

# Orange Peel Waste Valorization from Bioactive Compound Recovery to Functional and Industrial Applications

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## Abstract

Orange and citrus peel, often considered agro-industrial waste, are rich sources of bioactive compounds such as flavonoids, polyphenols, essential oils, and dietary fibers. This review comprehensively discusses the chemical composition and functional properties of orange peel, highlighting enzymatic biotransformation and advanced extraction strategies including ultrasound-, microwave-, and enzyme-assisted techniques. Special attention is given to green extraction approaches using deep eutectic solvents for sustainable recovery of hydrophilic and lipophilic bioactive compounds. The functional applications of orange peel-derived compounds in food, nutrition, pharmaceuticals, cosmetics, agriculture, and industrial materials are also presented, emphasizing their potential in developing eco-friendly biobased products and active food packaging. By integrating chemical profiling, sustainable extraction methods, and multifunctional applications, this review provides a systematic overview of the valorization of orange and citrus peel waste, promoting circular bioeconomy approaches and environmental sustainability.

## Keywords

Orange Peel, Citrus Waste, Bioactive Compounds, Green Extraction, Deep Eutectic Solvents, Flavonoids, Polyphenols, Functional Foods, Sustainable Valorization

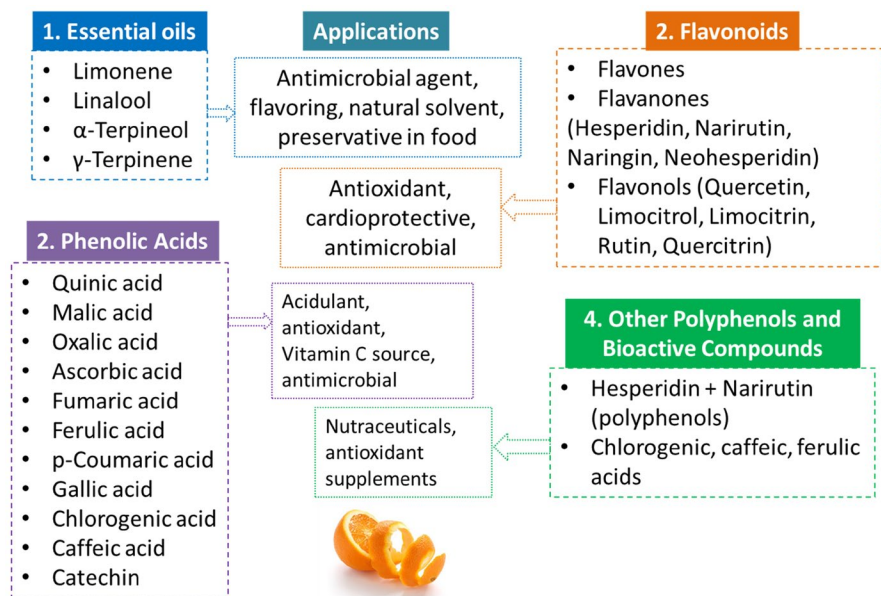
## 1. Introduction

Based on data from the U.S. Department of Agriculture, the average global orange production over the 2015-2024 marketing years is estimated at 48.66 million metric tons. Brazil is the leading producer, accounting for approximately 29% of global

output, with production projected at 13 million metric tons for the 2024–2025 marketing year. China is the second-largest producer, contributing about 17% of total production (7.62 million metric tons), followed by the European Union, which accounts for approximately 13% of global orange production, equivalent to 5.66 million metric tons [1]. Specifically, in the context of orange juice production, approximately 55 million tons of orange waste are generated worldwide each year [2].

### 1.1. Chemical Composition and Bioactive Profile of Orange Peel

Fruit peels are increasingly recognized as concentrated sources of bioactive compounds with notable nutritional and therapeutic potential. These compounds include phenolics such as quercetin, catechins, and mangiferin; flavonoids like hesperidin and naringin; carotenoids including  $\beta$ -carotene and lycopene; vitamins C and E; dietary fibers; and essential oils such as limonene and citral (Figure 1). Although structurally and functionally diverse, these bioactives often act synergistically to support human health and reduce disease risk. Specific examples include mango peels rich in mangiferin, apple peels containing quercetin, banana peels with resistant starch and serotonin precursors, and citrus peels abundant in limonene. Their combined antioxidant, antimicrobial, and anti-inflammatory activities underpin a wide range of biological benefits. The composition and concentration of these compounds vary according to fruit type, cultivar, ripeness, and processing methods [3]. Understanding this broad chemical composition sets the stage for exploring specific bioactive subclasses and their functional properties.

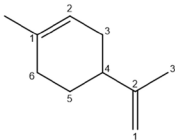
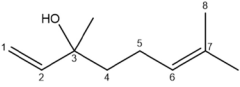
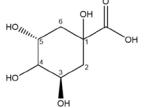
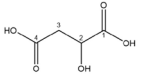

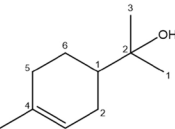
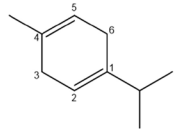
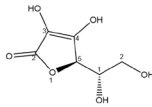
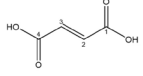
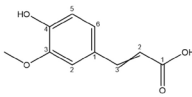
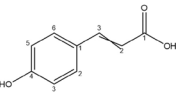
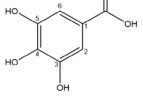
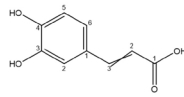
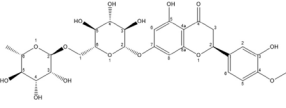
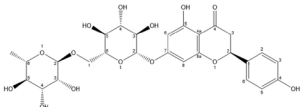
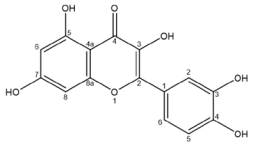
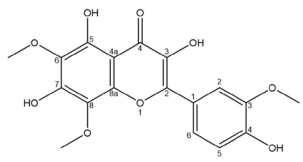
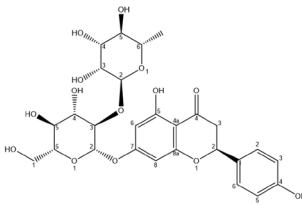
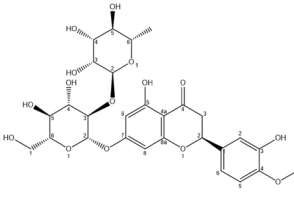
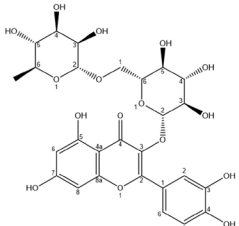
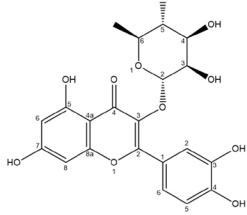


**Figure 1.** Orange peel chemical composition and applications.

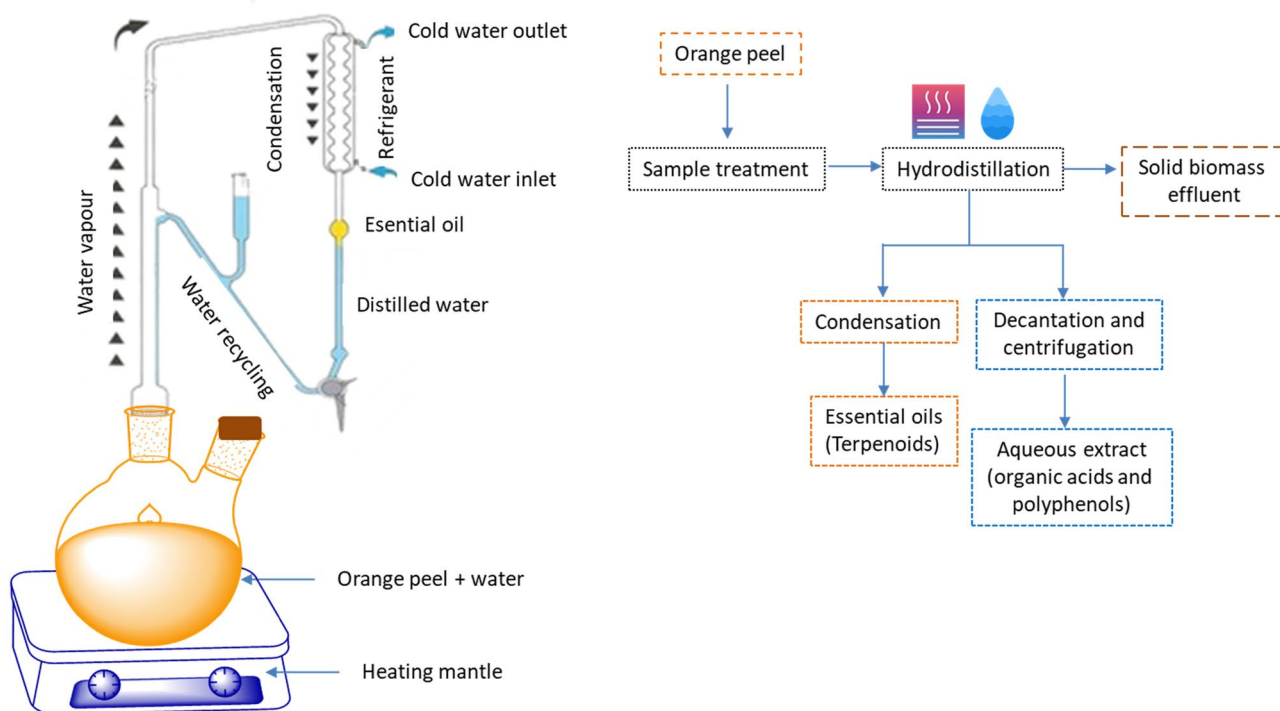
Valorization of orange peel is an effective approach that uses this biomass as a renewable, cost-effective, and abundant source of valuable natural compounds with significant industrial and economic potential. Orange peels are rich in limo-

nene (36.7% - 74.4%), linalool (2.2% - 5.3%),  $\alpha$ -terpineol (1.2% - 6.7%), and  $\gamma$ -terpinene (0.4% - 7.4%), exhibiting notable antioxidant and antimicrobial effects [4]. Aqueous extracts of orange peel contain significant amounts of quinic and malic acids, along with flavonoid glycosides. Orange aqueous extracts demonstrated a significant inhibitory effect on the growth of MCF-7 breast cancer cells. Principal component analysis highlighted the active organic acids and phenolic compounds responsible for these bioactivities. This research underscores the potential of orange peel as a source of high-value phytochemicals and supports sustainable, cost-effective extraction methods for eco-friendly citrus waste management. Orange peel contains a variety of bioactive compounds that contribute to its aroma, flavor, and potential health benefits. These compounds include essential oils, flavonoids, vitamins, and polysaccharides, each with distinct chemical structures and properties. **Table 1** summarizes the major chemical constituents found in orange peel along with their corresponding structural formulas, providing a clear representation of its complex phytochemical composition.

**Table 1.** Major chemical constituents of orange peel with their structural formulas.

Essential oil		Phenolic acids		
				
Limonene	Linalool	Quinic acid	Malic acid	Oxalic acid
				
$\alpha$ -terpineol	$\gamma$ -terpinene	Ascorbic acid	Fumaric acid	Ferulic acid
				
		p-Coumaric acid	Gallic acid	Caffeic acid
Flavanones		Flavonols		
				
Hesperidin	Narirutin	Quercetin	Limocitrol	
				
Naringin	Neohesperidin	Rutin	Quercetrin	

Orange peel essential oils obtained through hydrodistillation offer high yields, enhanced purity, longer shelf life, elevated limonene content, and low phototoxicity, making them suitable for industrial applications. Hydrodistillation is a simple, long-used technique applied to extract bioactive compounds present in plant essential oils. This method can be carried out in three main forms: water distillation, water and steam distillation, and direct steam distillation. The process relies on three physicochemical mechanisms, which are hydrodiffusion, hydrolysis, and heat-induced decomposition. Hydrodistillation is usually performed at temperatures higher than the boiling point of water and commonly employs a device called the Clevenger apparatus (Figure 2). In this system, plant material is mixed with water and heated until the volatile oil components evaporate. In the case of steam distillation, however, steam is passed through a bed of plant material to release the essential oil vapors [5]. In orange oil, monoterpene hydrocarbons are dominant, accounting for 84.9%, followed by oxygenated monoterpenes at 13.3% [4]. These compounds exhibit antimicrobial [6], antioxidant, and anti-inflammatory properties, supporting their potential medical and cosmetic uses. Orange peels are also rich in organic acids, including malic, quinic, ascorbic, fumaric, and notably oxalic acid, which is predominantly present in orange peel extracts. Prolonged extraction can degrade these acids, but they are widely applied in the food industry as antimicrobial agents, flavoring components, acidulants, and pH regulators.



**Figure 2.** Sustainable recovery of multiple products from citrus peels via hydrodistillation.

Regarding flavonoids, flavones are the most abundant phenolics in orange peel extracts, with compounds such as chrysoeriol 6,8-di-C-glucoside (stellarin-2) present at  $1.05 \pm 0.06$  mg/g. Flavanones, mainly polymethoxylated glycosides of eriod-

xytiol and naringenin, make up less than 15% of the flavonoids, and naringenin-O-deoxyhexosyl-hexoside is found in trace amounts (<0.3 mg/g) [4]. Flavonols, including glycosylated quercetin, limocitrol, and limocitrin, are present in minor quantities (<1 mg/g). Orange peels also contain phenolic acids such as ferulic, coumaric, and chlorogenic acids. These chemical characteristics, particularly of the essential oils, suggest that orange peel extracts may be especially useful for enhancing the safety of meat or dairy products prone to contamination by pathogens like *Campylobacter jejuni* [4].

The primary polyphenols identified in orange peel waste (OPW) include phenolic acids such as gallic acid, p-coumaric acid, and ferulic acid, as well as flavonoids including hesperidin and narirutin [7].

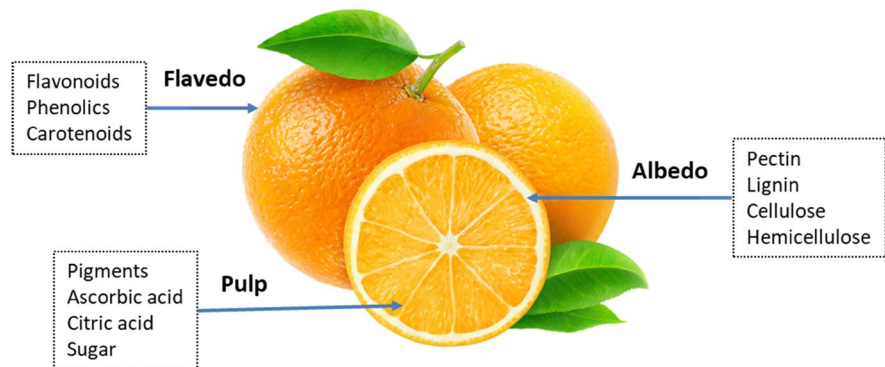
Research on citrus by-products shows that orange peel stands out for its rich composition and strong antimicrobial potential. Its extract contains notably high levels of phenolic compounds (39.54 mg GAE/g) and flavonoids (79.54 mg CE/100 g), supported by the presence of key phenolic acids such as chlorogenic, caffeic, ferulic, and catechin, along with abundant flavonoids including quercitrin, rutin, and hesperidin. Limonene is the dominant volatile in the peel oil, though its proportion decreases from 92.52% to 76.62% during processing [8].

When formulated into nanoemulsions [9], the orange peel extract produces particles between 29 and 66 nm with low polydispersity and stable zeta potential values, contributing to overall stability of more than 80%. These chemical features align with its strong antioxidant behavior and its superior antibacterial and antifungal effects compared to tangerine peel, particularly against Gram-positive bacteria and several *Aspergillus* species. Orange peel also demonstrates notable anti-aflatoxigenic activity, supported by molecular docking results showing strong inhibitory interactions, especially from hesperidin with enzymes involved in aflatoxin biosynthesis [8].

## 1.2. Flavonoid Composition and Enzymatic Biotransformation in Orange Peel

Orange peel is composed of two main layers: the flavedo, which is the outer layer, and the albedo, the white inner layer (Figure 3). Research has shown that the albedo contains a higher concentration of flavonoids compared to the flavedo [10]. Among the flavonoids present in the albedo are hesperidin, rutin, and naringin, the latter being the most abundant flavanone in orange peel albedo and attracting attention due to its biological activities, including antioxidant, cardioprotective, antibacterial, antiviral, and anticarcinogenic effects [11]. Naringinase is an enzyme complex that exhibits  $\alpha$ -L-rhamnosidase and  $\beta$ -D-glucosidase activities, enabling the hydrolysis of various flavanones in orange peel albedo. Prominent flavanones such as naringin, narirutin, hesperidin, neohesperidin, and rutin can be converted into their respective aglycones with high antioxidant activity, including naringenin and prunin, through enzymatic catalysis [12]. The mechanism of naringinase action begins with  $\alpha$ -L-rhamnosidase activity, which releases rhamnose and prunin from flavanones in the albedo. Subsequently,  $\beta$ -D-glucosidase acts on

prunin, releasing glucose and producing naringenin as the final product [13]. The catalytic specificity of naringinase has been extensively investigated due to its potential for biotransformation of flavonoid glycosides in citrus by-products.



**Figure 3.** Anatomy of the orange [14].

Beyond flavonoids, orange peel is also rich in functional dietary fibers that synergize with bioactive compounds to promote health.

### 1.3. Flavonoids and Functional Dietary Fibers in Orange Peel

Citrus peels contain a wide array of flavonoids and dietary fibers, both water-soluble (SDF) and insoluble, contributing to antioxidant, anti-inflammatory, and immune-modulating activities. Studies on navel orange peel have demonstrated that superfine grinding improves SDF extraction and functional properties, enhancing water retention, oil absorption, nitrite binding, and antioxidant activity. These fibers hold promise as functional food ingredients while supporting sustainable utilization of citrus waste. Flavonoids are abundant natural compounds in citrus species and play a key role in the biological value of citrus by-products. Phenolic compounds, especially flavonoids like anthocyanins, flavones, and flavanones, are commonly found in fruit peels [15]. These bioactive substances play an important role in the supporting human health. High flavonoid content can be utilized in the creation of natural food preservatives [16], while the substantial levels of anthocyanins reported by [17] highlight their potential for developing supplements with strong anti-inflammatory or anti-ulcer effects [18]. The selection of rootstocks also plays a key role in shaping the phenolic profile of blood orange peels, as rootstocks influence nutrient uptake, enzyme activity, and the metabolic pathways involved in phenol synthesis and accumulation. These factors ultimately determine both the amount and the specific types of phenolic compounds found in the fruit [17]. Zheng and colleagues [19] analyzed nine citrus peels from mandarins, oranges, and pomelos using ultra-performance liquid chromatography paired with quadrupole time-of-flight mass spectrometry, identifying fifty-one flavonoids across diverse subclasses, with polymethoxyflavones being the most prevalent. Several compounds, including icariin, sophora flavanone G, puerarin, dimeflin, vestitone,

and galangin, were reported for the first time in citrus peels. Extracts from specific citrus fruits from China, especially Sichuan MJ (orange—*Citrus reticulata* Blanco (MJ) from Sichuan Province) and Changshan HY (pomelo—*Citrus maxima* (HY) from Changshan Province), showed strong antioxidant and anti-inflammatory activities by lowering reactive oxygen species, improving A549 cell viability, and reducing transcription of key cytokines such as tumor necrosis factor alpha, monocyte chemoattractant protein one, interleukin six, and interleukin eight. These extracts also enhanced nuclear factor erythroid two related factor two and heme oxygenase one expression while suppressing p65 phosphorylation and nuclear factor kappa B activation. Gray correlation analysis highlighted dimeflin, eupafolin, and trans cinnamaldehyde as major contributors to these effects. Distinct metabolomic profiles further differentiated MJ and HY, revealing twenty-six down-regulated metabolites strongly associated with enhanced bioactivity [19].

Navel orange, a sweet variety of citrus, is cultivated extensively in many regions, with Jiangxi Ganzhou in China representing the largest production area. This region covers around 103,200 hectares and produces approximately 1.2 million tons annually, ranking third globally [20]. During fruit processing, nearly 30 percent of the fruit mass, primarily peels, is discarded, leading to environmental challenges. However, citrus peels are rich in bioactive substances, such as phenolic compounds, polysaccharides, limonoids, flavonoids, and dietary fibers, which exhibit antimicrobial, antioxidant, and anticancer properties [21]. Water-soluble dietary fiber (SDF) from navel orange peel has emerged as a promising functional ingredient due to its health-related properties. Superfine grinding of SDF enhanced its extraction yield, reduced particle size, and improved functional characteristics including water retention, swelling, oil absorption, nitrite binding, and antioxidant activity. In macrophage RAW264.7 cells, treated SDF reduced reactive oxygen species production, stimulated nitric oxide, and increased cytokine levels such as interleukin-1 $\beta$ , tumor necrosis factor alpha, monocyte chemoattractant protein-1, and interleukin-6. The fibers also upregulated phosphorylated P65 and heme oxygenase-1, indicating mild macrophage activation and improved immune function. These results suggest that superfine-ground navel orange peel SDF could serve as a bioactive component in functional foods [22]. Dietary fibers are classified into water-soluble and water-insoluble types, with soluble fibers showing stronger physiological benefits, including improved gut health, regulation of blood glucose and lipid levels, and reduced risk of chronic diseases. The functionality of SDF depends heavily on its chemical composition and physicochemical properties, which can be enhanced through processing methods like particle size reduction. Such treatments modify fiber structure and surface characteristics, resulting in improved bioactivity. Therefore, utilizing navel orange peel to produce high-value SDF not only adds economic value but also offers opportunities for sustainable waste management and the development of functional food ingredients [22]. The bioactive potential of flavonoids and fibers can be further amplified through enzymatic valorization strategies.

## 2. Extraction and Recovery of Bioactive Compounds from Orange Peel

The following section provides a detailed discussion of each method, emphasizing extraction efficiency, sustainability, and the potential health benefits of the recovered compounds. **Table 2** presents the principal methods for extracting bioactive compounds from orange peel, along with the major compounds obtained and their associated bioactivities.

**Table 2.** Key orange peel extraction methods, major compounds recovered, and their bioactives.

Method	Major Compounds	Key Bioactivities/Applications
Hydrodistillation	Limonene, $\alpha$ -pinene, D-carvone, sesquiterpenes, carotenoids, flavonoids	Aroma/flavor, antioxidant, antibacterial, anti-inflammatory, anticancer
Enzymatic Hydrolysis (Naringinase)	Naringin, hesperidin $\rightarrow$ prunin, naringenin	Anti-cancer, bioactive functional ingredients
Biorefinery (Pectinase + Filtration)	Phenolics (polyphenols)	Antioxidant, food-grade ingredients, circular economy
Ultrasound/Enzyme-Assisted Extraction	Flavonoids, phenolics	Antioxidant, functional foods, nutraceuticals
Microwave-Assisted Extraction	Limonene-rich volatiles, polyphenols	Antibacterial, cytotoxic, antioxidant, enzyme inhibition

### 2.1. Hydrodistillation and Chemical Composition of Essential Oil

Hydrodistillation was performed according to the guidelines of the Spanish Pharmacopoeia [23]. Citrus peels (300 - 450 g) were hydrodistilled with distilled water (1:10) for 8 h using a Clevenger-type apparatus. Essential oils were dried over anhydrous magnesium sulfate, stored at 4 °C in the dark, and yields expressed as mL per 100 g of fresh peel. The residual aqueous phase was recovered, freeze-dried, and stored for further analysis.

Guo *et al.* [24] conducted experiments using citrus fruits obtained from a local supermarket in Joensuu, Finland. The outer colored portion of the peels (epicarp) was carefully separated from the fruits, avoiding inclusion of the white inner layer (mesocarp). To minimize the loss of volatile compounds, the peel samples were stored at 4 °C prior to hydrodistillation. Hydrodistillation of orange peels was performed using a Clevenger-type apparatus equipped with a condenser. Chemical profiling of the extracted oils revealed 228 tentatively identified compounds, including terpenes, terpenoids, carotenoids, fatty acids, phenolic acids, flavonoids, and esters, of which over 100 compounds had not been previously reported in citrus peel essential oils or other citrus-derived products. Complementary analysis using high-resolution GC-QTOF MS detected 38 volatile compounds, including terpene isomers, providing a more detailed characterization of the essential oil composition [24].

Terpenes, particularly monoterpenes and sesquiterpenes, represent the dominant classes in citrus peel essential oils. In this study, GC-QTOF MS analysis revealed that the main compounds were monoterpenes, monoterpenoids, sesquiterpenes,

and sesquiterpenoids. Positive-ion APPI analysis indicated that monoterpenes (C<sub>10</sub>H<sub>16</sub>) were the most abundant class, with limonene identified as the major monoterpene by GC-QTOF MS. Other prevalent monoterpenes included  $\alpha$ -thujene,  $\alpha$ -pinene, sabinene,  $\beta$ -pinene,  $\beta$ -myrcene,  $\alpha$ -phellandrene,  $\alpha$ -terpinene,  $\beta$ -ocimene,  $\gamma$ -terpinene, terpinolene, and p-cymene (C<sub>10</sub>H<sub>14</sub>). Several monoterpenoids (C<sub>10</sub>H<sub>16</sub>O) were inferred from both APPI FT-ICR and GC-QTOF data, including  $\alpha$ -pinene oxide, cis-limonene oxide, trans-limonene oxide, nerol oxide, limonen-4-ol, trans-carveol, and D-carvone. Among these,  $\alpha$ -pinene oxide was the predominant monoterpenoid across the five citrus peel oils, contributing woody, pine-like, and slightly floral notes to the aroma. Cis-limonene oxide imparted a sweet, floral scent, while trans-carveol is a major volatile component in the flesh of dekopon, a hybrid citrus [25]. D-carvone, a pale yellow or colorless liquid with a caraway-like odor, is widely used as a flavoring agent and exhibits pharmacological activities including antibacterial, antifungal, antioxidant, anti-inflammatory, and anticancer effects [26]. Several sesquiterpenes (C<sub>15</sub>H<sub>24</sub>) were also detected, including (E)-caryophyllene, cis-thujopsene, and  $\beta$ -bisabolene. Lemon and lime peel essential oils contained higher levels of sesquiterpenes compared to grapefruit, mandarin, and orange peel oils. Oxygenated sesquiterpenes, such as turmerone,  $\beta$ -calacorene, 8-methoxy-p-cymene, cis-caryophyllene oxide, nootkatone, and farnesol, were additionally identified. Notably, sesquiterpenoids such as costunolide and urodiolenone were detected in citrus peel oils for the first time. Citrus fruits are among the richest natural sources of carotenoids, which are pigments responsible for the yellow, orange, and red hues in many plants. The most prominent carotenoid in citrus is  $\beta$ -carotene (C<sub>40</sub>H<sub>56</sub>). In the present study, additional carotenoids were detected using positive-ion APPI MS, including  $\beta$ -apocarotenal (C<sub>30</sub>H<sub>40</sub>O) and  $\beta$ -citraurin (3-hydroxy- $\beta$ -apocarotenal; C<sub>30</sub>H<sub>40</sub>O<sub>2</sub>) [24]. These compounds, which impart deep red to red-orange coloration, are particularly concentrated in the fruit peels. The predominant fatty acids identified in citrus peel essential oils included palmitic acid, linoleic acid, stearic acid, linolenic acid, lauric acid, decanoic acid, and oleic acid. The waxy outer layer of citrus peels typically contains fatty acids, alcohols, aldehydes, alkanes, and esters, which contribute to a protective and glossy surface. Palmitic acid and oleic acid are particularly common in this wax composition.

In this investigation [24], limonene was the predominant constituent in all analyzed samples. Beyond compounds previously documented in citrus peel essential oils and related citrus products, numerous additional constituents were tentatively identified using DI FT-ICR MS. This technique enables the detection of hundreds of semipolar and polar compounds that are generally inaccessible by conventional GC-MS, unless labor-intensive derivatization and extraction steps are performed. Conversely, high-resolution GC-QTOF MS offered complementary information, particularly through its ability to resolve isomeric terpene hydrocarbons and terpenoids. The combined application of these analytical approaches allows for the effective profiling of both polar and nonpolar compounds across a wide molecular weight range, highlighting the exceptional chemical complexity of citrus peel essential oils.

Many of these fatty acids also offer health benefits. For example, moderate consumption of linoleic acid may lower the risk of cardiovascular disease [27], while linolenic acid exhibits antithrombotic and anti-inflammatory properties [28]. Decanoic acid has additionally been reported to possess antibacterial and anti-inflammatory activities [29]. The major phenolic acids identified in the citrus peel essential oils were caffeic acid and ferulic acid, with smaller amounts of gallic acid and sinapic acid also detected. Caffeic acid was the predominant phenolic acid across all five citrus peel oils and plays key roles as an antioxidant and antimicrobial agent, contributing to the protective functions of the peel. Flavonoids, particularly flavones and flavanones, are highly concentrated in the peel, pith, and juice of citrus fruits and have been widely studied for their potential health benefits, including cardiovascular support and anti-inflammatory effects [30]. Hesperetin is the predominant flavanone present in citrus peels. Esters also contribute significantly to the distinctive aroma and flavor of citrus fruits, while potentially offering health benefits. Octyl acetate, for instance, exhibits a fruity and floral scent with subtle citrus notes and is commonly used in perfumes, flavorings, aromatherapy products, and orange-scented candles. Citronellyl formate, the most abundant ester in orange peel essential oil, is characterized by a floral, citrusy aroma.

## 2.2. Enzymatic Valorization of Flavanones from Orange Peel Albedo

Sustainable extraction of flavanones from orange peel albedo followed by enzymatic hydrolysis using naringinase generates bioactive aglycones with applications in cancer prevention. Enzyme immobilization improves reusability and stability, demonstrating a scalable, cost-effective approach to convert citrus by-products into high-value functional compounds. This study [31] focused on the sustainable and cost-effective extraction of flavanones from orange peel albedo as an alternative to purified substrates. The resulting extracts, enriched in naringin, naringutin, hesperidin, neohesperidin, and rutin, were subjected to enzymatic hydrolysis using a naringinase complex. The  $\alpha$ -L-rhamnosidase and  $\beta$ -D-glucosidase activities of naringinase converted these flavanones into prunin and naringenin. The bioactivity of the hydrolyzed compounds was assessed in SW480 colon cancer cell lines, revealing potential compounds for cancer prevention. Further optimization showed that purification of naringinase enhanced its  $\alpha$ -L-rhamnosidase affinity, while immobilization on corn cob powder improved enzyme reusability and stability, retaining up to 75% of initial activity after 10 consecutive cycles. By employing whole extracts without prior purification, this study presents a scalable and practical approach for valorizing citrus waste into biologically active compounds, supporting both economic and environmental sustainability. Complementary to enzymatic methods, biorefinery approaches enable recovery of phenolics at pilot and industrial scales.

## 2.3. Recovery of Phenolic Compounds Using Biorefinery Approaches

Pilot-scale, food-grade extraction of phenolics from orange peel using enzymatic

hydrolysis of pectin followed by filtration techniques yields a phenolic-rich solution with high antioxidant activity. These non-thermal, green processes align with circular economy principles and allow valorization of residual biomass for animal feed or fertilizers, supporting both sustainability and industrial applicability. Orange peels are rich in valuable polymers and bioactive compounds that can be recovered using biorefinery approaches.

Niglio *et al.* (2024) [32] developed a pilot-scale, food-grade, non-thermal process to extract phenolics from orange peel (OP) without organic solvents. At 1000 L scale, pectin was enzymatically hydrolyzed, followed by optional ultrasound-assisted extraction (UAE) at 54 W/g and 20°C. Solid-liquid separation via decanter centrifuge recovered a phenolic-rich solution, further purified through sequential micro-, ultra-, and nanofiltration. HPLC-ESI-TOF-MS revealed a diverse phenolic profile (3.3 g/L total polyphenols) suitable for food packaging or fortification. UAE minimally increased yield and could be omitted to save energy. Sequential filtration produced a high-phenolic product with notable bioactivities. The process aligns with green chemistry and zero-waste principles, using fresh OP, recycling water, and valorizing residuals for animal feed or fertilizers. Future work should optimize enzymatic hydrolysis to improve yield and reduce energy use. Life cycle and cost analyses are needed to assess feasibility. Scaling up and applying similar non-thermal methods to other agro-food wastes could enhance by-product valorization, supporting circular economy goals. The final product meets food-grade safety standards, confirming industrial applicability. Advanced extraction techniques can further improve recovery efficiency of bioactives from orange peel.

#### 2.4. Microwave-Assisted Extraction of Volatile and Non-Volatile Compounds

Microwave-assisted extraction (MAE) efficiently recovers both volatile (limonene-rich) and non-volatile (polyphenol-rich) compounds from orange peel. Volatile extracts show antibacterial and cytotoxic activities, while non-volatiles exhibit antioxidant, anti-inflammatory, and enzyme inhibitory effects, making MAE a rapid, sustainable method for valorizing citrus peel in nutraceutical and food applications. Compared to conventional solvents, ultrasound-assisted (UAE) and enzyme-assisted extraction (EAE) of Washington Navel, Navelina, and Brocade orange peels improve recovery of phenolics and flavonoids. Optimizing extraction parameters enhances antioxidant activity, highlighting UAE and EAE as eco-friendly, efficient methods for producing bioactive extracts suitable for functional foods and health-promoting products.

Wang *et al.* (2023) [33] optimized ultrasound-assisted extraction (UAE) of phenolic compounds from orange peels, including free, esterified, and glycosylated-bound fractions. UAE (17.6 mL/g, 28 min, 26°C, 60 W) yielded maximum TPC (24.07 mg GAE/g) and TFC (8.34 mg RE/g). Extraction method and phenolic fraction strongly influenced yield, composition, and antioxidant activity. UAE and

enzyme-assisted extraction (EAE) outperformed conventional solvent extraction, with key compounds including polymethoxyflavones, ferulic acid, and hesperetin. Solid-to-solvent ratio was the most critical factor for UAE efficiency. The study highlights UAE and EAE as eco-friendly, effective strategies to recover natural antioxidants from citrus peel for functional foods and circular economy applications.

Peels of Washington Navel (WAS), Navelina (NAV), and Vaniglia Apireno (VAN) oranges, abundant by-products of the Italian agro-food sector in Ribera, Sicily, were investigated for their bioactive potential [34]. Microwave-assisted extraction (MAE) using water efficiently recovered volatile (VE) and non-volatile (NVE) compounds from fresh orange peels. VEs, mainly limonene (>85%), showed antibacterial and cytotoxic activity, while NVEs exhibited antioxidant, anti-inflammatory, and enzyme-inhibitory effects, with polymethoxylated flavonoids as key contributors. MAE provides a fast, eco-friendly method to valorize orange peel by-products for nutraceutical applications, supporting circularity in the Sicilian orange industry.

## 2.5. Green Extraction Using Deep Eutectic Solvents (DESs)

The fortification of food products with bioactive compounds from natural and sustainable sources has gained increasing attention. Academic and industrial research has increasingly focused on sustainable techniques for recovering bioactive compounds from natural resources. Traditionally, the extraction of these compounds from plant matrices relies on organic solvents, which pose environmental hazards and potential toxicological risks for human consumption [35]. A promising alternative is the use of green solvents, which are safe for direct inclusion in food products and can reduce industrial costs by eliminating the need for downstream solvent removal [36]. Deep eutectic solvents (DESs), formed from eutectic mixtures of acids and bases with diverse anionic and cationic species, align with the principles of green chemistry and have shown superior performance compared to traditional organic solvents [37]. Components of DESs are often non-toxic and possess antioxidant properties, making them suitable for direct use in food formulations [38].

The extraction efficiency of DESs depends on the physicochemical affinity between the solvent components and the target bioactive compounds, considering factors such as polarity and pH. Two main categories of DESs exist: hydrophilic DESs, which are well-established for extracting polar compounds such as polyphenols, flavonoids, anthocyanins, and proteins [39], and hydrophobic DESs, recently developed for isolating nonpolar compounds such as carotenoids. Based on these considerations, the present study aimed to develop green extraction protocols to obtain high-value bioactive compounds, specifically polyphenols and carotenoids, from orange peel using both hydrophilic and hydrophobic DESs.

Applying these solvents enables selective recovery of both hydrophilic and hydrophobic bioactives from citrus peel.

### 2.5.1. Extraction of Hydrophilic and Hydrophobic Bioactives Using DESs

Orange peel extracts rich in bioactives were obtained using hydrophilic and hydrophobic deep eutectic solvents (DESs) [40] and evaluated for stability, antioxidant capacity, permeability, and biological activity for potential food applications. Hydrophobic DESs were most effective for carotenoid extraction, with DL-Menthol:Camphor (163.5 mg/100 g fw), DL-Menthol:Eucalyptol (168.7 mg/100 g fw), and Lauric acid:Octanoic acid (153.1 mg/100 g fw). Hydrophilic DESs, especially Proline:Malic acid (282.8 mg/100 g fw), yielded higher polyphenols. Antioxidant activity was superior in hydrophobic DES extracts. Carotenoid stability was highest with DL-Menthol:Camphor, while Lactic acid:Glucose and Proline:Malic acid better stabilized polyphenols. Permeability assays (PAMPA) showed improved absorption of carotenoids and polyphenols, and DL-Menthol:Camphor extracts reduced HeLa cell viability to 26.7%, demonstrating antiproliferative activity. These results indicate that DESs provide stable, permeable bioactive extracts from orange peels suitable for direct inclusion in food formulations, with other advanced extraction methods offering complementary efficiency and selectivity.

This study [41] valorized orange peel using ultrasound-assisted extraction with deep eutectic solvents (DESs). Choline chloride-lactic acid at a 1:10 molar ratio gave the highest total extraction yield (43.88% dry weight), while the 2:1 ratio maximized phenolic recovery (4.12 mg GAE/g), antioxidant activity (2.55 mmol Trolox/g), and antimicrobial effects. Key phenolics quantified at this ratio included naringin (1150.29 µg/g), caffeic acid (139.41 µg/g), and ferulic acid (379.96 µg/g). Polysaccharide extraction was most efficient with a 1:1 DES ratio, yielding 48.24% pectin. These results highlight DES combined with ultrasound as a green, efficient method for simultaneously recovering pectin, phenolics, and antimicrobial compounds from citrus by-products, supporting sustainable biorefinery approaches.

### 2.5.2. Hydrophobic DESs for Lipophilic Bioactive Recovery

While many bioactive compounds are hydrophilic and research often focuses on hydrophilic deep eutectic solvents (DES) for their extraction, numerous lipophilic compounds, such as carotenoids and tocopherols, are also present in natural substrates and require specialized extraction strategies [42]. Hydrophobic deep eutectic solvents (HDES) have emerged as effective media for extracting lipophilic molecules. HDES are typically formed by combining long-chain quaternary ammonium salts or terpenoids with long-chain carboxylic acids [43].

Carotenoids are antioxidant pigments whose lipophilic nature makes them sensitive to heat, light, and oxygen, limiting solubility and bioavailability. Sustainable extraction methods, such as HDES, provide efficient recovery while enhancing their stability and antioxidant capacity, preserving their functionality for food and pharmaceutical applications.

This study [44] screened 68 deep eutectic solvents (DESs) for carotenoid extraction from orange peels, identifying hydrophobic DESs especially Menthol:Camphor as most effective, yielding 163.5 mg/100 gfw and retaining 56%

stability after 60 days at 4°C. Ultrasound-assisted extraction optimized yield to 653.5 mg/100 gfw using 60% intensity, 20 mL/g solvent-to-solid ratio, and 20 min. Hydrophobic DESs offer a sustainable, edible, and non-toxic method for efficient, stable carotenoid extraction suitable for food fortification and health applications.

### 2.5.3. Revalorization of Orange Peels Using Hydrophobic Natural DESs

The recovery of bioactive compounds from food by-products offers significant opportunities for their use as functional additives in the food industry. Carotenoids, in particular, are valued for their antioxidant, antimicrobial, and natural colorant properties. In this study [45], evaluated hydrophobic natural deep eutectic solvents (HDES) for extracting carotenoids from orange peels and assessed in vitro bioactivity, including cytotoxicity, antiproliferative, antioxidant, and antimicrobial effects against *Staphylococcus aureus* and *E. coli*, as well as bioaccessibility using the INFOGEST digestion protocol. Among HDES, Octanoic acid:Proline (C8:Pro) and Menthol:Eucalyptol (Me:Eu) showed high selectivity for colorectal cancer cells. C8:Pro yielded the most carotenoids (168 µg/100 g lutein, ORAC 34.8 mM TE) and was the only extract with antibacterial activity against both bacteria. Its β-cryptoxanthin bioaccessibility increased 10.1%, demonstrating stability during digestion. These results highlight orange peel-HDES extracts as promising functional ingredients for the food industry, with green extraction methods further enhancing carotenoid recovery.

## 2.6 Advanced Extraction Techniques

The extraction of bioactive terpenoids from citrus peels is traditionally performed using conventional techniques such as cold pressing, hydro-distillation, or steam distillation [46], while phenolic compounds are commonly obtained through solid-liquid extraction with hydroalcoholic solvents [47]. However, these conventional methods often fall short of the twelve principles of Green Analytical Chemistry, due to their low extraction efficiency, limited selectivity, and the use of large solvent volumes, which increase processing costs and require additional removal steps [48]. To overcome these limitations, more sustainable and efficient extraction techniques have been developed. Advanced methods applied for isolating phenolic compounds from citrus peels include ultrasound-assisted extraction (UAE) [49], microwave-assisted extraction (MAE), infrared-assisted extraction (IRAE), enzymatic-assisted extraction (EAE), and pressurized liquid extraction (PLE) [50] [51]. Recently, these techniques have been combined with natural deep eutectic solvents (NaDES), a new generation of environmentally friendly, non-toxic, and easily prepared solvents. NaDES are composed of two or more natural components, including a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA), forming a eutectic mixture with a melting point lower than that of the individual constituents [52]. Supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>) extraction is recognized as a highly sustainable method for recovering non-polar compounds, owing to the non-toxic, inert nature of CO<sub>2</sub>, its ease of acquisition, and the ability to

recirculate the solvent. This approach has been successfully applied to extract essential oils from various citrus peels, including orange and tangerine [53]. Hydrophobic bioactives like carotenoids, however, require specialized solvents and strategies to maintain stability and bioavailability.

### 2.6.1. Ultrasound-Assisted Extraction of Carotenoids Using Olive Oil

The extraction of carotenoids from citrus peel waste using olive oil as a green solvent in combination with ultrasound-assisted extraction (UAE) has been investigated by Wei *et al.* [54]. Under optimized conditions (55°C, solid-to-oil ratio 6:10, 70 min), carotenoid extraction yielded 157.07 µg/g. The resulting emulsion was stable, homogeneous, and showed moisturizing effects for up to 8 h, with the 8% carotenoid formulation providing the highest hydration. Antioxidant and antibacterial activities increased with carotenoid concentration, particularly against *Staphylococcus aureus* and *Escherichia coli*, while sensory evaluation indicated good consumer acceptance comparable to commercial products. These findings support the use of olive-oil-extracted citrus peel carotenoids as sustainable, bioactive ingredients in cosmetic emulsions. Citrus peel carotenoids including  $\alpha$ - and  $\beta$ -carotene, lycopene, lutein, and zeaxanthin exhibit strong antioxidant and health-promoting properties, making them valuable for food, pharmaceutical, and cosmetic applications. Compared with conventional organic solvents, vegetable oils offer a greener extraction alternative. Olive oil is especially effective due to its high carotenoid solubility, oxidative stability, and favorable composition, while ultrasound-assisted extraction enhances yield by promoting cell disruption. This green approach enables direct use of carotenoid-rich oils, reducing processing steps and supporting sustainable valorization of citrus peel waste, alongside other bioactives such as polyphenols.

### 2.6.2. Extraction and Membrane-Based Purification of Polyphenols

Solid-liquid extraction is commonly used to recover phenolics from citrus peel waste using food- and cosmetic-grade solvents such as ethanol, water, or their mixtures, with extraction efficiency influenced by solvent ratio, time, and temperature. Because crude extracts also contain pectins and sugars, purification is required. Membrane technology offers an effective, mild, and scalable method to separate and concentrate polyphenols, preserving their integrity while enabling energy-efficient and continuous processing through sequential membrane steps. Alonso-Vázquez *et al.* [55] applied an integrated membrane process to orange extracts, using ultrafiltration (UF) as a pretreatment to remove pectins and reduce fouling. The UP010 (10 kDa) membrane showed the best performance, removing ~50% of pectins while preferentially allowing flavonoids to pass into the permeate. Subsequent concentration by forward osmosis with a 58 g L<sup>-1</sup> NaCl draw solution achieved a 2.03-fold increase in polyphenols and an overall concentration factor of 1.47, with minimal losses to the draw solution. UF pretreatment and optimized membrane operation were highlighted as key steps for scalable, sustainable recovery of high-purity polyphenols from citrus peel waste.

### 2.6.3. Extraction of Flavonoids Using Heat Reflux and Ultrasonication

This study compared heat reflux (HR) and ultrasonication (US) using four solvents to extract flavonoids from navel orange peel and evaluate their biological activities [56]. The ethyl acetate HR extract showed the highest phenolic and flavonoid contents and the strongest antioxidant and  $\alpha$ -glucosidase inhibitory activities, while the ethyl acetate US extract was richest in polymethoxyflavones and exhibited superior antiglycation, antibacterial, and acetylcholinesterase inhibitory effects. Correlation analysis linked chemical composition with bioactivity, highlighting HR and US as simple, cost-effective methods to obtain bioactive, flavonoid-rich extracts for food, pharmaceutical, and cosmetic applications.

## 3. Valorization of Citrus Peel Waste

**Table 3** summarizes the main bioactive compounds in citrus peel, the sustainable extraction methods used to recover them, and their applications in food, nutraceutical, and cosmetic industries. It highlights the diversity of compounds such as polyphenols, flavonoids, carotenoids, and terpenoids and the use of green technologies, including deep eutectic solvents, ultrasound-assisted extraction, and membrane processes, underscoring the potential of citrus peel as a valuable by-product in a circular bioeconomy.

**Table 3.** Summary of citrus peel bioactives, sustainable extraction methods, and industrial applications.

Topic	Key Points	Applications
Citrus Peel Bioactives	Polyphenols, flavonoids, carotenoids, terpenoids, vitamins, fiber; antioxidant, anti-inflammatory, antimicrobial	Food, nutraceuticals, cosmetics, pesticides
Green Extraction (DESs)	Hydrophilic: polar compounds; Hydrophobic: non-polar compounds; food-safe, stable	Functional foods, supplements
HDES and Revalorization	Efficient carotenoid recovery; improves bioactivity and bioaccessibility	Functional additives in food, supplements, industrial applications
Advanced Techniques	Ultrasound-assisted, microwave-assisted, infrared-assisted, enzymatic-assisted, pressurized liquid, and supercritical CO <sub>2</sub> extraction, often combined with natural DES; improve yield, selectivity, and sustainability	Extraction of terpenoids, carotenoids, polyphenols, flavonoids for food, pharma, cosmetics
Carotenoid Extraction (UAE + Olive Oil)	Stable emulsions; antioxidant, antibacterial, moisturizing	Food fortification, cosmetics, nutraceuticals
Polyphenol Purification (Membranes)	Ultrafiltration and forward osmosis remove pectin and sugars; concentrates polyphenols about twofold while preserving bioactivity	High-purity polyphenols for functional foods, nutraceuticals, cosmetics
Flavonoid Extraction (HR and US)	Heat reflux with ethyl acetate maximizes phenolics, flavonoids, and antioxidant activity; ultrasonication with ethyl acetate maximizes polymethoxyflavones, antiglycation, antibacterial, and AChE inhibition	Food supplements, pharmaceuticals, cosmetic ingredients

### 3.1. Citrus Peel as a Rich Source of Bioactive Compounds

Citrus fruits such as oranges, lemons, and mandarins are among the world's most

important crops, with global production exceeding 124 million tons annually [57], yet their peels are largely discarded despite being rich in phytochemicals. Citrus peels contain phenolic acids, flavonoids, terpenoids, and alkaloids that exhibit antioxidant, anti-inflammatory, and health-protective effects, including antiviral and therapeutic activities, underscoring their potential as valuable sources of natural bioactive compounds.

Fruit processing generates substantial byproducts, with peels representing 30% - 50% of the total fruit mass [3]. Orange peel waste, a by-product of orange juice production, is generated in large quantities worldwide, with an estimated annual output of 8 - 20 million tons [58].

In the European Union, sweet orange (*Citrus sinensis*) cultivation is primarily concentrated in Mediterranean countries, including Italy, Spain, and Greece, with a total annual production of approximately 6.0 million tons according to recent USDA reports. In Italy, cultivation is mainly focused in the southern regions, with Sicily representing the largest area at 55,000 hectares [59].

Oranges can be categorized into two main types, white oranges, which are widely grown in many citrus-producing regions, and blood oranges, which require specific climatic conditions and are therefore cultivated in limited areas [60]. Blood oranges are valued for their deep reddish color and unique flavor. They are rich in bioactive compounds, especially phenolics and anthocyanins, which offer strong antioxidant benefits. The red pigmentation in blood oranges is influenced by the variety, rootstock, and environmental conditions, particularly the difference between daytime and nighttime temperatures.

Citrus fruit peels represent a plentiful but largely underexploited agricultural byproduct, containing a wealth of bioactive compounds with potential uses in cosmetics, food formulations, biodegradable pesticides, and diverse techno-chemical applications [24].

Several studies have highlighted citrus peels as a valuable source of bioactive terpenoids and phenolic compounds [61] [62]. Both terpenoids and phenolic compounds are noted for their antioxidant and anticholinergic activities, which may help prevent the onset of neurodegenerative disorders such as Alzheimer's disease. Their neuroprotective effects are primarily associated with the inhibition of acetylcholinesterase (AChE) and butyrylcholinesterase (BChE), thereby reducing acetylcholine depletion linked to Alzheimer's pathology [63].

Citrus peels are rich in polyphenols, flavonoids, carotenoids, vitamins, essential oils, fiber, and pectin, with antioxidant, anti-inflammatory, antimicrobial, and anticancer activities. They may also support cardiovascular, gut, glycemic, and bone health. Using peel-derived bioactives in foods and nutraceuticals provides a sustainable way to valorize waste, but efficient, green extraction methods and further research on safety, clinical efficacy, and regulation are needed.

### 3.2. Applications of Orange Peel By-Products

Orange peel, a major citrus by-product, is rich in dietary fiber, bioactive com-

pounds, and functional biopolymers, making it valuable for food, pharmaceutical, cosmetic, environmental, and material applications. Though high in moisture (80% - 90%) and organic content (~95%), which can pose environmental risks, its albedo contains cellulose, hemicellulose, pectin, and phenolics, while the flavedo is rich in flavonoids, carotenoids, and essential oils. These compounds can be converted into citric acid, oligosaccharides, bio-flavorings, bioethanol, and functional ingredients, offering antioxidant, antimicrobial, anti-inflammatory, antiviral, and cardioprotective benefits [64].

**Table 4** summarizes the key applications of orange peel by-products and their associated benefits.

**Table 4.** Overview of orange peel by-product applications across food, pharmaceutical, industrial, and environmental sectors, highlighting their key benefits.

Sector	Application	Key Benefits
Food & Nutrition	Gluten-free bakery, snacks, cereals	Improves fiber content, dough texture, antioxidant activity, and supports probiotics
Pharmaceutical & Cosmetic	Bioactive compounds, pectin, acne treatment	Antioxidant, anti-inflammatory, antimicrobial, antitumor, and prebiotic effects
Industrial/Material Science	Biopolymer composites, active packaging, meat preservation	Sustainable materials, antimicrobial/antioxidant packaging, natural meat preservatives
Environmental	Water treatment, heavy metal adsorption	Removes turbidity and Cr(III), low-cost eco-friendly coagulant

### 3.3. Food and Nutritional Applications

#### 3.3.1. Gluten-Free Bakery Products

Gluten-free (GF) diets have gained popularity globally, driving a market valued at nearly USD 6 billion in 2021 with projected annual growth of ~10% through 2030, especially in bakery products [65]. In response, this study explored the use of orange peel (OP) by-products as a functional ingredient in GF rice and corn flatbreads [66]. Orange peel (OP) powder, high in soluble dietary fiber, improved dough consistency and extensibility when added to pregelatinized rice (64 g/100 g) and corn (67 g/100 g) flours. At 0 - 9 g/100 g incorporation, optimal hydration (164 g/100 g for corn, 169 g/100 g for rice) reduced dough hardness and enhanced folding. The resulting flatbreads had higher dietary fiber and ash, intense yellow color, and differed in texture: corn-based breads were stiffer with lower gelatinization enthalpy, while rice-based breads were more extensible.

Gasparre *et al.* (2024) [66] demonstrated that orange peel residue serves as an effective nutritional and technological enhancer for gluten-free flatbreads, providing a sustainable and cost-efficient way to valorize citrus industry by-products. Incorporating sensory evaluations in future studies could further gauge consumer acceptance and support the development of fiber-rich, appealing gluten-free bakery products.

#### 3.3.2. Functional Foods and Health Benefits

Orange peel-derived products support functional nutrition by modulating im-

immune function, oxidative stress, and metabolic balance. Many health problems such as inflammation, digestive disorders, and oxidative stress arise from imbalances in the immune system and overall physiology. Functional foods aim to restore overall balance rather than target a single symptom. For example, diabetes management involves not only controlling blood sugar but also addressing diet, stress, emotional health, and body weight. Maintaining a healthy weight is crucial, as excess weight can raise LDL, total cholesterol, and triglycerides while lowering HDL, increasing cardiovascular disease risk [67].

The rising interest in healthier food choices continues to challenge food manufacturers to create products with improved sensory qualities and enriched levels of micronutrients and natural bioactive compounds. In this context, the present study investigates a novel approach for producing osmosonicated orange peel snacks enriched with plant-derived extracts during the osmodehydration process [68]. Extracts from purple corn, camu-camu, lucuma, carob, and prickly pear were used as hypertonic solutions, while a sucrose solution served as the control.

The formulated snacks had suitable moisture, high dietary fiber, and slightly reduced protein. Compared to sucrose-treated controls, snacks with plant extracts had lower carbohydrates and preserved or even enhanced bioactive compounds, with ferulic acid as the main phenolic. Antioxidant activity increased through osmosonication and thermal dehydration, while color varied with the extract used. Sensory evaluation showed high acceptance, with lucuma-enriched snacks rated most favorably.

Orange peel has broader functional applications in foods. In pearl millet biscuits, partial incorporation boosts fiber, phenolics, and antioxidant capacity, highlighting its nutraceutical potential. In jams, adding orange peel enhances dietary fiber, vitamin C, carotenoids, phenolics, antioxidant activity, and improves texture (elasticity, gumminess, chewiness). In probiotic yogurt, orange peel supports the survival of *Lactobacillus acidophilus*, *Streptococcus thermophilus*, and *Bifidobacterium* species during refrigerated storage.

### 3.3.3. Citrus Peel in Cereal and Snack Products

The breakfast cereal industry, valued at approximately seventy billion US dollars and growing at an annual rate of four point one percent, can benefit from orange peel incorporation [69]. It increases dietary fiber and bioactive compounds, reduces added sugars while maintaining taste and texture, and enhances nutritional and functional qualities to support health and reduce the risk of chronic diseases.

## 3.4. Pharmaceutical and Cosmetic Applications

The pharmaceutical and cosmetic industries have shown increasing interest in the recovery and purification of these compounds due to their recognized antioxidant, anti-inflammatory, and antimicrobial properties. Hesperidin, for instance, has potential therapeutic applications for conditions such as hypertension, hemorrhoids, varicose ulcers, varicose veins, and rheumatoid arthritis, and it has been reported to reduce pain and cholesterol levels [70]. Orange peel (OP) proteins

were hydrolyzed using *Aspergillus niger* WA 2017 protease to assess antioxidant and antitumor activities. Hydrolysates collected after 24 h showed the highest DPPH antioxidant activity. Single-factor optimization increased hydrolysate content 3.7-fold and DPPH activity 1.7-fold, while further optimization via Central Composite Design enhanced hydrolysate content 1.6-fold and DPPH activity 1.1-fold. Central trial samples achieved the highest DPPH activity (62.37%) compared to 20% in the control [71].

Comprehensive in vitro assays (DPPH, ABTS, FRAP, and reducing power) confirmed the hydrolysate's strong antioxidant activity. In vitro antitumor tests showed 60.62% cell death in Ehrlich Ascites Carcinoma cells. In vivo, tumor volume in untreated mice was 1.4 times higher than in hydrolysate-treated mice, and oral or intraperitoneal administration increased lifespan by 13.91% and 19.42%, respectively. These findings demonstrate that enzymatically derived orange peel protein hydrolysates are potent natural antioxidants with notable antitumor effects, supporting their potential in nutraceuticals and functional foods.

Natural food additives are increasingly sought after by consumers and the food industry due to their health-promoting properties. Flavonoids, abundant in various fruits and vegetables, have been widely recognized for their biological benefits. This study [72] isolated and characterized flavonoid fractions from the flavedo and albedo of orange peel to evaluate their potential as natural food additives. Water, ethanol, and ethyl acetate extracts were prepared, with total phenolic (TPC) and flavonoid (TFC) contents measured. Flavonoids were purified via column chromatography and analyzed by HPLC, identifying flavones, flavanones, and flavonols. The flavedo showed higher TPC and TFC than the albedo, and ethyl acetate was the most effective extraction solvent. The isolated fractions exhibited broad antimicrobial activity, cytotoxicity against cancer cell lines, and strong anti-inflammatory effects, highlighting orange peel as a valuable source of bioactive flavonoids for functional foods.

Acne is a chronic inflammatory disorder of sebaceous glands that can cause scarring and psychological effects. This study [73] optimized orange peel fermentation using *Aspergillus oryzae* var. *effuses* AS3.2825 and evaluated ethanol extracts for acne-related activity. After 96 h, the extract had the highest flavonoid and phenolic contents (7.52 mg/g). HPLC identified hesperidin, narirutin, nobiletin, and sinensetin, with hesperidin increasing from 0.239 mg/g to 1.115 mg/g post-fermentation. The fermented extracts modulated serum sex hormones, reduced inflammatory markers, and lowered triglycerides, with the high-activity fermented extract (HAG) showing the strongest effects, highlighting its potential for acne treatment.

Dietary fiber from orange peels, rich in indigestible polysaccharides, has been used to develop prebiotic foods that support gut health. It helps balance intestinal microbiota, regulate lipid metabolism, and promote healthy fecal bulk. As a widely discarded byproduct, orange peels provide an inexpensive and sustainable source of beneficial fiber [74].

When fermented by gut microbes, orange peel fiber produces short-chain fatty acids that improve intestinal health by lowering gut pH and limiting harmful bacteria. There is growing demand for affordable dietary fibers that promote probiotic activity and a healthy gut microbiome, which is crucial for nutrient absorption, immune support, gut barrier function, and detoxification. Diet strongly influences microbial diversity, balance, and activity.

Orange peels support probiotic growth and enable microbes to convert peel polyphenols into compounds with enhanced health benefits. Combined, they can form synbiotic products that promote wellness. In contrast, typical Western diets high in refined sugars, trans fats, and sodium harm gut microbiota. Foods rich in fiber and polyphenols, like orange peels, are ideal for nurturing beneficial microbes.

Pectin, a complex heteropolysaccharide from orange peels, is a multifunctional biopolymer with applications in food and pharmaceuticals [75], including potential roles in cancer therapy. Despite being abundant and renewable, pectin remains underutilized. Its functional performance depends on structural characteristics such as molecular weight, degree of esterification, and functional groups. Pectin can serve as a texturizer in foods and as a bioactive compound or drug carrier in pharmaceuticals. Challenges in extraction, purification, and analysis exist, but research into sustainable, high-value pectin-based products is promising.

### 3.5. Industrial Applications

#### 3.5.1. Valorization for Eco-Friendly Biobased Polymer Composites and Active Food Packaging Materials

Eco-friendly polymer composites are increasingly important for creating sustainable materials with added functionalities [76]. In this study, fully biobased blends were prepared by melt mixing the bio-polyester poly(butylene succinate-co-adipate) (PBSA) with up to 20 wt% orange peel powder to produce active polymer composites. Orange peels, used as a natural filler, are abundant food waste by-products rich in phenolic compounds and exhibiting antioxidant activity, as confirmed by experimental assays [77].

Incorporating orange peel filler into PBSA composites slightly reduced crystallization temperature and crystalline fraction but had minimal impact on thermal stability. Mechanical testing showed increased elastic modulus, decreased tensile strength, and unchanged elongation at break. The filler also imparted antioxidant and antibacterial properties absent in the pristine matrix, highlighting the potential of orange peel-enriched composites for functional, sustainable materials. Sustainable management of citrus waste, especially orange peel, is increasingly explored for eco-friendly applications like paper and food packaging. Abundant, biodegradable, and rich in biopolymers, essential oils, and aroma compounds, orange peel offers valuable valorization opportunities. This review examines orange peel composition and the use of its powder, extracts, and biopolymers (cellulose, hemicellulose, pectin) in sustainable packaging and paper production, including surface-engineered cellulose for improved performance. By combining functional

biopolymer extraction with nanocellulose reinforcement in polymer matrices, mechanical strength, thermal stability, and barrier properties of biodegradable films and coatings can be enhanced. Overall, orange peel waste presents a renewable resource for innovative, industrial-scale sustainable packaging solutions [78].

This study [79] developed orange peel extract (OPE)-loaded biopolymer films using Arabic gum (AG) and carboxymethyl cellulose (CMC) via casting, evaluating their bioactivity, mechanical, and barrier properties for active food packaging. GC-MS analysis confirmed that OPE contained flavonoids and phenolic acids, contributing to strong antioxidant activity (DPPH 100%) and antibacterial effects (inhibition zones: 31.7 mm *Salmonella enterica*, 29.0 mm *E. coli* O157). Three film formulations AG/CMC, OPE4@AG/CMC, and OPE6@AG/CMC were tested, with OPE6@AG/CMC showing the highest total phenolic content (165.3 mg GAE/g) and FRAP antioxidant activity (453.8  $\mu$ M Trolox/mg). Antibacterial tests revealed the greatest inhibition (26.33 mm *S. enterica*, 23.67 mm *E. coli*, 19.0 mm *S. aureus*) and rapid bacterial growth reduction within 12 h. Toxicity assays confirmed excellent biocompatibility. OPE addition also improved water vapor and oxygen barrier properties, enhancing suitability for food preservation. Overall, OPE6@AG/CMC demonstrated superior antioxidant, antimicrobial, and barrier performance, highlighting its potential as a sustainable, functional active packaging material.

Converting waste into valuable products addresses solid waste challenges while benefiting the environment and human health. This study [80] developed biocomposite films using pectin reinforced with eggshell microparticles (ESMP, 0.10 - 0.20 g/150 mL) and orange peel essential oil (OPEO, 14% - 36% w/w). Five films were fabricated via particle dispersion and solvent casting. ESMP and OPEO increased film thickness and altered transparency. Micromorphology and spectroscopy showed enhanced hydrophobicity and structural compactness, with reduced free OH bonds, sharper CH signals, and stronger C=C, C-O, and C-O-C bonds. Films exhibited improved thermal stability and tensile strength, slightly reduced flexibility, excellent moisture barrier properties, and effective antioxidant and antibacterial activity against *Staphylococcus aureus* and *Bacillus cereus*. Soil burial tests confirmed biodegradability, highlighting their potential for food packaging.

Meat products are prone to spoilage from lipid oxidation and microbial growth, driving demand for natural preservatives. This study [81] evaluated pomegranate (PPE), orange (OPE), and satkora (SPE) peel extracts in chicken meatballs stored at 4 °C for 21 days. PPE showed the highest antioxidant activity (DPPH 76.05%) and strongest antimicrobial effects against *Escherichia coli* (15.5 mm) and *Streptococcus agalactiae* (11.16 mm). Meatballs with PPE had lower peroxide values and TBARS, indicating superior lipid oxidation protection, and reduced microbial counts. PPE consistently outperformed OPE and SPE, supporting the use of fruit peel extracts as natural antioxidants and antimicrobials. Future studies should explore additional assays, sensory evaluation, synergistic effects, applicability to other meats, and stability of bioactive compounds under storage to enable large-

scale implementation.

### 3.5.2. Orange Peel Waste as Adsorbents for Chromium Removal and Turbidity Control

Hernández Maldonado *et al.* (2021) [82] investigated the adsorption of Cr(III) from aqueous solutions using treated orange peel (OPT) as an adsorbent at 15, 30, and 45 °C with adsorbent-to-solution ratios of 8 - 40 g L<sup>-1</sup>. The Sips model best described the adsorption isotherms, with a maximum capacity of 15.3 mg g<sup>-1</sup> and up to 95% Cr(III) removal. The process was endothermic and spontaneous, following pseudo-second-order kinetics. Structural and compositional changes in OPT before and after adsorption were analyzed via XRD, FTIR, UV-Vis, and SEM, confirming its dual role as a source of functional compounds and an effective Cr(III) adsorbent.

Surface water in developing regions often has high turbidity from suspended solids, typically treated with costly coagulants like alum that generate hazardous sludge. Orange peel powder, an agro-waste byproduct, was evaluated as a sustainable alternative for turbidity removal [83]. Characterization included proximate analysis (moisture 8.65%, volatile matter 67.35%, ash 6.72%, fixed carbon 17.28%), bulk density (0.45 g/cm<sup>3</sup>), viscosity (9 mPa·s), pH (4.3), conductivity (2.7 μS/cm), SEM, FTIR, and BET surface area (1.2 m<sup>2</sup>/g). Hydroxyl, carboxyl, and carbonyl groups were active in coagulation, supported by FTIR and pH<sub>pzc</sub> (~6.5). Response Surface Methodology (Box-Behnken Design) showed that pH, coagulant dose, and slow mixing time (SMT) strongly affected performance. Maximum turbidity removal (61.63%) occurred at pH 7, 1 g/L dose, and 40 min SMT. Although treated water showed reduced turbidity and suspended solids, turbidity (145.11 NTU), BOD<sub>5</sub>, COD, and coliform counts remained above WHO standards, indicating a need for post-treatment. The study highlights orange peel as a low-cost, sustainable coagulant for decentralized water treatment.

## 4. Future Perspectives

Future research should optimize green extraction and bioprocessing to enhance orange peel compound yield, stability, and bioactivity for scalable, cost-effective use. Orange peel extracts can be incorporated into functional foods, beverages, and supplements, while its biopolymers support sustainable packaging and biocomposites. Isolated flavonoids, aglycones, and protein hydrolysates may offer therapeutic benefits, and orange peel can aid water purification. Integrating these applications in biorefinery approaches promotes waste valorization, circular economy practices, and expanded industrial and health uses.

## 5. Conclusions

Orange peel, a major by-product of citrus fruits, is a rich and versatile source of bioactive compounds including flavonoids, phenolics, carotenoids, vitamins, dietary fibers, essential oils, and polysaccharides. These compounds exhibit strong antioxidant, antimicrobial, anti-inflammatory, anticancer, cardioprotective, and

neuroprotective activities, making orange peel a promising raw material for functional foods, nutraceuticals, pharmaceuticals, cosmetics, and sustainable materials. Efficient extraction techniques such as hydrodistillation, microwave-assisted extraction, ultrasound-assisted extraction, enzymatic hydrolysis, and green solvents enable recovery of these bioactives while preserving their stability and activity.

Despite its potential, the valorization of orange peel faces several challenges. The variability in composition due to fruit type, cultivar, and processing conditions can affect bioactive content and functional performance. High moisture content and susceptibility to microbial spoilage create difficulties in storage and handling. Additionally, scaling up extraction processes in a cost-effective and environmentally sustainable manner remains a key hurdle. From an economic perspective, transforming orange peel from waste into valuable products offers opportunities to reduce disposal costs and generate additional revenue streams, but requires careful optimization of extraction methods, processing efficiency, and market integration.

Overall, orange peel represents a renewable and low-cost resource with significant potential for health, industrial, and environmental applications, provided that technical and economic challenges are strategically addressed.

## Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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