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A Review on Innovative Biotechnological Approaches for Upcycling Citrus Fruit Waste for Engendering Value-Added Bioproducts

Running head: Upcycling Citrus Fruit Waste into Value-Added Bioproducts

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SUMMARY

Around the globe, the cultivation of citrus fruits has greatly increased due to the rising demand among consumers. The citrus processing industry globally produces approximately 110 to 120 million

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tons of citrus waste annually. This in turn contributes to landfills, and pollution and poses a risk to human health and the ecosystem**.** Proper utilization of citrus waste helps reduce environmental pollution and also acts as a sustainable source for producing different bio-based products. Abundant bioactive compounds in citrus waste impart immense economic value for the production of various useful products. Furthermore, bioactive compounds found in citrus wastes exhibit diverse biological properties, including antioxidant, anticancer, antimutagenic, antiplatelet, cardio-protective, and antiviral activities. Instead of directly disposing, the upcycling approach of citrus wastes can be transformed into various value-added products including single cell protein, biopolymers, pectin, biofuel, biofertilizer and bioenergy. Citrus fruit peels serve as a cost-effective reservoir of nutraceuticals and represent an affordable dietary option for addressing degenerative disorders. The waste of citrus which is used as biofertilizers that are a rich source of phenolics, and carotenoids helps to increase the food's shelf life. The objective is to maintain economic viability and sustainability with the help of recent innovations in the economy. This review discusses recent advancements in the valorization of citrus fruit waste, highlighting innovative biotechnological approaches to extract valuable bioactive compounds such as limonene, flavonoids, and pectin. These compounds are applied in diverse industries, from food and pharmaceuticals to bioenergy. Techniques such as microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) demonstrate high yields and energy efficiency. Techniques in sampling, pre-treatment, phytochemical extraction, purification and identification of citrus fruit waste are also studied. Additionally, this review emphasizes the environmental benefits of waste valorization as part of a circular economy approach, contributing to both economic sustainability and pollution reduction.

Keywords: citrus waste; sustainability; bioactive compounds; antioxidants; biorefinery; green extraction techniques; circular economy

INTRODUCTION

Citrus fruits belonging to the *Rutaceae* family are produced throughout the regions in the tropics and subtropics (*1*). Brazil, China, and the United States are the largest producers of citrus fruit, and India, Mexico, and Spain are subsequent producers (*2*). Northern Myanmar and several southern parts of the Himalayas are known to be the true origin of Citrus (*1*). Important *Citrus* species in this family include the *Citrus sinensis* (sweet orange), *Citrus medica* (citron), *Citrus limon* (lemon), *Citrus aurantifolia* (lime), *Citrus paradisi* (grapefruit), *Citrus aurantium* (sour orange), *Citrus reticulata* (tangerine), and *Citrus maxima* (shaddock or pamelo) (*3*). Citrus fruits are rich in vitamin C, besides its composition also contains a wide variety of beneficial vitamins and nutrients including folate, thiamine, minerals, fiber, carotenoids, flavonoids, and limonoids. There is a lot of data that demonstrates that consuming citrus fruits could lower the possibility of cardiovascular disease and obesity and also helps in weight reduction. In 2020, the annual citrus production was found to be 124.4 million tons and China became the biggest contributor, with 28.66 % of overall citrus yield (*4*). Though the citrus fruits are consumed raw due to their health benefits, to some extent one-third of the overall production is being processed (*5*). Peels, seeds, pulp, and segment residues occupy 50 % to 60 % of the fruit's composition, and these are re-obtained after processing the fruit. Around 80 % of citrus peel contains moisture and is classified into mesocarp/albedo (a white soft layer) and flavedo/epicarp (colored outer surface). The source of pectin is albedo, while terpenoids are produced from flavedo. The polysaccharides in the cell wall of the peel are composed of three major components including pectin, cellulose, and hemicelluloses (*6*). The composition of citrus by-products including sugar, cellulose, hemicellulose, lignin, and pectin from various citrus species is illustrated in Fig. 1.

(Fig. 1)

Various phytochemicals are formed during the growing phase of the fruit, which includes relatively low molecular mass phenolics, terpenoids, stilbenes, acetophenone, flavonoids, and tannins (in condensed form) (*7*).The peels of *Citrus sinensis* contain plentiful fiber, vitamin C, phenolics, and flavonoids, which serve as potent antioxidants. Nevertheless, these citrus residues are typically disposed of as waste. The by-products include seeds, pulp, and peels and these are considered to be harmful for the environment, human health and aquatic life, when not disposed properly due to their organic nature (*1*). Conventional methods for managing citrus peel waste include depositing it in landfills, utilizing it for composting, extracting pectin, and using it as animal feed. But some of these alternatives are not eco-friendly and cost effective. So there is a need to address this issue (*8*). Citrus are rich in bioactive compounds. Phenolic compounds in citrus peels act as antioxidants and prevent free radical damage to cells (*8*). Flavonoids and plant secondary metabolites have anti-cancer, anti-inflammatory, and neuroprotective properties. Studies have revealed that flavonoids are associated with a decreased risk of developing inflammatory bowel disease (IBD) and other degenerative disorders. Essential oils found in the citrus fruits are categorized majorly into oxygenated monoterpenes, oxygenated sesquiterpenes, hydrocarbon monoterpenes, and hydrocarbon sesquiterpenes (*9*). With the growing popularity of bioactive components and the concept of functional foods, foods enriched with citrus peel have come onto the market (*10*). Citrus peel wastes are also exploited for the preparation of enzymes and bio-flocculants. On the other hand, Citrus seeds are made up of two main constituents: (i) protein and (ii) seed oil, in which protein constitutes about 14 % and 36 % of seed oil (*11*). Citrus seeds possess significant nutritional value and oil content, making them valuable for use as food supplements or pharmaceuticals (*3*). Citrus wastes are best suited as dietary supplements as these non-edible components are inexpensive and easily accessible (*12*). The fruit of *Citrus bergamia*, Bergamot is exclusively employed for the production of essential oils that have been extracted from its peel. Due to its notable antibacterial and antiseptic properties, citrus finds extensive application in the pharmaceutical industry. Meanwhile, citrus waste is utilized as a fragrance creation in cosmetics applications, such as in soaps and perfumes. In the food industry, it contributes to aroma enhancement in products like teas, confectioneries, and liquors (*13*). Different extraction techniques have been employed to extract these compounds from citrus waste.

In order to reduce the waste, the citrus byproducts can be utilized for the production of bioactive compounds and value-added products. The predominant methods for extracting essential oils and extracts usually involve hydro and steam distillation, solvent extraction, and cold pressing. However, newer and more environmentally friendly extraction techniques, such as microwave extraction, ultrasound extraction, and supercritical fluid extraction, have come into use as they consume less energy and solvent, committing to sustainability. These methods are also employed for extraction of other bioactive compounds (*14*). This review provides knowledge on effective utilization of citrus waste for obtaining bioactive compounds. Further compounds are identified, analyzed and utilized for its health benefits and its use in various industries. The main objective is to reduce waste, mitigate environmental pollution and promote a circular economy. Also, this review claims the potential of citrus waste to convert into bioactive compounds and value-added products. The methods involved in phytochemical extraction also ensure not only profitable but also eco-friendly. This aims for further development in innovation and research. By focusing on the latest advancements, this review contributes to closing these gaps, highlighting scalable techniques that minimize environmental impact.

CITRUS WASTE: CHARACTERISTICS AND COMPOSITION

Relatively, 33 % of produced citrus fruits are processed, which gives rise to 50–60 % of organic waste containing peel, pulp, and seeds (*15*). Wastes are commonly generated during the processing of citrus fruits which include solid (*e.g.* peels, seeds, rags, and sludge), liquid (*e.g.* cannery effluents, fruit-washing, and sectioning wastewater), and distilled effluents (e.g. citrus molasses, citric acid, and pectin effluents) are isolated by solid-liquid separation. The primary by-product resulting from citrus fruit processing is citrus peel waste.

The analysis of citrus waste shows notable variation in nutritional composition among peel, pulp, and seed. Citrus peel exhibits a moisture content of (76.43±0.61) %, with high crude fiber ((27.70±0.53) %) and crude protein ((12.43±0.20) %) levels. It also contains (2.85±0.26) % crude fat and 7.8 ± 0.01 % total ash (23–25). In contrast, citrus pulp contains more moisture ((85.7±0.0) %) but significantly lower amounts of crude fiber ((7.34±0.8) %) and protein (8.6±0.0) %). The pulp's fat and ash contents are 4.9 ± 0.0 % and 6.5 ± 0.0 %, respectively. Citrus seeds, although lacking moisture data, are characterized by a high crude fat content (52.0 %) and comparatively lower levels of fiber (5.5 %), protein (3.1 %), and ash (2.5 %) (*16*).

Traditional disposal methods of citrus processing waste such as incineration and landfilling are inadequate and complicated with environmental consequences, as these processes produce harmful methane gas, emit a foul odor, consume a lot of energy, and have slow reaction kinetics (*17*). Unauthorized disposal of citrus processing waste can pollute both soil and water bodies, in some situations, the aquatic environment can be destroyed, especially when there is insufficient water to properly dilute these wastes. In recent times, there has been ongoing research into various ways to utilize citrus processing wastes, aimed at lowering management expenses and preventing environmental degradation (*17*). This process is known as “waste valorization” in which upcycling these waste materials into new products by upholding and enhancing its value into renewable, chemicals, fuels and energy.

Citrus processing waste along with other vegetal matrices makes them good compost. Combining orange peel waste with the organic fraction of municipal solid waste (OFMSW) during composting resulted in a 37 % reduction in odor production, which contributes to mitigating soil erosion (*18*). An economical and straightforward approach for managing large quantities of waste produced by citrus processing industries is by incorporating citrus by-products into animal feed. Fresh by-products of citrus are high in carbohydrates and contain low lignin which comprises about 28.5 %, and 3.5 % respectively. This makes them ideal for ruminant animals' digestive systems and the fiber content helps to enhance the animal diet, which in turn enhances the meat quality (*19*). These by-products of citrus waste encouraged scientific interest in developing eco-friendly solutions due to their potential to be valorized.

A novel approach called the 'biorefinery' concept has emerged, emphasizing the recycling or reutilization of citrus by-products to meet increasing demands. These by-products and biomass are employed to extract bioactive compounds for the creation of value-added industrial goods through sustainable methodologies (*1*). At the heart of this concept lies the idea of converting various organic waste streams, including agricultural, industrial, and municipal wastes, into a diverse range of valuable products, such as biofuels, chemicals, and materials (*20*). The biorefinery concept applied to citrus waste involves the integration of various technologies and processes to extract and convert the diverse range of compounds present in the waste into a wide array of products. Citrus peels, for instance, are a rich source of valuable compounds such as essential oils, flavonoids, and pectin, which can be extracted and utilized in the development of food, cosmetic, and pharmaceutical products. Additionally, the residual biomass can be further processed to produce biofuels, biochemicals, and even biobased materials (*21*).

The key to the success of the biorefinery approach lies in its ability to maximize the valorization of all components of the citrus waste stream. The circular bioeconomy concept, which emphasizes the sustainable and efficient use of renewable resources, is closely aligned with the biorefinery approach. By incorporating the principles of the circular bioeconomy, the biorefinery concept can strive for zero waste, where every byproduct or side-stream is utilized to generate additional value-added products. Through the integration of various technologies, such as fermentation, hydrothermal processing, and extraction, the biorefinery can transform citrus waste into a diverse array of high-value compounds, thereby contributing to the transition towards a more sustainable and circular economy (*22*).

The biorefinery concept applied to citrus waste has the potential to address several environmental and economic challenges. By recovering and converting the organic matter in citrus waste into valuable products, the biorefinery can reduce the environmental burden associated with the disposal of this waste, which can otherwise lead to pollution and greenhouse gas emissions. Additionally, the biorefinery can create new revenue streams and employment opportunities, contributing to the economic development of regions with a strong citrus processing industry. The citrus waste valorization into valuable products demonstrates a waste reuse and recovery concept transitioning towards a circular economy (Fig. S1).

Extraction technologies of citrus waste

Conventional techniques like Soxhlet extraction, liquid-liquid extraction (LLE), infusion, maceration, and solid-liquid extraction (SLE), as well as essential oil extraction from citrus peel waste, requires high energy expenditures, prolonged extraction times, and additional reagents. Researchers explored microwave-assisted hydro-distillation (MAHD) as a method for extracting essential oil from moist citrus peel waste. This approach reduced costs, eliminated the need for preservatives, and improved process efficiency, suggesting it could serve as a viable alternative to traditional methods. Over the last decade, various integrated technologies including ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), pulsed electric field (PEF), and enzymatic hydrolysis were used to extract and separate valuable compounds such as carotenoids, polyphenols, and essential oils from citrus processing (*23*). Physical, thermal, chemical, and biochemical processes can be used for converting citrus waste into biofuel (*24*).

Solid-state fermentation (SSF) has been employed to extract antioxidant-rich biological components from citrus by-products such as orange, lemon, tangerine, and grapefruit. D-galacturonic acid is obtained by the hydrolytic action of pectinases on pectin present in the citrus pulp. This is a potential platform chemical in citrus waste biorefineries. The employment of solid-state fermentation (SSF) in such biorefineries significantly reduces the production cost of pectinases used in this process (*25*). The citrus peel waste valorization serves great potential in the bio-economy transition. Furthermore, the citrus processing industry's negative environmental impacts emphasize valorization strategies' importance. It is crucial to develop environmentally friendly valorization methods that promote an inclusive biorefinery system in order to mitigate adverse environmental impacts (*15*). MAE offers speed and energy efficiency, UAE balances fast extraction with minimal solvent use, and SFE provides highly selective and pure extractions with environmental benefits, albeit with longer processing times. Table 1 shows the extraction technologies of extracting different bioactive compounds.

(Table 1)

EXTRACTION OF BIOACTIVE COMPOUNDS FROM CITRUS WASTE

Processing of citrus waste and extraction techniques for phytochemicals

Citrus plants, being extensively processed fruits, generate abundant by-products rich in bioactive compounds such as pectins, essential oils, and both water-soluble and insoluble antioxidants. Some of these wastes are currently being valorized extraction methods, which support the idea of waste valorization. This approach not only enhances earnings but also yields high-quality bioactive compounds. Primarily, it is essential to identify the optimal source for the desired bioactive ingredient, considering that the quantity and distribution of different compounds vary significantly among citrus species. Although bioactive compounds are typically present in fruits and processing by-products, they can also be found in significant concentrations in discarded portions, aligning effectively with the objective of waste reduction in numerous scenarios. After selecting the material, for each matrix and compound, the extraction conditions and techniques must be optimized. In this context, it is not only the quantity of extracted compounds that is significant, but also the desired bioactive ingredients. Nevertheless, purification processes may involve the use of non-environmentally friendly solvents, leading to a considerable increase in overall production costs. After incorporating the extracted compounds into products, it is essential to evaluate their bioavailability and bio-accessibility, as well as consider interactions between the compounds and other components of the matrix that could impact their availability (refer to Fig. S2) (*26*).

However, several of this extraction procedure may have some major drawbacks such as extended extraction durations, high-temperature degradation of the chemicals of interest, and, healthrelated hazards (*27*). Table 2 (*28-42*) depicts the diverse bioactive compounds extracted from citrus waste, along with their origin, extraction method, and respective functions.

(Table 2)

Sampling

Sampling involves collection of samples from various regions. Choosing a sample depends on the climate and the type of product to be recovered. After collecting, the samples undergo drying or made to powder form and stored. Those sampling methods for citrus waste vary depending on region, product type, and desired outcome. The most efficient methods involve freeze-drying and powdering, as seen with *Citrus sinensis* and *Citrus aurantifolia*, which preserve bioactive compounds and are ideal for high-value extraction. Freeze-drying at –40 °C ensures long-term preservation of compounds, while air drying and blending, as used for *Citrus limetta*, are more accessible but may result in some degradation of volatile compounds. Each method is selected based on the specific goals of the valorization process, such as maximizing bioactive compound recovery or facilitating transportation for further processing. Sampling of citrus waste varies by source and region. Sweet orange from the UK was freeze-dried and ball-milled, then stored at −40 °C (*43*). In Bangladesh, *Citrus sinensis* was dried and powdered, stored at 4 °C (*44*). *Citrus aurantifolia* from Thailand was powdered and vacuum dried (*45*). Italian samples of *Citrus reticulata, C. japonica*, and *C. clementina* were homogenized with a mortar and pestle and stored at −20 °C (*46*).

Pre-treatment

The main objective of pretreatment is to remove the structural barriers and make the citrus waste matrix available for chemical or enzyme action. The enzyme action causes hydrolysis which leads to the formation of sugars. It is to ensure that there are no barriers which could hinder hydrolysis and this process should not produce any by-products. This process also ensures that there is no contamination. Pretreatment can be performed by physical (drying, maceration, grinding and freeze drying), chemical (solvent treatment, acid hydrolysis and alkaline hydrolysis) and biological treatment (enzyme treatment and microbiological treatment) (*47*).

Physical methods

Drying is a process for the removal of moisture and there are several drying techniques. Sun drying of citrus waste is performed by using solar energy. Though this process is economical, requires huge space, constant monitoring of drying rate, also the samples are prone to contamination. Hot air drying, also known as oven drying, is performed by providing constant air flow from inside to outside of the waste to prevent contamination. A modern method of drying consuming less time is Microwave drying, it's limited due to involvement of high temperature which could affect or degrade heat sensitive compounds. In freeze drying, the moisture content in the citrus waste is crystallized and removed by going through phases i.e.; liquid to solid and then to vapor phase (*48*). Another physical method is ball milling. This method is cost effective and environmentally friendly. The procedure involves increasing the surface area by reducing the particle size. Mill balls grind the citrus waste into powder form with the use of mill equipment (*49*).

Chemical methods

Acid pretreatment (e.g. sulfuric acid) is effective in disrupting lignocellulose structure by glycosidic linkage cleavage. This leads to conversion of polysaccharides into monosaccharides and oligosaccharides. Dilute acid pretreatment is beneficial when compared to concentrated acid, as it is cost effective, economic, environmentally friendly and also does not corrode as much as concentrated acid does (*50*). Alkali pretreatment is another chemical method which involves using chemicals like NaOH. The alkali action upon the citrus waste leads to swelling of lignocellulose, solubilizing lignin partially in the solution leaving the cellulose intact. A limitation in using alkali lies in the retention time i.e. the more the retention time, the more the recovery of solvent is expensive. Sonication pretreatment assisted by alkali pretreatment can also be used to disrupt the lignin effectively to decrease the retention time. The cavitation of bubbles at the interface of the biomass by sonication treatment ensures the faster disruption of the lignocellulosic matrix, especially when combined with alkaline treatment (*51*).

Biological treatment

Enzymes like cellulases, pectinases, xylanases, proteases or peptidases are being utilized for pre-treatment. Cellulases hydrolyse the cellulose while pectinases break pectin into monomeric units. Majority of the cell wall and hemicellulose is composed of xylan, hence xylanases hydrolysexylan. Peptide bonds can be broken down into peptides and protein with use of proteases or peptidases. This enzyme treatment is performed to make the substrate (citrus waste) available for microbes for fermentation process (*52*). Similarly, microbes producing enzymes can be made use for pre-treatment. Lignocellulosic biomass consists of lignin, cellulose and hemicellulose. *Pleurotus* spp. belong to white rot fungi produce enzymes like laccases, versatile peroxidases and manganese peroxidases have the ability to degrade lignocellulosic biomass (*53*).

Extraction of phytochemicals

Conventional techniques

Solvents like methanol, water, acetone and ethanol can be purposed for flavonoids, vitamin and phenolic compound extraction. Cold pressing of citrus seeds can be performed to extract lemon seed oils. This technique is eco-friendly and cost effective but has a disadvantage with lower yields compared to solvent extraction. Hydrodistillation involves boiling a sample with water in a flask. The water vapor along with the volatile citrus compound will be sent to a column and then to the water cooler where the vapor is cooled and collected as distillate. Essential oils can be extracted with this method (*54*).

Solvents used in extraction

Methanol is a highly polar solvent utilized for extracting phenolic compounds and flavonoids from citrus waste by breaking down cell walls and dissolving polar bioactive compounds. It is particularly effective in extracting a diverse range of phenolic acids, including those with significant antioxidant properties. Ethanol, another polar solvent, is employed for extracting flavonoids and phenolics and is preferred in food and pharmaceutical sectors due to its lower toxicity relative to methanol. Its efficacy is enhanced for flavonoid glycosides, which are prevalent in citrus peels. Acetone serves to extract both polar and nonpolar compounds, making it suitable for a variety of bioactive components, including flavonoids and carotenoids. Its intermediate polarity facilitates the dissolution of compounds that exhibit partial solubility in water and other solvents, yielding specific compounds like naringenin. Water, often used in conjunction with other solvents, aids in extracting water-soluble bioactive compounds such as vitamin C and certain flavonoids. It is also instrumental in techniques like ultrasound-assisted extraction and microwave-assisted extraction to improve the solubility of compounds in solvent mixtures. Hexane, a non-polar solvent, is predominantly used for extracting non-polar compounds such as essential oils and limonene from citrus peels. Its capacity to dissolve hydrophobic components renders it ideal for the extraction of volatile oils.

Recent advanced techniques

Microwave-assisted extraction (MAE) emerges as a highly efficient method for extracting phenolic compounds, with high extraction rates, rapid processing times, and good product quality, provided with reduced cost. Through the solvent, the heat irradiated from the microwave is transmitted to the citrus sample, where it absorbs the heat and produces moisture. The moisture evaporates due to which, a high vapor pressure is created. This leads to breakage of cell wallsand the release of determined bioactive compounds from the sample (*55*). Pulsed electric field (PEF) follows electroporation of cells. In this technique, electric field strength varying from 100 V/cm to 300 V/cm for batch process and 20 kV/cm to 80 kV/cm for continuous process is utilized. The electrical force leads to puncturing of the cell membrane which improves the cell membrane permeability. Hydrophilic pores are created by which; the bioactive compounds can be extracted. In Ultrasound assisted extraction (UAE), cavitation bubbles are produced, when collapses, cause shockwaves and inter-particle collision due to which fragmentation of cellular structure occurs. Pores are created in the cell membrane due to sonoporation and the structure of the cell membrane is broken down due to the shear force created by the cavitation. The bioactive compound is then released and gets dissolved in the solvent (*56*). A supercritical fluid (SCF) is a substance found at temperatures and pressures exceeding its critical point, where the usual differentiation between liquid and gas phases is not present. SCFs can diffuse into solid matrices like gasses, allowing them to dissolve substances much like traditional liquids. Since many Supercritical Fluid Extractions (SFEs) take place near the fluid's critical point, slight changes in pressure or temperature cause significant fluctuations in the fluid's density. The density-dependent selectivity in SFE procedures is responsible for the extraction of phytochemicals (*57*). The various extraction methods employed including the solvent used are given in Table 3 (*58*-*62*).

(Table 3)

Isolation and purification

The citrus waste is first pre-treated, and immersed in a solvent in a specific solvent-to-solid ratio to ensure complete coverage of the material. The mixture is agitated using a shaker or a stirrer for a predetermined amount of time, usually ranging from several hours to overnight, to allow the solvent to permeate the cell structure and dissolve the target bioactive compounds. After extraction, the mixture is filtered using filter paper or centrifuged to separate the solid residue from the liquid extract containing the dissolved compounds. The solvent is then evaporated using a rotary evaporator under reduced pressure to concentrate the bioactive compounds, leaving behind a semi-solid or powdered extract. There is a need to purify the isolated bioactive components for commercial purpose. Crystallography, chromatography, partition techniques etc. are available for product purification. Variety of chromatographic methods including size exclusion, gel filtration partition, adsorption and more, are available. The method of chromatography is chosen based on the component to be extracted. In chromatography, a stationary phase and a mobile phase is involved, through which, required components from a mixture of compounds can be separated. Thin layer chromatography can detect the presence of a phytochemical. A column chromatography is nothing but a column filled with a stationary phase. Sample is injected at the stationary phase and the separation process occurs through the mobile phase consisting of a solvent (*63*).

High pressure liquid chromatography (HPLC) is considered the best and safe method for obtaining phytochemical purity. HPLC consists of solvent delivery pump, injection valve, analytical column, guard column, detector and a recorder. The citrus sample is introduced into the carrier stream through the injector. The compounds are retained based on the physico-chemical interactions between analytes in the citrus sample and the stationary phase (*64*).

Identification and determination of compounds

Identification and determination involve determining the presence of a functional group, multiple bonds and rings, arrangement of carbon and hydrogen. The methods involved are as follows. High-performance liquid chromatography (HPLC) is used to separate molecules in the column, after which the molecules are eluted and detected. The detector detects the molecules based on the physico-chemical property of the compound. This signal is converted into a graphic output called chromatogram where the peaks are displayed (*65*). Mass spectroscopy (MS) identifies compounds according to molecular weight and structure. It comprised detection of functional groups, presence of multiple bonds and rings, hydrogen and carbon arrangement as well as full structural elucidation. The molecules are bombarded with electrons due to which ions get charged and the signal is detected and recorded in the form of percentage peak. Nuclear magnetic resonance spectroscopy (NMR) detects the presence of isotopes of hydrogen, carbon and protons. It also reveals the physical properties of bioactive compounds like array and number of the carbon atom (*66*).

Gas chromatography and mass spectroscopy (GC-MS), along with other methods such as liquid chromatography and mass spectroscopy (LC-MS) and fractionation, merges the capabilities of GC and MS (*67*). GC separates compounds, while MS offers precise detection and structural insights. GC excels due to its simplicity and superior separation abilities compared to methods like LC. For analyzing small, volatile molecules, GC-MS stands out for its unmatched sensitivity, efficiency, and productivity. D-limonene has been extracted as a major component from *Citrus maxima* Todarii and has been given in Table 4 (*68-71*).

(Table 4)

Bioactive compounds and their uses

Flavonoids

Flavonoids are found naturally in plants as secondary metabolites that have important biological properties. Flavonoids are essential for protecting plants from ultraviolet (UV) exposure (*72*). More than 60 types of flavonoids are present in various citrus plants, which are divided into flavonols, flavones, flavanones, polymethoxylated flavones, flavanonols, and anthocyanins.

Flavanones account for over 95 % of the total flavonoid composition in citrus-derived flavonoids. Recent findings have proven that the peel of most citrus fruits contains a notably higher concentration of flavonoids compared to the pulp, with levels diminishing as you approach the flesh (*73*). The peels of citrus were considered to be the source of polymethoxyflavones (PMFs) notably tangeretin, nobiletin, (3,5,6,7,30,40-hexamethoxyflavone), and sinesetin (3,5,6,7,8,30,40-hexamethoxyflavone). Polarity dependent flavonoid extraction was also performed. The process involves using a deep eutectic solvent (DES), a green solvent. Extraction was conducted through ultrasonic irradiation, where Deep Eutectic Solvents (DES) was introduced to 0.1 g of citrus peel. The resulting mixture underwent extraction under consistent conditions in an ultrasonic bath. Subsequently, the mixture was centrifuged at 10000 rpm for 10 minutes, after which the supernatant was collected and diluted with 30 % methanol, tenfold. This process yielded higher extraction amounts, approximately 65.82 mg/g of total flavonoids (*74*).

Flavonoids are utilized as a food preservative as they act as coloring and flavoring agents. Citrus flavonoids exhibit diverse biological effects such as antibacterial, antioxidant, anti-inflammatory, and antiviral properties. Recent studies suggest they may also reduce the risk of conditions like cancer, type 2 diabetes, neurological disorders, and osteoporosis. Flavonoids contribute to heart protection by mitigating oxidative stress and inflammation, inducing vasodilation, and modulating apoptotic processes in the endothelium. Moreover, they interact with lipid metabolism and inhibit platelet aggregation, thereby aiding in the prevention of various cardiovascular diseases (*75*).

Phenolic compounds

Phenolic compounds (PCs) are present in various parts of plants, including fruits, stems, seeds, leaves, and roots. In these plant structures, all phenolic compounds contain at least one aromatic ring with one hydroxyl group (*76*). Many phenolic components have been reported to be efficient antioxidants, anticancer, antibacterial, cardioprotective agents, anti-inflammation, immune system boosting, skin protection from UV radiation, and an effective constituent for medical and pharmaceutical use (*77*). PCs function as antioxidants due to their structure, which comprises a hydrogen atom and/or an electron, thereby disrupting the oxidation chain reaction of free radicals. Oxidative and nitrosative stress in organisms can lead to the production of free radicals, which attack cells and contribute to the development of various diseases such as atherosclerosis, cardiovascular disease, neurological disorders, cancer, hypertension, and diabetes mellitus (*78*). The majority of phenolic compounds in nature exist as mono- and polysaccharide conjugates containing one or more phenolic groups. Additionally, 277 phenolic compounds can be associated with esters and methyl esters. The structural diversity of phenolic compounds leads to a wide spectrum of such compounds found in nature. Currently, 8000 different structures of phenolic compounds have been identified. Polyphenols are involved in regulating diameter growth, pigmentation, and defense against pathogens. They also serve as signaling molecules to recognize symbionts. As natural antioxidants, polyphenols possess important characteristics such as inhibiting carcinogenesis and lipid peroxidation, as well as exhibiting antibacterial activity. They also act as naturally occurring phytohormones and directly constrict capillaries. Additionally, polyphenols stabilize ascorbic acid and have various other beneficial effects (*79*). Various extraction methods are utilized for extracting phenolic compounds, including conventional solid-liquid extraction (SLE), subcritical water extraction and liquid-liquid extraction (LLE), or accelerated solvent extraction (ASE), microwave-assisted extraction (MAE), supercritical fluid extraction (SFE), ultrasound-assisted extraction (UAE), and pressurized liquid extraction (PLE). The Soxhlet equipment is used in the traditional Solid-Liquid extraction (SLE) procedure. Solvent evaporation, a straightforward process, is performed, where the extract and an organic solvent are separated based on their difference in boiling temperatures, resulting in the isolation of the solvent and the final product in their purest forms (*80*). The extraction of total phenolic content from Persian lemon waste using Deep Eutectic Solvents (DES) resulted in yields ranging from 1.28 to 2.90 mg GAE/g OP. Among the DES tested, [Ch]Cl:Gly 1:2 showed the highest yield of 2.90 mg GAE/g OP (*81*). The process of ultrasound-assisted extraction involves the extraction of phenolic compounds from Persian lemon waste (PLW) using a 130W-rated Ultrasonic Liquid Processor. This processor is equipped with a 13 mm diameter probe and operates at a frequency of 20 kHz (*82*). In microwave-assisted extraction, the interaction of polar molecules like water with waves destroys citrus debris due to a rise in pressure and heat inside the cell walls. Because of the porosity, molecules were more easily transferred, increasing extract yield without affecting the chemicals (*83*).

Limonoids

Limonoids belong to the phytochemical family found predominantly in citrus fruits like lemon, orange, mandarin, grapefruit, lime, and bergamot. Limonoids are secondary polycyclic metabolites that are rich in oxygen and are chemically associated with terpenoids (*4*). Citrus fruits have so far produced 17 different forms of limonoid glycosides and 36 different types of limonoids. Numerous citrus fruit seeds contain limonin and nomilin at concentrations of around 6 mg/kg (*84*). The two categories of limonoids are limonoid glucosides and limonoid aglycones. Limonoid aglycones, which contribute to the bitter taste of citrus fruits and juices, are predominantly found in citrus peels and seeds, accounting for 80 % and 70 % of the total content, respectively. Through enzymatic action by limonoid glucosyltransferase, bitter limonoid aglycones are converted into tasteless limonoid glucosides. These glucosides, soluble in water, are mainly present in citrus fruit pulp and juice, comprising 61 % and 76 % of the total content, respectively (*85*). Despite this, citrus juices eventually develop significant bitterness after processing, a condition known as delayed bitterness, which has a detrimental impact on the standard and acceptance of fruit juice. As a result, many physical, chemical, and microbiological processes for debittering citrus juices have been developed to improve their quality and customer appeal. Benzene was used to extract limonoids, petroleum ether was used to precipitate them, and dichloromethane was used to crystallize them. Benzene is hazardous, so its use has been limited and forbidden in recent years.

Limonoid aglycones are low-polarity chemicals that are generally not soluble in water and using the reflux techniques by organic solvents, they can be extracted. Using open-column chromatography, limonoid aglycones are separated after extraction. Afterward, the process involves fractionation using silica gel chromatography, followed by additional purification through HPLC. Subsequently, spectroscopic techniques are employed to analyze the obtained results. In contrast, polar molecules that are obtained using polar solvents are limonoid glucosides. In recent times, contemporary methods for extracting limonoids from citrus waste, such as hydrotropic extraction and supercritical carbon dioxide (SC-CO_2) extraction, have been developed (*86*). Supercritical carbon dioxide (SC-CO_2) extraction allows the conservation of huge amounts of organic solvents, which is good for the environment. The expense of consuming energy to generate high pressure, on the other hand, restricts its practical applicability. The efficiency of a flash extraction technique for the large-scale production of limonin from *Citrus reticulata* Blanco seeds was investigated. This method demonstrated a limonin yield of 6.8 mg/g with a purity of 95 %, and a limonin recovery yield of 97.1 % (*87*). Limonoid aglycones and glycosides have been shown to possess a wide array of pharmacological benefits, encompassing anticancer, antioxidant, antibacterial, antidiabetic, and insecticidal properties (*88*). Citrus limonoids are considered to have anticarcinogenic properties because of their capacity to induce phase II enzymes. The phase II enzymes [NADH: QR (quinine reductase) and glutathione S-transferase (GST)] play a role in the removal of harmful metabolites. The various limonoids of citrus such as limonene, limonin, nomilin, defuran limonin, deacetyl nomilinic acid glucoside, deacetyl nomilin, limonin glucoside (LG), deacetyl nomilinic acid glucoside, limonin-7-methoxamine, and isoobacunioc acid have been shown to induce NADH:QR (quinine reductase) and glutathione S-transferase (GST) (*89*). Chemically induced neoplasia has also been observed to be inhibited in the foregut, small intestine, oral cavity, colon, skin, and lungs of animals. In addition, it inhibits breast cancer cells (BCCS) from proliferating *in vitro* (*7*).

Alkaloids

Alkaloids are primarily indole ring derivatives. Alkaloids are categorized as heterocyclic (typical) or non-heterocyclic (atypical) metabolites based on their skeletal structure. Alkaloids are abundant in citrus species. Alkaloids were also found to be more abundant in orange and lemon peel than other phytochemicals (*90*). In dried citrus peels, synephrine is the most prevalent alkaloid. Octopamine, tyramine, and n-methyltyramine are other alkaloids isolated from *Citrus aurantium.* Synephrine has a molecular formula of $C_9H_{13}NO_2$ and is a sympathomimetic alkaloid that is present in bitter orange extracts as a primary constituent (*86*). About 90 % of citrus phytoalkaloids are composed of it. Water and ethanol extracts can be obtained from the dried and unripe fruit of *Citrus aurantium.* Acriquinoline B and acriquinoline A were also extracted from *Citrus reticulata* in a similar manner (*91*). O-synephrine, m-synephrine, and p-synephrine are the three isomeric forms of synephrine. Young fruits have the highest content of p-synephrine, its concentration decreases as it matures. Its concentration ranges from 53.6 to 158.1 g per liter in juice, 1.2 to 19.8 mg per gram in dried fruits, and 0.20 to 0.27 mg per gram in citrus pulp. Synephrine is extracted from aqueous extracts with a strong cation-exchange phase using solid-phase extraction, and then derivatized with proper reagents. The p-synephrine derivative is analyzed utilizing chromatographic and spectroscopic methods like nuclear magnetic resonance (^{1}H and ^{13}C NMR), GC–MS, GC–FID, and LC-MS (*92*).

Alkaloids, prominent secondary metabolites present in citrus fruits, exert various beneficial effects on human health, such as neuroprotection, anticancer properties, antioxidant activity, and cardiovascular protection. For thousands of years, dried citrus peels have been utilized in China as an excellent anti-asthmatic treatment (*93*). Current studies have discovered that the alkaloid fraction of dried citrus peels possesses anti-asthmatic properties. Emetic, anticholinergic, anti-cancer, anti-hypertensive, sympathomimetic, myorelaxant, anti-viral, diuretic, hypnoanalgesic, anti-depressant, anti-tussigen, anti-inflammatory, and anti-microbial properties of alkaloids have been reported. p-Synephrine is also a stimulant that has been shown to have cardiovascular benefits as well as favorable effects on sports performance and energy expenditure, carbohydrate mobilization and hunger control, fat oxidation and weight loss, mental attention, and cognition (*94,95*).

Carotenoids

Carotenoids, a group of isoprenoid metabolites, are extensively distributed and synthesized by various photosynthetic organisms, including Cyanobacteria, algae, and plants, as well as certain non-photosynthetic organisms such as fungi, bacteria, archaea, and animals. The presence of carotenoids is responsible for the yellow, red, and orange hues observed in a variety of fruits and vegetables (*73*). Carotenoids are broadly classified into two main groups: hydrocarbon carotenoids often referred to as carotenes (examples include β-carotene and lycopene), and oxygenated carotenoids, commonly known as xanthophylls (such as β-cryptoxanthin, violaxanthin, and lutein). Citrus fruits were found to have more than 115 carotenoids and their isomers, contributing to their brilliant and appealing colors. Citrus peels are rich in carotenoids such as β-carotene, β-cryptoxanthin, zeaxanthin, α-carotene, and lutein (*96*). β-carotene serves as a coloring agent in commercially produced foods such as dairy products, pasta, margarine, and confectionery (*7*). Surpassing the USD 2000-million mark, the demand for carotenoids is rising steadily, and the worldwide carotenoid market is expected to rise from 2018 to 2023 at a rate of 4 % annual pace (*97*).

Carotenoids are traditionally extracted with the aid of organic solvents due to their hydrophobicity (*98*). Non-polar solvents such as hexane, tetrahydrofuran (THF), or petroleum ether are typically employed for extracting esterified xanthophylls or non-polar carotenes. Conversely, polar solvents like ethyl acetate, ethanol, and acetone are more suitable for extracting polar carotenoids (*99*). Various protocols for carotenoids extraction from natural sources are accelerated solvent extraction (ASE) (also called PLE: pressurized liquid extraction), supercritical fluid extraction (SFE), pulsed electric field (PEF) assisted extraction, enzyme-assisted extraction (EAE), and atmospheric liquid extraction (UAE: ultrasound-assisted extraction or with Soxhlet, maceration, MAE: microwave-assisted extraction). The primary function of the peel is to extract the carotenoids from citrus waste. Forty terpenoid carbon bonds (double and single) joined by the molecule's core make its structure. Because of their molecular structure, certain carotenoids have a greater affinity for polar solvents like acetone, while others prefer non-polar solvents such as hexane. However, these solvents pose significant challenges due to their harmful nature and difficulties in disposal. "Green" methods such as the UAE treatment have been developed using environmentally acceptable solvents for carotenoid extraction (*83*).

Ultrasound-assisted extraction (UAE) yielded a 40 % higher carotenoid concentration compared to conventional extraction (*37*). Ionic liquid combined with ultrasonic-assisted extraction utilized 1-butyl-3-methyl-imidazolium-chloride [BMIM][Cl] as an ionic liquid to extract carotenoids from citrus peel. This method yielded a greater total carotenoid content compared to traditional acetone extraction (*73*). In the food industry, extracting valuable nutrients and beneficial compounds from fruit by-products remains a notable challenge. To enhance extraction processes and maximize the extraction of carotenoids, green extractions employing GRAS solvents may serve as a useful alternative (*98*).

VALUE-ADDED PRODUCTS

Single-cell protein

Microorganisms cultivated in a deceased and dehydrated state on various carbon sources, serving as protein supplements, are known as single cell proteins (SCP) (*100*). SCP is composed of carbohydrates, fats, vitamins, nucleic acids, and minerals, and contains about 60 % to 82 % protein by dry cell weight. It acts as a source of a lot of necessary amino acids as it contains lysine and methionine, which are limited in most plants and animal foods. SCP is a nutritional supplement that can be used to substitute costlier sources such as beans and fishmeal. Citrus waste contains various nutrients; hence it shows great potential as raw material to produce SCP by fermentation (*101*). The different products from different sources of citrus waste are mentioned in Table 5 (*24,102-111*).

(Table 5)

SCP was produced by fermenting orange waste using *Saccharomyces cerevisiae* and *Aspergillus niger* as a source. SCP was obtained through batch cultivation using orange waste from species including *Citrus aurantium, Citrus sinensis,* and *Citrus paradisi* as the substrate. The crude fiber, crude fat, ash, and protein in fungal biomass for each batch were estimated and found to an increase in protein contents in *Aspergillus niger* (29.0 %,29.75 %, and 27.15 % for *C. aurantium*, *C. paradisi, and C. sinensis* respectively) when compared to *S. cerevisiae* (23.50 %, 22.50 % and 22.06 % for *C. paradisi*, *C. sinensis,* and *C. aurantium* respectively). The high monosaccharide content of *A. niger*, when compared to *S. cerevisiae*, resulted in a rise in protein content. SCP showed low amounts of crude fat, crude fiber, and ash content (*112*).

SCP was accomplished by production by submerged fermentation with different media like Fruit Hydrolysate medium (FHM), Supplemented Fruit Hydrolysate (SFH), Glucose Supplemented Fruit Hydrolysate (GFH) by *S. cerevisiae.* A high percentage (60.31 %) of protein was obtained when grown on Supplemented Fruit Hydrolysates (SFH). The increase in yield was due to the presence of a carbon source like glucose. Fruit hydrolysate medium (FHM) yielded 53.4 % of protein while supplemented fruit hydrolysate (SFH) yielded the lowest at 17.47 %. It indicates that low supplementation of nitrogen results in decreased SCP production (*113*).

Pectin

Pectin is a complex polysaccharide present in plant cell walls, commonly found in citrus waste and other by-products of fruit and vegetable processing. It mainly consists of a lengthy linear polymer of α-1,4-glycoside-linked D-galacturonic acid (GalA) known as homogalacturonan (HG). The content in mature fruits ranges from 3 to 7 % by dry weight and 0.1 to 1.1 % by fresh weight. Pectin can be used as a bulking and coating agent, as well as viscosity modifiers, chelators, water-soluble biodegradable films, and emulsifiers (*29*). It is a gelling and thickening substance used in the food industry. Detoxification, blood glucose reduction, and antidiarrheal properties are some of their medical uses. Pectin has been utilized for various purposes, including reducing blood lipoprotein levels, acting as an agglutinant in blood therapy, addressing high triglyceride and cholesterol levels, and potentially preventing prostate and colon cancer. It is also considered for treating gastroesophageal reflux disease (GERD) and diabetes, as well as for its potential in preventing poisoning from heavy metals such as lead and strontium (*114*). It may also help to reduce the risk of heart disease and gallstones.

Mano Sonication extraction was performed by using peels of *Citrus unshiu* to extract RG-I enriched pectin. The physicochemical and macromolecule properties of pectin were studied. When compared with conventional maceration extraction of (18.3 %) pectin, mano-sonication extraction showed a higher yield of (25.5 %) (*115*). Pectin was extracted from citrus pomace biomass utilizing mild organic acid like acetic acid. A high concentration of acetic acid (9 % v/v) was used and preliminary experiments were performed on citrus biomass for 2, 4, and 6 h. An increase in extraction yield was observed in 6 h (23.7 %) of treatment when compared to 2 h (16.4 %). Using a more environmentally friendly method, like MAE, to obtain pectin from *Citrus limetta* peel is an efficient way to turn waste into a useful food additive. Under optimal conditions, which consisted of 600 W microwave power, pH of 1, and a duration of 180 seconds, the highest yield of 32.75 % was achieved. FTIR and ^{1}H NMR spectra were used to validate the integrity of the pectin skeleton (*116*).

Essential oils

Essential oils (EOs) are aromatic compounds primarily located in oil glands or sacs distributed throughout the fruit peel, particularly in the cuticles and the outer colored layer known as the flavedo. These are insoluble in water but soluble in ethers, natural oils, and alcohols. Citrus EOs are a sustainable, natural alternative, and environmentally favorable when compared to chemical preservatives and other synthetic antioxidants widely used in food preservation, like sodium nitrates, nitrites, or benzoates (*86*). Citrus-based EOs are derived mostly from citrus peels, which are commonly disposed of as waste and contribute to environmental issues. Citrus oils extracted from discarded peels are not only environmentally friendly but may also be used in a wide range of applications including food preservation (*7*).

Citrus essential oils include around 200 constituents, including sesquiterpenes, terpenes, alcohols, esters, and aldehydes, and may be defined as a blend of oxygenated chemicals, terpene hydrocarbons, and non-volatile residues. Terpenes are unsaturated compounds that degrade rapidly when exposed to light, heat, and oxygen. To mitigate undesirable flavors, terpenes are often eliminated. They constitute approximately 80-98 % of the majority of citrus peel oils (*117*). Limonene, being the most volatile constituent of citrus essential oils, plays a crucial role in determining their chemical, physical, and biological characteristics. Its concentration in essential oils varies depending on the variety, ranging from 32-98 %. For instance, lemon essential oil typically contains 45-76 % limonene, sweet orange essential oil contains 68-98 % limonene, and bergamot essential oil contains 32-45 % limonene (*118*).

Cold pressing is the conventional process where essential oils can be extracted from citrus peels. Cold pressing removes cuticle oils and peels mechanically, leaving a dilute emulsion followed by centrifugation to extract essential oils (*119*). Simple distillation and steam stripping processes have been found effective in extracting oil components from oil-milled sludge. When citrus fruit peels are exposed to boiling water or steam evaporation, essential oils are released through distillation. The vapors of essential oil condense which are collected in a separate chamber called a “Florentine flask” (*86*). Essential oil extraction was performed with lemon peel waste by microwave-assisted extraction with a yield of 2.03 % ± 0.21 %. Results revealed the essential oil composition and detected the presence of 65.082 wt. % limonene, 14.517 wt. % β-pinene, and 9.743 wt. % γ-terpinene (*120*).

Activated carbon

Activated carbons were also made from the residue following pectin extraction and H_2PO_2 activation had a high methyl orange adsorption capacity. Many chemical activating agents, such as zinc chloride ($ZnCl_2$) and potassium hydroxide (KOH), are very corrosive and hence detrimental to the environment. Due to its low corrosion and nontoxicity, K_2CO_3 is considered environmentally benign. It has also been proven to be a highly potent activating agent to manufacture the activated carbon. Thus, activated carbon from citrus waste is useful for the production of high-performance super-capacitors, metal ion removal from wastewater, and the adsorption of pesticides from water (*121*).

Groundwater treatment and biowaste treatment were employed, which involves removing fluoride with $FeCl_3$-activated carbon extracted from *Citrus limetta* peel wastes (AC-CLPs). To prepare the $FeCl_3$-activated carbon (adsorbent) the biomass was activated with $FeCl_3$ and carbonized at 250 °C and 500 °C. The obtained adsorbents are denoted as AC-CLP250 and AC-CLP500. They analyzed the removal efficiency of fluoride by activated carbon. There was an increase in the primary fluoride concentration of about 0.0025 % and a decrease in the efficiency of removal of fluoride to 5.0 % for AC-CLP250 from 94.7 % and to 33.3 % for AC-CLP500 from 94.8 % (*122*).

Citrus waste packaging films

With the rise in food consumption, the demand for food packaging has increased as well. Currently, petrochemical-based plastic films are widely used as packaging materials due to their wide accessibility, water vapor permeability, heat sealability, and good mechanical strength. But these plastics pose disposal issues as they were produced using non-renewable resources and also, they are completely non-biodegradable. The foodstuff often gets contaminated by the toxic chemical ingredients which are present in the packaging material. Given the problems with plastic packaging, the development of biodegradable packaging is not only a practical need but also a vital environmental requirement (*123*).

Pectin is abundant in citrus peel. Different reinforcing agents can be used to make pectin-based edible films. Pectin is a water-soluble component that polymerizes into films when a film-forming solution is cast and dried. Pectin films have high mechanical strength and barrier qualities (oils and oxygen), but they have a weak moisture barrier. Citrus by-product cellulose is used as a glass fiber alternative and can add mechanical strength to biodegradable and bio composite films (*124*). The physical properties of the orange waste-based biodegradable film are similar to those of commodity plastic. In addition, the produced film had good antibacterial, thermal, and mechanical properties (*125*). Packaging films are considered good materials if they exhibit antibacterial, biodegradable, and antioxidant properties (*126*).

Recent research highlights the advantages of utilizing citrus waste by producing edible grapefruit pectin (GFPec) based films. This innovative film offers various benefits, such as antimicrobial properties for food packaging, prolonging the shelf life of perishable goods, and serving as eco-friendly alternatives to synthetic packaging materials. The oxidation of lipids in food can be controlled by antioxidants, hence why they are used to enhance the shelf life and the nutritional value of stored foods. Bio-based high-density polyethylene (bio-HDPE) films were developed using phenolic compounds available in the citrus wastes. Extrusion was used to melt-mix each natural additive (0.8 parts per hundred resin) of bio-HDPE, and the resulting pellets were then thermo-compressed as films (*127*).

Food-grade kraft paper

Kraft paper is commonly utilized for packaging flour, sugar, dried fruits, and vegetables (*128*). Utilizing renewable energy sources for food coating and packaging is imperative on a global scale, as opposed to relying solely on synthetic materials. However, paperboard and paper are biodegradable and have more mechanical strength, which is unsuitable for packing foods with high moisture content due to their ability to contain pores, which allows moisture and some gasses to pass through. The growing concern over plastic waste accumulation has sparked interest in developing biodegradable materials with moisture barrier properties. Studies have found that incorporating wax or zein into kraft paper can effectively enhance its quality by creating vapor barriers. The water permeability of a composite film made with lipid-hydroxypropyl methyl, cellulose, and wax is dramatically reduced (*16*).

Hydrophobic material based biological materials, like citrus wastes, improve water barrier qualities while avoiding mechanical property degradation and, ultimately, destruction. The leaf extract (10 mL) was utilized to treat the kraft paper (terpene and limonene hydrocarbons). Subsequently, the solvent was evaporated entirely at a temperature of 25 °C over a period of 24 h using a mechanical shaker (at 60 rpm). The components of the extracts were partially dissolved in the available space and dispersed among the cellulose fibers of the papers. This process resulted in the formation of a thin layer of extracts on the paper surface. Limonene is a mixture of isoprenoid-related chemical compounds that are extracted in large amounts by distilling the oil from citrus peels. The strength of kraft paper was enhanced upon application of a coating derived from dissolved expanded polystyrene waste (EPS) in limonene (*129*).

Biopolymers

Biopolymers are generated through intricate cellular metabolic pathways facilitated by enzyme-driven polymerase chain reactions. These biodegradable polymers find extensive utilization across various industries, with a notable emphasis on medical fields. Natural biodegradable polymers encompass polysaccharides like chitosan, cellulose derivatives, and starch, as well as protein-based polymers such as albumin, collagen, and gelatin. Synthetic biodegradable polymers, on the other hand, comprise polyamino acids, aliphatic polyesters, phosphorus-based polymers, poly (alkyl cyanoacrylates), poly-anhydrides, and acrylic polymers. Extraction or fermentation could be used to produce biopolymers from food wastes, irrespective of pretreatment, to obtain fermentable sugars using solid-state fermentation (*130*). Biopolymer scaffolds stimulate the regeneration of bone tissue as well as promote cell adhesion, differentiation, and proliferation. There are two types of biopolymers utilized as scaffolds: natural and synthetic. Using synthetic polymers offers benefits such as enhanced elasticity, degradation rate, and mechanical strength. Conversely, natural scaffolds exhibit favorable capabilities in interacting with host cells and tissues, have lower immunogenicity, and possess significant bioactive potential. D-limonene, present in citrus waste, is used as a flexible feedstock for producing biopolymers. Tert-butyl hydroperoxide (TBHP) is used to oxidize it, yielding limonene oxide and tert-butyl alcohol (TBA). When CO_2 and a catalyst β-diiminate zinc acetate complex co-polymerize with one another, poly limonene carbonate (PLC) is created (*131*).

Biopolymer manufacture was evaluated by mixed microbial culture. Citrus wastewater was used to produce biopolymers in an MBR (membrane bioreactor) system. They also produced biopolymers using acetate and the rate of acetate conversion to biopolymers. When CO_2 and a catalyst-diiminate zinc acetate complex co-polymerize with one another, poly-limonene carbonate

(PLC) is created. The concentration of biopolymer was found to be 0.56 mg COD/mg COD when fermented citrus wastewater was utilized. The findings indicate that citrus wastewater can serve as a cost-effective substrate, with a productivity rate of 56 %, comparable to that of acetate at 55 % (*132*).

Bio-energy

Citrus waste has been recognized as a viable biomass reservoir for the production of renewable energy and liquid fuels. Within the realm of renewable energy, bioenergy holds the predominant portion. Various methodologies have been developed and simulated, encompassing gasification, pyrolysis, grate-firing, biogas plants, hydrothermal liquefaction, combined quad generating plants, and biogas upgrading, aimed at generating electricity from biomass (*133*). The physicochemical characteristics of citrus wastes make them suitable for anaerobic digestion (AD), a process that reduces their organic content and yields valuable products such as biogas. Combining the energy provided by solid waste burning with the energy supplied by anaerobic digestion could yield enough steam to me*et al*l of the facility's process steam and power needs. According to a recent study, citrus waste that had been de-oiled after the essential oil extraction could generate 322.6 mL/g volatile solids (VS) of biogas at a 30 % Fenton dosage (*134*).

Biofertilizer

The irrational disposal procedures for fruit and vegetable waste (FVW) are all too common, posing a significant environmental and economic risk. Converting vegetable and fruit waste through anaerobic digestion into biofertilizers holds the potential to mitigate pollution and enhance soil nutrition simultaneously. The main issue in bioprocessing FVWs is the high carbon and low nitrogen content (*135*). The production of biofertilizer is another important application of citrus processing waste, which may be made by changing the pH, the ratio of C/N, and the water content of the waste to 6 respectively. Animal manure was used in the batch process for anaerobic digestion of olive and citrus industry waste. The feedstock content was substantially connected to the composition of the generated biofertilizer (digestate). The findings suggest that achieving significant volumes of biogas requires a specific blend of animal dung, citrus pulp, and maize silage. Additionally, the resulting digestate, rich in antioxidants, can be effectively utilized in agriculture as a fertilizer (*136*).

Biofuels

Biofuels are renewable fuel alternatives for reducing anthropogenic greenhouse gas emissions. As compared to thermochemical conversions, the by-products of citrus waste are much more suitable for producing biofuel through biochemical conversions as these citrus wastes contain high moisture content. The yields of biomethane and bioethanol from the citrus by-products are expected to vary between 300 to 600 mL and 50 and 60 L/ton of waste CH_4 per gram volatile solids (VS), respectively (*23*). When biofuels are compared with petroleum fuel or gasoline, they are projected to decrease carbon dioxide emissions by as much as 80 % (*137*). About 70 % of the citrus trash is carbohydrates and may produce 1200 million liters of ethanol around the world. A horizontal fixed-bed pyrolysis reactor (FBR) was employed to perform slow pyrolysis and torrefaction experiments within the temperature range of 200–650 °C, aiming to extract bio-oil and charcoal from orange and lemon peel wastes (*138*). Pyrolyzing the peel residues within the temperature range of 400–650 °C results in the production of high-energy biochar and tars (*139*). Of all the bio-based fuels, biodiesel is categorized as a type of fuel that is employed as a persistent replacement for diesel. The substantial oil content ranging from 27-52 % found in bitter orange seeds (BOSs) renders them an economically viable and dependable source for biodiesel production (*140*). The lemon peel oil (LPO) can be used as a substitute for diesel. They used lemon peel oil to mitigate NOx and smoke emissions. They formulated emulsified fuel samples labeled as $LPOE_1$, $LPOE_2$, $LPOE_3$, and $LPOE_4$, with $LPOE_2$ and $LPOE_4$ demonstrating enhanced stability compared to the others. The brake thermal efficiency was evaluated and found to be 32.41 % for $LPOE_2$ and 33.5 % for $LPOE_4$ (*141*).

VALORIZATION OF BYPRODUCTS

The valorization process, which aims to maximize the value and utilization of agricultural and industrial byproducts, involves a complex interplay of various stakeholders, each with their own unique perspectives, goals, and challenges. Farmers, for instance, are often the primary producers of these byproducts and may seek to find cost-effective and sustainable ways to manage and repurpose them (*142*). Meanwhile, industry players, such as food and beverage manufacturers, are interested in harnessing the potential of these byproducts as high-value ingredients or materials for their own products, contributing to a more circular economy. Policymakers, on the other hand, play a crucial role in shaping the regulatory landscape, incentivizing sustainable practices, and fostering an environment that enables the successful valorization of byproducts. Farmers, as the first link in the valorization chain, are faced with the challenge of managing the vast quantities of byproducts generated from their agricultural activities. These byproducts, which were previously viewed as waste, can now be transformed into valuable resources through the biorefinery concept, where they are repurposed into biomaterials, biochemicals, and biofuels (*143*). This shift in perspective not only offers farmers additional income streams but also contributes to the overall sustainability of their operations by reducing waste and environmental impact.

Industry players, on the other hand, are increasingly recognizing the potential of these byproducts as high-value inputs for their own products. Utilizing food waste and other byproducts as feedstock for the production of microbial oils, carotenoids, and other valuable compounds aligns with the circular bioeconomy concept, which aims to minimize waste and maximize the value of available resources (*144*). Policymakers play a crucial role in facilitating the valorization process by creating a regulatory environment that incentivizes sustainable practices and enables the successful commercialization of valorized products. Integrated product policies, such as those implemented in the European Union, create market-based incentives for companies to engage in the continuous supply and demand of valorized products, thereby encouraging the adoption of valorization practices across various industries. The successful valorization of byproducts requires the active collaboration and coordination of these diverse stakeholders. Farmers, industry players, and policymakers must work together to overcome challenges, share knowledge, and develop innovative solutions that enhance the viability and social acceptability of the circular bioeconomy (*22*).

Life cycle assessment of citrus waste valorization

Researchers have explored various valorization processes to recover and utilize the valuable compounds from citrus waste. These processes can be broadly classified into three main categories: extraction of specific compounds, conversion of waste into biofuels, and development of value-added products, such as food additives, dietary supplements, and packaging materials. The extraction of specific compounds from citrus waste has been a major focus of recent research. The outer peel of citrus fruits can be divided into two regions, the flavedo and the albedo, each of which contains different bioactive compounds. These compounds can be selectively extracted and purified for use in pharmaceutical, cosmetic, and other high-value applications (*145*).

In addition to compound extraction, citrus waste can also be converted into biofuels, such as bioethanol and biogas, through fermentation and anaerobic digestion processes. These processes not only valorize the waste but also reduce the environmental impact of waste disposal. Furthermore, citrus waste can be used to develop a variety of value-added products, including dietary fibers, pectin, and natural food colorants and preservatives. These products can be incorporated into a wide range of food, pharmaceutical, and cosmetic formulations, providing a sustainable and cost-effective alternative to synthetic ingredients (*86*).

The valorization of citrus waste is a complex process that requires a comprehensive life cycle assessment to ensure the economic, environmental, and social sustainability of the various valorization strategies. Such assessments should consider the entire lifecycle of the valorization process, from the collection and transportation of the waste to the production, distribution, and end-use of the recovered products. By implementing a circular economy approach to citrus waste valorization, the environmental impact of waste disposal can be reduced, while simultaneously creating new revenue streams and fostering sustainable development (*146*).

Industrial application of citrus waste valorization

One industry that has already embraced the potential of citrus waste is the agricultural sector. Citrus peels, which account for nearly 50 % of the fresh fruit mass, can be used as a natural source of antioxidants, dietary fiber, enzymes, and organic acids, finding applications as soil amendments and animal feed supplements. The cosmetics and personal care industries have also leveraged the unique properties of citrus waste. The essential oils and bioactive compounds extracted from citrus peels and seeds have been incorporated into a variety of products, ranging from fragrances to skincare formulations, capitalizing on their antimicrobial, anti-inflammatory, and anti-aging properties (*86*). Furthermore, the pharmaceutical industry has recognized the potential of citrus waste-derived compounds as sources of natural antimicrobials, anti-inflammatory agents, and antioxidants. The high levels of sugars present in citrus waste also make it a suitable substrate for bioethanol production, contributing to the development of sustainable biofuel alternatives (*96*). The versatility of citrus waste-derived products extends beyond these traditional applications. Recent studies have explored the use of citrus peel-derived compounds in the development of biodegradable polymers and functional materials, catering to the growing demand for eco-friendly, bio-based solutions across various industries (*83*). The valorization of citrus processing waste has emerged as a promising avenue for the development of a wide range of industrial applications. By harnessing the rich chemical profile of these by-products, researchers and industry players are transforming waste into valuable, high-value commodities, contributing to the transition towards a more sustainable and circular economy (*17*).

CURRENT CHALLENGES AND ETHICAL ASPECTS

The advantageous impacts of bioactive compounds in citrus fruits have primarily been explored through *in vitro* and *in vivo* studies, their therapeutic application remains constrained by the absence of sufficient clinical evidence. To establish their effectiveness in treating human disorders, further clinical trials focusing on aspects such as intake, metabolism, and cytotoxicity of these bioactive chemicals are necessary. The citrus processing industry generates significant amounts of waste, including peels, seeds, and pulp residues, which can lead to environmental concerns if not managed effectively. The utilization of citrus waste in various industries making them a potential resource for value added products presents both opportunities and challenges, which require careful consideration of ethical, and safety aspects.

The use of citrus waste in the food and pharmaceutical industries is subject to various regulations and guidelines to ensure product safety and quality. In the food industry, the incorporation of citrus waste as an ingredient or additive must comply with food safety regulations, such as those set by the Food and Drug Administration or the European Food Safety Authority. These regulatory bodies establish guidelines on the acceptable levels of contaminants, the use of food additives, and the overall safety of the final product. Similarly, in the pharmaceutical industry, the use of citrus waste-derived compounds must adhere to strict regulations governing the development and production of pharmaceutical and nutraceutical products (*147*).

Ethical considerations also play a crucial role in the utilization of citrus waste. The potential environmental benefits of diverting waste from landfills or incineration must be weighed against any potential negative impacts on local communities or ecosystems. For example, the extraction and purification of certain valuable compounds from citrus waste may require the use of solvents or other chemicals, which could have adverse effects on the environment if not properly managed (*148*). Moreover, the safety of the final products derived from citrus waste must be rigorously evaluated. Citrus fruits are known to contain certain compounds, such as limonoids and furocoumarins, which can have potential toxic effects or interactions with medications. Thorough testing and safety assessments are necessary to ensure that the use of citrus waste in food and pharmaceutical applications does not pose any risks to human health or the environment (*149*).

The consumer acceptance of products derived from citrus waste is hindered by the negative perception of "waste." Consumers may be reluctant to buy such products despite their superior nutritional and functional qualities. To mitigate this issue, it is vital to reframe perceptions by emphasizing the sustainability and eco-friendliness of these products, showcasing their role in waste reduction and environmental preservation. Another obstacle involves addressing consumer apprehensions regarding the safety and quality of citrus waste-derived products. There may be concerns about contamination or harmful compounds in these items. To address these issues, rigorous quality control and transparent communication about manufacturing processes and product safety are imperative (*22*). Moreover, the effective commercialization of citrus waste-derived products may necessitate innovative marketing strategies and consumer education on their advantages. By underscoring the nutritional, functional, environmental, and economic benefits, manufacturers can alter consumer perceptions and enhance the acceptance of these innovative products.

FUTURE PERSPECTIVES

Nanomaterial production from food processing citrus waste is a relatively new topic of study, with limited investigations on the utilization of citrus peel waste for nanomaterial production. A few large micropores in mesoporous cellulose from the orange peels were yielded with microwave treatment. In another investigation with citrus waste, nanocellulose with length and average diameter of 458 nm and 10 nm respectively, was recovered. The mandarin peel waste is used for obtaining purified cellulose, from which cellulose nanofibrils of width 2-3 nm were made. Additional literature describes the use of pectin-derived citrus peel waste (CPW) for creating microspheres and employing CPW as a substrate for the production of nano bacterial cellulose. As a result, the technologies that are most adaptable to the environment will thrive in the future.

Incorporating concepts such as circular bioeconomy and upcycling fruit waste into new resources is imperative for enhancing the sustainability of citrus waste utilization. The researchers explored various strategies for converting citrus waste into more valued products, like biopolymers, biofuels, and dietary supplements. By adopting circular economy principles, they emphasized the importance of closing the loop in citrus waste management, thereby reducing environmental impact and promoting resource efficiency.

Additionally, a life cycle assessment was conducted to scrutinize the social, environmental, and economic facets of upcycling fruit waste, particularly citrus waste. This assessment aimed to evaluate the environmental impacts of various waste management strategies, emphasizing the potential advantages of repurposing fruit waste into new resources. They emphasized the importance of considering social acceptability, environmental responsibility, and financial viability when implementing upcycling initiatives. Overall, these studies underscore the significance of incorporating circular bioeconomy principles and upcycling strategies into citrus waste utilization efforts. By leveraging research findings from online sources, policymakers, researchers, and industry stakeholders can develop innovative solutions to address environmental challenges and promote sustainable resource management.

CONCLUSIONS

Processed citrus waste represents a sustainable bio-waste remnant that has undergone several valorization researches to recover its energy and matter. The valorization of citrus waste combines eco-innovation, environmental responsibility, and financial viability into products such as single-cell proteins, essential oils, activated carbon, citrus waste packaging film, and food-grade Kraft paper offers significant economic potential by developing new markets, companies can get access to new and rising markets, as well as increase revenue and lower operating costs. However, challenges persist, particularly in the large-scale implementation of these technologies and the environmental impacts of waste disposal if not properly managed. Improper disposal of citrus waste can lead to soil, water, and air contamination, with harmful emissions of greenhouse gasses. The present review critically evaluated various valorization techniques, highlighting both conventional and non-conventional methods. Conventional processes, while effective, often involve drawbacks such as long extraction times, heat-induced degradation, and the use of toxic solvents. To address these limitations, modern extraction methods such as microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and supercritical fluid extraction (SFE) have emerged. These techniques are more efficient, require less time, and produce higher yields with minimal solvent use. Moving forward, there is a need for more extensive life cycle assessments and industrial-scale trials to evaluate the long-term sustainability and economic feasibility of these processes. Further research should focus on overcoming scalability challenges and optimizing biotechnological methods for broader application.

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CONFLICT OF INTEREST

The corresponding author (Kuan Shiong Khoo) would like to declare the following article was invited by Special Issue Editor (Jun Wei Lim) who has collaborated previously. Therefore, all authors have requested to mark him as a blinded Guest Editor during the peer reviewing process.

SUPPLEMENTARY MATERIALS

Supplementary materials are available at www.ftb.com.hr.

AUTHORS’ CONTRIBUTION

M. Divyasakthi contributed to the design of the study, data analysis, drafting and preparation of the article. Y.C.L. Sarayu contributed to the design of the study, data analysis, revising and preparation of the article. D.K. Shanmugam contributed to the data analysis, revising and preparation of the article. G. Karthigadevi contributed to the data analysis, revising and preparation of the article. R. Subbaiya contributed to the revising and preparation of the article. N. Karmegam contributed to the data analysis, revising and preparation of the article. J.J. Kaaviya contributed to the revising and preparation of the article. W. J. Chung contributed to the revising and preparation of the article. S.W. Chang contributed to the revising and preparation of the article. B. Ravindran contributed to the project management, data analysis, revising and preparation of the article. K. S. Khoo contributed to the project management, data analysis, revising and preparation of the article.

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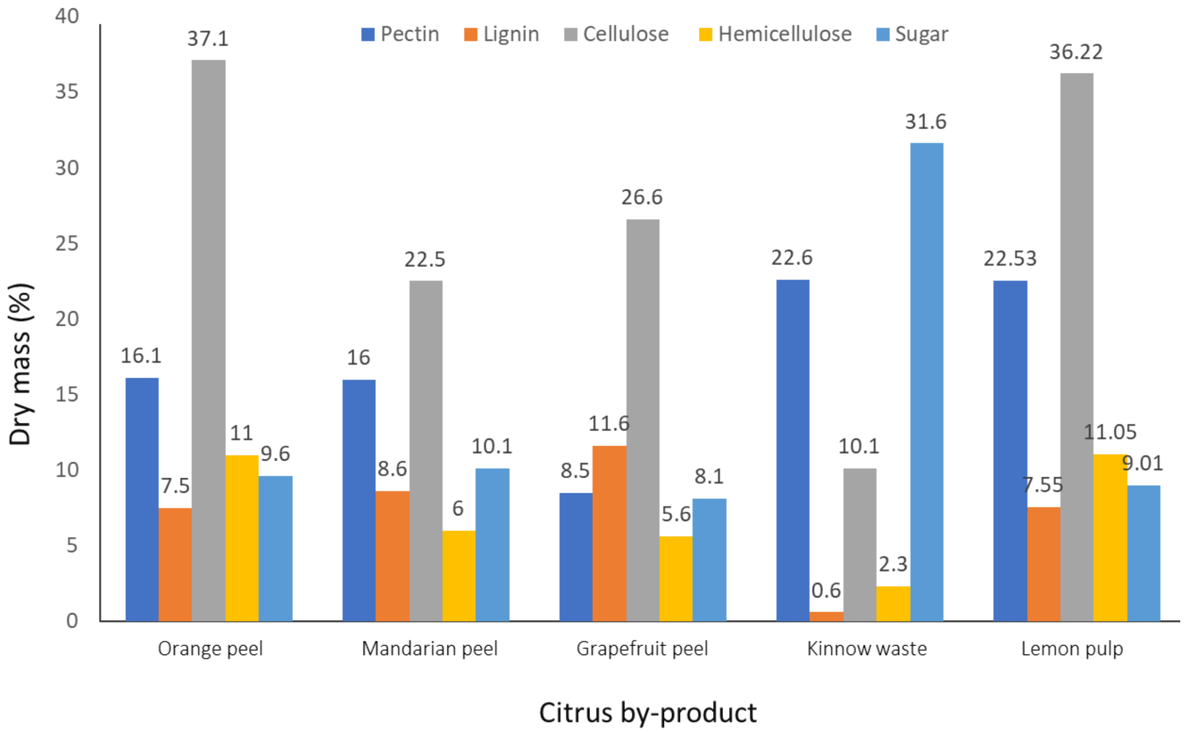

Fig. 1. Proximate composition of by-products in citrus fruits (*7,29*)

Table 1. Methods of extracting different bioactive compounds

Extraction Method	Principle	Solvent Used	Power/ Settings	Process	Advantage	Example parameter for citrus waste
Microwave-Assisted Extraction (MAE)	Microwaves cause molecular rotation and friction in polar solvents, rapidly heating the solvent and plant material.	Ethanol, Methanol, Water, Acetone-water or ethanol mixtures	Power: 300-900 W, Time: 5-30 min	Citrus waste is dried, ground, and mixed with a solvent, heated using microwaves, and filtered. Solvent is evaporated to concentrate extracts.	Fast extraction, higher yield, energy-efficient, reduced solvent use	Power: 600 W, Solvent: 80 % ethanol, Solvent-to-sample ratio: 10:1, Time: 10 min, Temp: 60-80 °C, Target: Flavonoids, phenolics, oils
Ultrasound-Assisted Extraction (UAE)	Ultrasound waves cause cavitation, which creates high energy, disrupting cell walls and releasing compounds.	Ethanol, Methanol, Water, Ethanol-water mixtures	Frequency: 20-40 kHz, Power: 300 W, Time: 10-20 min	Citrus waste is dried, ground, and mixed with solvent, subjected to ultrasound waves, filtered, and solvent is evaporated to concentrate extracts.	Efficient cell disruption, fast extraction, reduced solvent use, low energy consumption, retention of bioactive compounds	Frequency: 20 kHz, Power: 300 W, Solvent: 70 % ethanol, Solvent-to-sample ratio: 10:1, Time: 15 min, Temp: 25-30 °C
Supercritical Fluid Extraction (SFE)	Supercritical CO_2 dissolves non-polar	Supercritical CO_2, Ethanol as co-solvent	Pressure: 100-350 bar, Temp: 35-	Citrus waste is dried, ground,	Selective extraction, high purity, eco-friendly,	Pressure: 250 bar, Temp: 40 °C,

	compounds; ethanol as a co-solvent increases polarity to extract polar compounds.		80°C, Time: 30 min to several h	placed in an extraction vessel, and subjected to supercritical CO_2 and ethanol. The extracts are separated, and CO_2 is recycled.	no thermal degradation, broad range of compounds	Solvent: CO_2 with 5 % ethanol, Flow rate: 2 mL/min, Time: 1 h, Target: Oils, flavonoids, phenolics

Table 2. Bioactive compounds from citrus waste

Bioactive compound	Source	Method of extraction	Best performing solvent	Function	Reference
Limonene	*Citrus sinensis* peel	Solvent extraction	[C_2mim] Cl	Anticancer, Antifungal	(*28,29*)
Flavonoids	*Citrus reticulata* peel	Pulsed discharge extraction	HP-β-cyclodextrin	Anti-oxidant, anti-inflammatory, antimutagenic, antimicrobial, anti-carcinogenic	(*30,31*)
Flavonoids (Hesperidin)	*Citrus limon* pomace	Ultrasonic-assisted extraction	Water	Antioxidant, Antimicrobial	(*32*)
Flavonoids (Naringin)	*Citrus paradisi* peel	Supercritical Fluid Extraction (SFE)	CO_2 + EtOH	Anti-inflammatory, anti-cancer	(*33,34*)
Flavonoids Narirutin and hesperidin)	*Citrus reticulata* peel	Solid-phase extraction (SPE)	Ethanol	Antifungal	(*35*)
Phenolics	*Citrus sinensis* peel	Liquid-liquid extraction	Ethanol	Antifungal	(*36*)
Carotenoid	*Citrus sinensis* peel	Ultrasonic-assisted extraction (UAE)	d-limonene	Antioxidant	(*37,38*)
Pectin	*Citrus aurantifolia* peel	Microwave heating extraction	Citric acid	Anti-cancer	(*39,40*)
Pectin	*Citrus grandis* peel	Solvent Extraction	Deep Eutectic Solvent (DES) (lactic acid−glucose−water with a ratio of 6:1:6)	Antimicrobial	(*41,42*)

Table 3. Methods employed for extraction of phytochemicals

Extraction method	Source	Component used for extraction	Extracted phytochemical	*m*(sample)/g	Yield/%	Reference
Solvent extraction	Dry albedo of *Citrus sinensis*	Hot Methanol	Hesperidin	16.5623	2.8	(*58*)
Soxhlet extraction	Peel of *Citrus limon*	N- hexane	D-Limonene	50	3.56	(*58*)
Cold pressing	Seeds of *Citrus latifolia*	-	Seed oil	-	34.4	(*59*)
Hydrodistillation	*Citrus limon*	-	Pectin	100	16.58	(*60*)
Microwave assisted extraction (MAE)	*Citrus aurantium*	Acetone	Flavonoid	100	16.7	(*60*)
Pulsed electric field (PEF)	Peel powder of *Citrus unshiu*	Water	Narirutin	3	33.6	(*61*)
Ultrasound-assisted extraction (UAE)	Peel powder of *Citrus x sinensis*	Hydroethanolic mixture	Citric acid	100	6.4	(*61*)
Subcritical water extraction (SWE)	Peel powder of *Citrus unshiu*	Methanol	Hesperidin	1	5.0027	(*62*)

Table 4. Citrus compound identification techniques

Identification method	Equipment	Source	Compound identified	Reference
HPLC-QTof Mass Spectrometry	Alliance 2695 HPLC system (Waters Corporation, Milford, MA, USA) coupled to a QTof Premier mass spectrometer	*Citrus australasica*	Flavonoids, phenolic compounds, organic acid, limonoids and phenolic acids	(*68*)
NMR	-	Sweet orange	Naringenin, hesperitin, chrysoeriol, sinensetin, 3,5,6,7,3',4'-hexamethoxyflavone, nobiletin, 5-methoxysalvigenin, 3,5,6,7,8,3',4'-heptamethoxyflavone, 3,5,6,7,4'-pentamethoxyflavone and isosakuranetin	(*69*)
GC-MS	DB-5MS non-polar column	*Citrus maxima*	D-Limonene (21.72 %), β-linalool (19.58 %), 4-carene(7.90 %), *α*-terpineol (6.20 %), neryl acetate (4.64 %), geranyl acetate (4.70 %), and *γ*-terpinene (4.57 %)	(*70*)
LC-MS	Agilent 1290 LC system (Agilent Technologies, Palo Alto, CA, USA) coupled to a time-of-flight mass spectrometer (Agilent Technologies, Palo Alto, CA, USA)	*Citrus reticulata*	Ferulic acid, nobiletin, 3,5,6,7,8,3',4'-heptamethoxyflavone, kaempferol and hesperidin	(*71*)

Table 5. Products obtained from citrus waste

Source	Pretreatment	Process	Product obtained from citrus waste	Reference
Feedstock citrus pulp	Steam explosion	Fermentation	Animal feed	(*102,103*)
Citrus residue	Electro fluidic pre-treatment	Distillation	Essential oil	(*104,105*)
Citrus biomass	Acid-catalyzed steam	Gasification	Biofuel	(*24,106*)
Citrus peel in the combination with leaf extract	alkaline peroxide assisted and hydrothermal pre-treatment	Solvent evaporation	Food coating and packaging biodegradable plastics	(*107*)
Citrus peel	Acidification with mineral or organic acids	(Milling enzymatic inactivation extraction) Vacuum impregnation supercritical fluid extraction	Gelling agent	(*108*)
Citrus waste produced from juice extraction	Drying	Spray drying emulsification	Encapsulating agent	(*109*)
Citrus peel	Chemical activation using orthophosphoric acid (H_3PO_4)	Pyrolysis	Activated carbon	(*110*)
Citrus seed	Drying	Sodium methoxide-catalyzed transesterification	Biodiesel	(*111*)

SUPPLEMENTARY MATERIALS

Fig. S1. Citrus waste valorization into valuable products and transitioning towards a circular economy

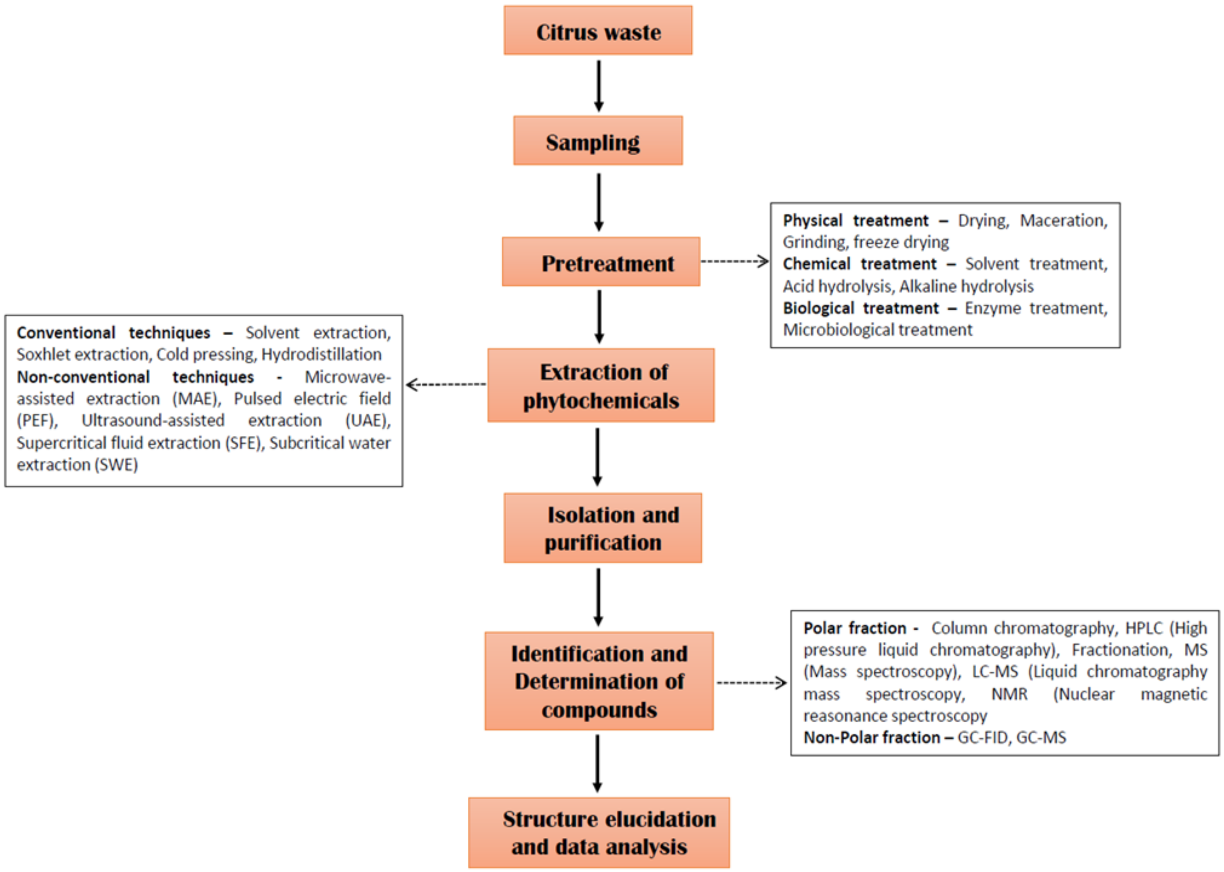

Fig. S2. Overall processing of citrus fruit waste and extraction of bio-active compounds