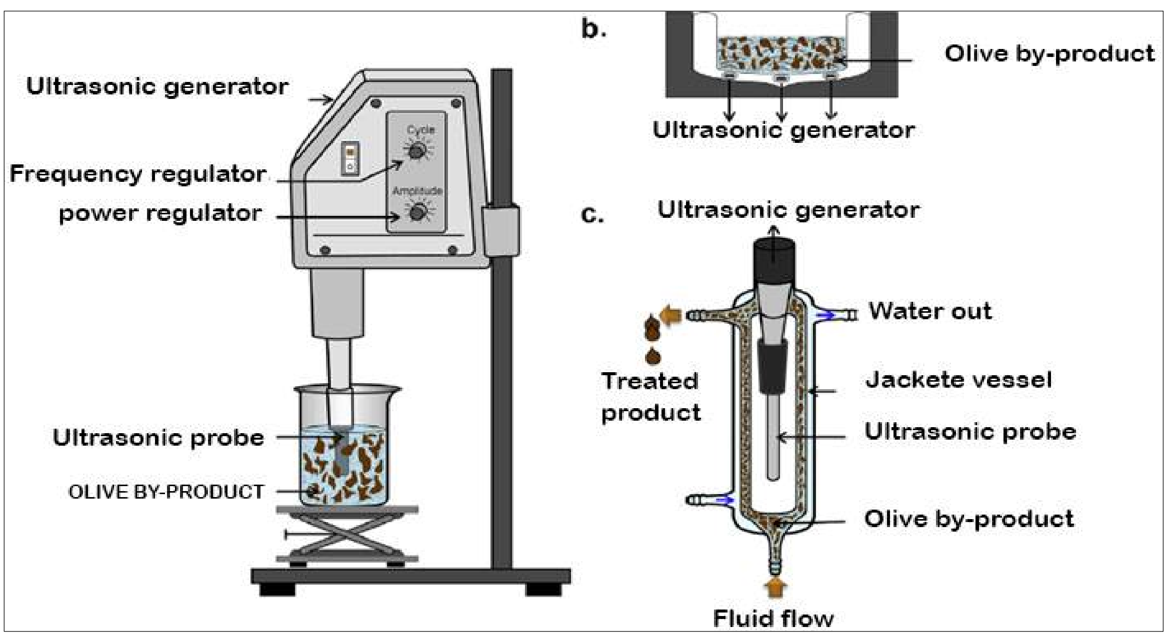

Figure 3. Schematic representation of ultrasonic systems (a), Ultrasonic bath rod type probe system (b), Continuous probe system used for liquid food processing (c) (Roselló-Soto et al., 2015)

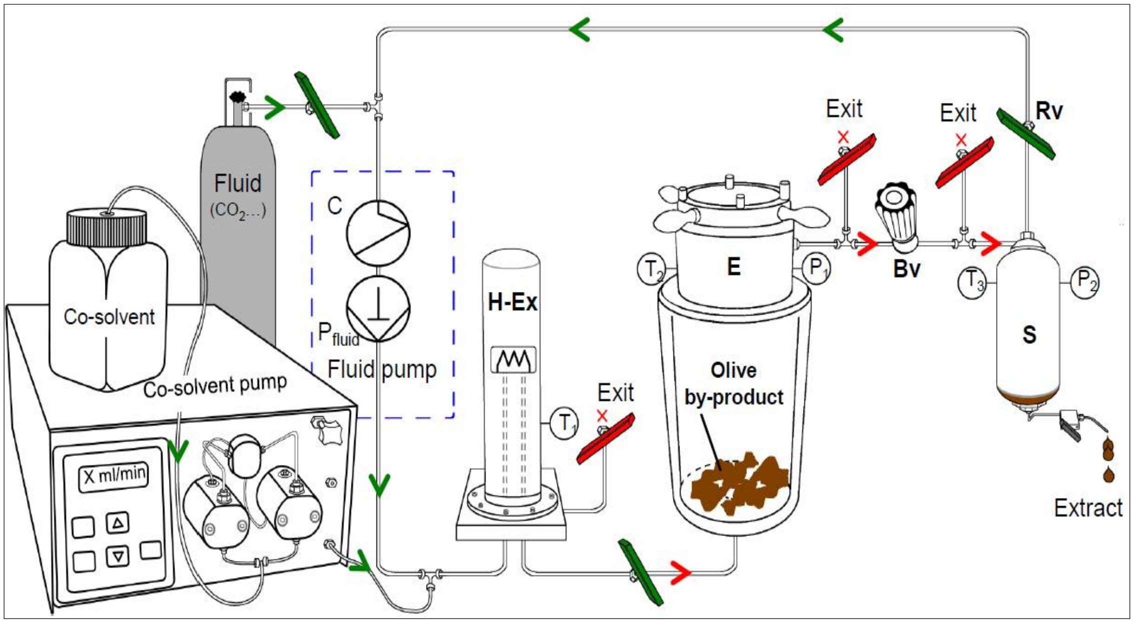

Figure 4. Schematic setup of a laboratory scale for high-added-value compound extraction using supercritical fluids and co-solvent. C: condenser, H-Ex: heat exchanger, E: extraction, Bv: back pressure valve, Rv: return valve, S: separator, P: pressure sensor, T: temperature sensor (Roselló-Soto et al., 2015)

Lafka et al. (2011) describe a comparison between $SCeCO_2$ and conventional organic solvent extraction for the recovery of phenolic compounds from OMWW. The ideal conditions for polyphenol solvent extraction, according to the authors, are 180 minutes with ethanol, a solvent-to-sample ratio of 5:1 v/w, and a pH of 2. The percentage of phenolic content, expressed as caffeic acid equivalents on a dry basis (% w/w), varied between 0.43% and 1.29% when extracted using solvent and 0.76% when extracted using SCeCO2 (Oliveira & Oliveira, 2000).

1.10.3 Pressurized liquid extraction

Pressurized liquid extraction (PLE) is regarded as a sophisticated extraction method that makes use of unique extraction conditions. During the extraction process, extracting solvents are heated to high temperatures and kept under high pressure to preserve their liquid state (Oliveira & Oliveira, 2000). This results in improved analyte solubility, enhanced water diffusion, and decreased water viscosity, which enhances penetration into the matrix and boosts mass transfer rate, ultimately improving extraction efficiency (Teo et al., 2010). As a result, this extraction method offers a number of benefits, including increased selectivity, a shorter extraction time, and a reduction in the amount of hazardous organic solvents (ethanol and water are the most often used solvents). Furthermore, this extraction method offers a controlled extraction environment and lower operating costs, giving the extract a specific composition both in terms of quality and quantity (Herrero et al., 2011).

Because these non-conventional extraction technologies operate in different ways-such as faster and non-thermal conditions-and have different operating modes that allow for better and novel properties to be produced at lower costs and with less of an impact on the environment. Subcritical fluids are pressurized fluids that are utilized in the recovery of valuable compounds. Subcritical fluids are actually created when a parameter, such as temperature or pressure, is raised above the critical point. Water is the best solvent for the industrial extraction of high-value compounds from plants because it is inexpensive and eco-friendly. However, its use is restricted because of its low extraction efficiency for the majority of organic compounds. Subcritical water, whose chemical characteristics are comparable to those of organic solvents, is used to achieve advanced water extraction properties, such as polarity, surface tension, viscosity, and dissociation constant. Water is actually regarded as sub-critical at critical points when one of the parameters (temperature or pressure) falls below 374 degrees Celsius or 22.1 MPa. Consequently, polar (at lower temperatures) and non-polar (at higher temperatures) organic compounds can be dissolved in subcritical water. The following list includes some of the most significant uses of unconventional technologies for the extraction of highly valuable compounds from olive waste and by-products.

1.11 Purification

High-complexity extracts are produced during the extraction process; therefore, these extracts must go through the purification step, which can be completed with the aid of a membrane (Figure 5). Separation technology, absorption-rejection technology, or a mix of the two to produce a higher-quality, polyphenol-rich end product that can be utilized in the food, cosmetic, and pharmaceutical sectors. Three primary objectives can be achieved through large-scale membrane separation processes: Using pressurized membrane techniques, such as microfiltration (MF 0.1-5 m, 1-10 bar) and/or ultrafiltration (UF 0.5-100 nm, 1-10 bar), suspended and colloidal materials are removed during the extraction phase. Reducing the volume of polyphenol-rich streams using pressurized membrane techniques like reverse osmosis (RO < 0.5 nm, 35-100 bar) and/or nanofiltration (NF 5-10 nm, 10-30 bar) and/or ultrafiltration (UF) (Zagklis & Paraskeva, 2020). Selective separation of polyphenols from other families of co-extracted compounds (e.g., sugars, proteins, lipids).

Membrane technologies offer several benefits, such as minimal energy and temperature requirements, excellent separation efficiency, easy scalability, straightforward equipment, high permeate flux efficiency, and lack of phase transition (Cassano et al., 2017). The functional properties of the desired compounds are preserved because membrane processes, as opposed to traditional concentration methods (evaporation, spray drying, etc.), do not subject the product to high temperatures or alter the solvent′s physical state (Bazinet & Doyen, 2017).

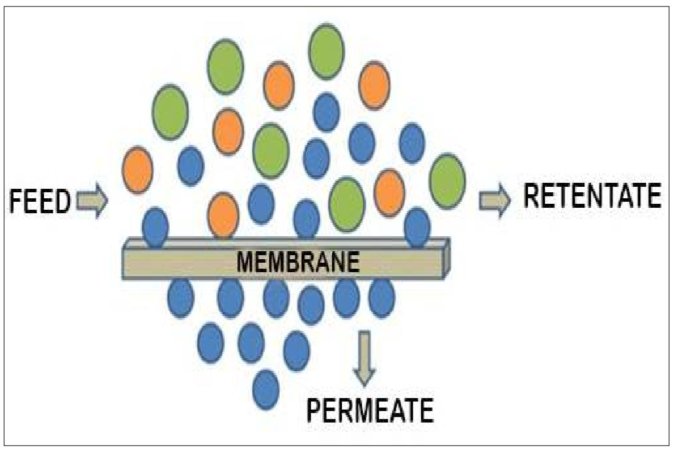

Figure 5. A view of purification using a membrane

The recovery of treated polyphenol streams and high-quality water for subsequent use is made possible by the integration of membrane processes. In earlier stages, MF is used to eliminate suspended solids and stop membranes from becoming initially fouled. UF membranes distinguish between the macromolecules that stay in the chamber, such as sugars, lipids, and proteins, and the polyphenols in the permeate stream. The concentration and selective recovery of low molecular weight polyphenols can also be achieved with NF. Finally, according to (Castro-Muñoz et al., 2016), reverse osmosis (RO) can be utilized for purification. It has also been demonstrated that membrane separation techniques are an appropriate pretreatment step for polyphenol recovery from olive mill residues (Kontos et al., 2016). The table below (Table 2) shows instances of the use of various membrane techniques for polyphenol recovery from olive mills (Tapia-Quirós et al., 2022).

For instance, Zagklis and Paraskeva (2020) proposed a treatment plant design to recover polyphenols from olive leaves and wastewater from olive mills. Their process was based on the integration of UF, NF, and RO techniques followed by a final adsorption step. The authors reported polyphenol concentrations of 378 g GAE L-1 and 98 g GAE L-1, respectively. Polysaccharides and polyphenols can be effectively separated using UF and the NF membrane, which rejects polyphenols by over 92% and produces concentrated solutions with strong antioxidant activity. According to Ianni et al. (2021), a three-step membrane filtration process (microfiltration, ultrafiltration, and reverse osmosis) is a technologically sound way to extract and purify OMWW phenols for use in subsequent field applications. It can be utilized in cosmetics, food, or medication.

Table 2. Instances of the use of various membrane techniques for polyphenol recovery from olive mills

Sample	Technique	Polyphenols Concentration
OMWW	MF, UF, and NF	2456 to 5284 µg mL-1 (MF) 1404 to 3065 µg mL (UF) 373 to 1583 µg mL (NF)
OMWW	MF and UF	7.2 g L-1 of hydroxytyrosol -1
OMWW	MF, UF, and NF	250, 250, and 430 mg in the NF retentate of the first, second, and third treatment, respectively
Olive pomace	NF and RO	1063.9, 1069.4 and 1234.3 mg GAEL-1 for NF270, NF90 and BW30, respectively
Green leaves, dried leaves, and pitted olive pulp	MF, UF, NF and RO	244.15, 57.63, and 289.93 mg g for green leaves, dried leaves, and pitted olive pulp, respectively
OMWW, grape marc, and olive leaves	UF, NF, and RO	378, 98, and 190 g GAEL-1 for OMWW, olive leaf extract, and grape marc extract, respectively

1.11.1 Some applications of extracted polyphenols in the food industry

In a review, they gathered information on the addition of OMWW extracts to enhance meat and meat products; the findings indicated that the obtained antioxidants improve the health conditions and rheological characteristics of the final products. Olive phenolic compounds have shown better performance in the treatment of raw meat because they were able to prevent lipid oxidation. A study by Galanakis (2018) reported the incorporation of olive oil by-products as concentrates or ingredients in the formulation of novel foods across various agricultural and food supply chains. According to a study by Veneziani et al. (2017), polyphenols extracted by OMWW in fermented sausages and white meat hamburgers enhance quality metrics and lengthen their shelf life. In particular, the addition of extracts prevented the growth of fungi and the germination of spores in fermented sausages. Additionally, this group assessed how polyphenols from OMWW in PVC-wrapped white meat hamburgers improved the burgers′ sensory and health attributes and demonstrated how adding phenolic extract at various concentrations (0.75 and 1.50 g/kg) inhibits the growth of aerobic mesophilic bacteria.

Using phenolic extract added at 0.1 and 0.05% w/v (Troise et al., 2014), tested the antioxidant activity of OMWW phenolic extract on the inhibition of the Maillard reaction

(MR), which reduced the formation of Activated carbonyl in heat treatment, showing more stability without any detrimental sensory effects of the samples. The butter formula was used to test the addition of OMWW extract and olive pomace at various concentrations (2, 4, 6, and 8 mg/100 g butter) (Mikdame et al., 2020). The results indicated that the highest concentration led to resistance. In opposition to oxidative stress. It prevents the growth of yeast, mold, Staphylococcus aureus, and total coliform when stored at 25°C for three months. In their study, Galanakis (2018) investigated the antimicrobial and antioxidant properties of OMWW phenolic extracts in combination with other antioxidants, which decreased oxidative damage during bread and toast baking. At 200 mg/kg of flour, *B. subtilis*, *E. coli*, and *P. aeruginosa* were detected.

1.12 Advanced Green Extraction Techniques

In response to the reviewer's recommendation, a dedicated subsection on green extraction technologies-including Ultrasound-Assisted Extraction (UAE) and Supercritical Fluid Extraction (SFE)-has been added to the revised manuscript.

1.12.1 Ultrasound-Assisted Extraction (UAE)

UAE significantly improves the recovery of phenolic compounds from OMWW by inducing cavitation, which enhances solvent penetration and mass transfer. In a notable comparison of extraction methods, Klen & Vodopivec (2011) demonstrated that ultrasound-assisted extraction yielded higher individual and total phenol recoveries versus conventional methods. Significantly, using freeze-dried OMWW and pure methanol, ultrasonication yielded high-quality phenol profiles without inducing degradation.

1.12.2 Supercritical Fluid Extraction (SFE)

SFE with supercritical CO_2 stands out for its eco-friendly nature, owing to its use of non-toxic, recyclable solvents and tunable selectivity through pressure and temperature control. While direct studies on OMWW are limited, SFE has proven effective for phenolic compounds in similar olive-derived matrices. For instance, a study on olive leaves reported higher phenol recovery with supercritical CO_2 (modified with methanol) compared to traditional solvent extractions (Le Floch et al., 1998). Additionally, preliminary work on lyophilized OMWW indicates that SFE can effectively recover oil and lipophilic compounds, although challenges remain in balancing selectivity for hydrophilic phenols (Dali et al., 2022).

1.13 Limitations and Challenges of Extraction and Purification Methods

While advanced extraction and purification methods show great promise for recovering polyphenolic compounds from olive oil mill wastewater (OMWW), several limitations and challenges must be considered to ensure practical feasibility and scalability.

1.13.1 Solvent-Based Extraction

Conventional solvent extraction, despite being simple and widely used, raises concerns regarding the toxicity, safety, and environmental persistence of certain organic solvents, such as methanol. Regulations (e.g., EU Regulation 2009/32/EC) limit methanol use in food-related applications, necessitating safer alternatives such as ethanol or water-ethanol mixtures. However, ethanol's lower polarity compared to methanol may result in reduced selectivity and altered phenolic profiles (Mahesar et al., 2010).

1.13.2 Ultrasound-Assisted Extraction (UAE)

Although the UAE significantly enhances extraction efficiency, cavitation generates reactive radicals (hydroxyl and hydrogen peroxide species), which may degrade antioxidant compounds. This requires precise optimization of parameters (frequency, power, duty cycle, and solvent system) to minimize degradation while maximizing yield (Priego-Capote et al., 2004).

1.13.3 Supercritical Fluid Extraction (SFE)

While SFE with supercritical CO_2 is considered an environmentally friendly technique, its high capital investment and operational costs remain barriers to industrial adoption. Additionally, CO_2 is a non-polar solvent, often requiring co-solvents such as ethanol or methanol to efficiently extract polar polyphenols, which complicates process design and increases cost (Lafka et al., 2011).

1.13.4 Pressurized Liquid Extraction (PLE)

PLE benefits from rapid extraction and lower solvent requirements; however, the high temperatures and pressures used may cause degradation or structural alteration of sensitive phenolic compounds. The operational cost of high-pressure systems and the requirement for specialized equipment also limit large-scale use (Herrero et al., 2011).

1.13.5 Membrane Technologies (MF, UF, NF, RO)

Membrane filtration offers high selectivity under mild conditions, but practical challenges include membrane fouling, reduced permeability over time, and high cleaning costs. Fouling by proteins, polysaccharides, and other organic matter in OMWW can significantly reduce process efficiency and the lifespan of the membranes. Furthermore, energy requirements for high-pressure NF and RO systems may offset some of the environmental benefits if not properly optimized (Zagklis & Paraskeva, 2020).

1.13.6 Biological Activity Loss

Across all methods, there is a risk of reduced biological activity due to exposure to harsh chemical, thermal, or mechanical conditions. This may compromise the functional properties of phenolic compounds, particularly when intended for food, pharmaceutical, or cosmetic applications. Thus, gentle processing and careful optimization are crucial to preserve bioactivity.

In conclusion, although the reviewed technologies offer significant potential for polyphenol recovery from OMWW, their limitations highlight the need for further optimization, hybrid process integration, and life-cycle assessments. Addressing these challenges is critical to ensure that polyphenol extraction technologies are not only efficient but also sustainable, safe, and economically viable for industrial applications.

1.14 Environmental and Sustainability Considerations

The valorization of OMWW should not only focus on the recovery of valuable polyphenolic compounds but also on the broader environmental sustainability of the employed technologies. Conventional solvent extraction, although effective, often generates significant solvent waste and requires substantial energy input, raising concerns over environmental footprint.

In contrast, emerging methodologies such as Ultrasound-Assisted Extraction (UAE), Supercritical Fluid Extraction (SFE), and Pressurized Liquid Extraction (PLE), particularly when using subcritical water, offer lower energy demands and reduced solvent usage. For example, SFE with supercritical CO_2 eliminates toxic organic solvents and enables solvent recycling-features supported by its recognition as an eco-friendly and efficient technique in biomass processing (Dali et al., 2022; Słota et al., 2025). UAE reduces processing time and energy usage, while PLE with green solvents further enhances eco-compatibility.

Membrane-based purification techniques, such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), represent sustainable approaches. Operating under mild conditions, they preserve the bioactivity of polyphenols without reliance on high-temperature processes, thus minimizing thermal degradation and energy costs.

To comprehensively assess the environmental and economic feasibility of these methods, future research should incorporate life cycle assessments (LCAs) and eco-efficiency analyses. Rodríguez-Pérez et al. (2025) demonstrate how LCA can help evaluate process sustainability, and similar frameworks should be applied to OMWW valorization. Such assessments would ensure that technological advancements in extraction are aligned with circular economy and sustainability goals.

1.15 Relevance of Polyphenol Purity for Industrial Applications

The purity of polyphenolic compounds plays a crucial role in determining their suitability for specific industrial uses. For food applications, high-purity extracts are essential to ensure safety, reproducibility of antioxidant activity, and compliance with food safety regulations. Impurities such as residual solvents, proteins, or lipids may alter sensory attributes or reduce shelf-life stability (Galanakis, 2012).

The purity of recovered polyphenolic compounds critically determines their suitability across various industries:

1.15.1 Food Applications

High-purity polyphenol extracts are essential to ensure both safety and functionality in food systems. Impurities, including residual solvents or non-phenolic components, can adversely affect flavor, shelf life, or regulatory compliance in food products (Rathee et al., 2023).

1.15.2 Cosmetic Applications

Plant-derived polyphenols are valued in dermocosmetics for their antioxidant and antimicrobial properties. High purity is necessary to maintain formulation stability, prevent oxidation, and avoid unintended skin irritation (AdamskaSzewczyk & Zgórka, 2019).

1.15.3 Pharmaceutical Applications

Achieving pharmacopeial-grade purity is mandatory for therapeutic use of phenolic compounds, as impurities can alter bioactivity and compromise safety-though specific quantitative standards vary across jurisdictions (this remains an identified research need).

Ultimately, while extraction yield is important, it is the post-extraction purification strategies (e.g., resin adsorption or membrane technologies) that ensure the final extract meets the required purity for industrial applications.

2. Conclusion

This study reviewed advanced extraction and purification techniques for recovering polyphenolic compounds from OMWW. The extraction methods examined included Ultrasound-Assisted Extraction (UAE), Supercritical Fluid Extraction (SFE), and Pressurized Liquid Extraction (PLE). These approaches not only improve extraction efficiency but also minimize solvent consumption and energy use, thereby offering both economic and environmental advantages.

2.1 Ultrasound-Assisted Extraction (UAE)

The application of high-frequency ultrasound waves enhances extraction yield by creating negative pressure and cavitation, which disrupts cell membranes and increases solvent-matrix interactions. This technique is characterized by its rapid processing and reproducibility. However, radicals formed during sonication may degrade antioxidant compounds, making parameter optimization essential.

2.2 Supercritical Fluid Extraction (SFE)

Based on the use of supercritical CO_2, SFE represents a promising green extraction technology due to its low toxicity, affordability, and tunable solvent properties. Its high selectivity and ability to replace traditional organic solvents underscore its environmental advantages. Moreover, adjustable operating conditions allow selective recovery of polyphenols.

2.3 Pressurized Liquid Extraction (PLE)

Operating under elevated pressure and temperature, PLE improves solubility and solvent penetration, leading to shorter extraction times, higher selectivity, and reduced use of hazardous solvents. Subcritical water has also been applied as a green alternative, offering efficient and sustainable recovery of phenolic compounds.

Following extraction, purification is a crucial step. Membrane-based separation technologies such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) enable efficient separation of contaminants and concentration of phenolic fractions. These methods preserve the bioactivity of phenolic compounds under mild conditions and offer a sustainable alternative to conventional high-temperature processes.

Polyphenolic compounds recovered from olive oil mill wastewater have demonstrated potential applications in the food, cosmetic, and pharmaceutical industries. In food systems, OMWW-derived extracts improve oxidative stability, extend shelf life, and enhance sensory attributes. Studies show their effectiveness in inhibiting microbial growth and lipid oxidation in products such as sausages and hamburgers, as well as improving freshness in bakery products. In summary, advanced extraction and purification technologies provide an efficient, sustainable pathway for valorizing olive oil mill wastewater. These methods mitigate environmental concerns associated with wastewater disposal while producing high-value phenolic extracts for diverse applications. By integrating green technologies, polyphenol recovery aligns with circular economy principles and represents a viable, sustainable strategy for industrial implementation.

While the reviewed extraction and purification methods show significant promise for valorizing OMWW, the transition to industrial-scale applications necessitates the development of robust standardization and quality control protocols. A major challenge in utilizing recovered polyphenols, particularly from a natural and variable waste source like OMWW, is ensuring batch-to-batch consistency in terms of phenolic profile, concentration, and bioactivity. This variability is inherent due to factors such as olive cultivar, geographical origin, climatic conditions, maturity of the fruit, and the specific technology used for olive oil extraction (three-phase vs. two-phase).

Therefore, for the successful integration of OMWW-derived polyphenols into high-value markets (food, pharmaceutical, and cosmetic industries), it is imperative to move beyond extraction efficiency and focus on standardizing the final product. Future research and development must prioritize:

2.3.1 Establishing Standardized Analytical Methods

Defining consensus methods for the quantification of key bioactive compounds (e.g., hydroxytyrosol, tyrosol, oleuropein) is crucial. Techniques like HPLC-MS/MS should be standardized for accurate fingerprinting.

2.3.2 Defining Purity Benchmarks

Setting industry-accepted thresholds for the minimum concentration of target phenolics and maximum limits for undesirable components (e.g., heavy metals, pesticide residues, microbial contaminants) is essential for safety and efficacy.

2.3.3 Implementing Process Controls

Integrating Quality by Design (QbD) principles into the development of extraction and purification processes can help identify critical process parameters (CPPs) that directly impact the critical quality attributes (CQAs) of the final extract, ensuring reproducibility.

2.3.4 Biological Activity Standardization

Since antioxidant capacity is a key selling point, standardizing extracts based on their biological activity (e.g., ORAC value, IC50 for radical scavenging) rather than solely on chemical concentration could provide a more reliable measure of potency and functionality across different batches.

Addressing these standardization gaps is a critical next step to build trust among end-users and regulatory bodies, ensuring that OMWW evolves from a variable waste stream into a reliable and consistent source of high-value, bioactive compounds.

Authors' Contributions

Ali Ammarlo: Conceptualization; Data curation; Investigation; Writing original draft; Writing-review & editing. **Arash Ghaitaranpour:** Conceptualization; Supervision; Writing-review & editing. **Adel Mirza Alizadeh:** Conceptualization; Visualization; Writing-review & editing. **Nesa Azimzadeh:** Writing-original draft.

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Conflicts of Interest

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Ethical Considerations

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Using Artificial Intelligence

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