

Citrus Fruit Waste: A Treasure Trove for the Recovery of Pectin, Extraction Methods and its Multifunctional Applications – A Review

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Abstract

Citrus fruit waste, especially peels, represents a significant agro-industrial by-product rich in high-value compounds such as pectin. This review critically examines the potential of citrus waste as a sustainable source of pectin within the framework of circular economy and sustainable development. Various conventional and green extraction techniques—including acid hydrolysis, microwave-assisted, ultrasound-assisted, and enzyme-based methods—are discussed with respect to efficiency, yield, and environmental impact. The structural and functional characterization of extracted pectin using techniques such as FTIR, SEM, DSC/TGA, rheometry, GPC, and degree of esterification analysis is outlined.

Furthermore, the review highlights the multifunctional applications of citrus-derived pectin in food, pharmaceuticals, and biodegradable packaging, emphasizing its role in promoting zero-waste biorefinery concepts.

Keywords: Citrus waste, Pectin extraction, Circular economy, Sustainable development, Greentechnologies, Food industry.

I. INTRODUCTION

Wastes from fruits after being collected from their sources gets dumped at landfill sites or without pre disposal processing they gets incinerated. Because of this Ecology and Ecosystem faces several adverse effects. Dumping of fruit waste produces harmful gaseous

pollutants which includes hydrogen sulphide, methane, causing groundwater pollution (Khan et al., 2024). Air pollution, flue gas emission, ash production, results from direct fruit waste burning, which causes respiratory problems; Thus, the need for managing fruit waste sustainably arises (Sial et al., 2024). Waste valorization ensuring zero-waste human habitat is a solution which can tackle such issues. Valorization of fruit waste leads to the reduction in the quantity of waste generated and its adverse environmental effects (Rapa et al., 2024). For example- utilization of pectin in food industry, after its recovery and extraction from fruit waste. According to a study from Sweden, due to brown markings and bruising banana is the most wasted fruit (Mattsson et al., 2018). About 25-57 million tons per year of waste is generated from common fruits such as lemon, orange, watermelon, banana (Leong et al., 2022). In agri-food industry, fruits are used widely which generates huge quantity of wastes which includes seeds, pomace, rind, and peel (Ratu et al., 2023). About 15-60% waste accounts from fruit peel (Zhang et al., 2020). Waste from fruits processing also

have bioactive molecules which are valuable for example pectin, secondary metabolites, polysaccharides, and, essential oils (Russo et al., 2021). There are great opportunities for valorizing extraction of bio-based products that are having great potential in marketing. Extracting the valuable products from waste by fruit-processing; minimizes waste after recycling, promotes sustainability for the well-being of environment (Nadar et al., 2022). According to Wolfe et al., peels of apples in terms of their phenolic content contains upto 3300mg/100g of dry matter. Formerly it was reported that mango peels have total phenolic content which is roughly 32% higher than that of seeds and 13-47% higher than that of the flesh. Wastes of citrus fruits when compared with their final product has higher amount of bioactive content (Russo et al., 2021). Thus, by-products of citrus fruit waste are rich source of bioactive compounds such as phenols, pigments, minerals, dietary fibre, organic acids, and other molecules such as pectins etc; thus these bioactive compounds are known for having antimicrobial properties and are anti-

tumour and have other health benefits also (Russo et al.,2021).

In 1825, Henry Braconnot first described and isolated the term pectin (Devi et al.,2014). Pectin is a hydrocolloid, structural carbohydrate and a natural polymer which is explored widely among the other value-added products extracted from waste by fruit peocessing (Sarangi et al.,2023). α -(1 \rightarrow 4) linkages are present between the galacturonic acid units of pectin-a complex polysaccharide (Muhidinov et al.,2024). Pectins consists of galacturonic acid monomers which help in estimating its purity (Robledo et al.,2019). Worldwide annual production of pectin is rouhghly 40,000 metric tons. Pectins are found in the peels of fruits and provides strength to plant tissues (Picot-Allain et al.,2022). Citrus peel contains(25-35% -pectin on dry basis), apple pomace (10-15% - dry basis), sugar beet contains (10-20%) and sunflower (15-25% -dry basis) (Venkatachalapathy et al.,2021). On the basis of degree of esterification pectins are classified. percentage of carbonyl groups esterified with methanol is defined as degree of methylation (Naqash et al.,2021). High Methoxy

pectins are the pectins in which more than 50% carboxyl groups are methylated whereas Low Methoxy pectins are pectins in which less than 50% carboxyl groups are methylated (Liang et al.,2022). In an ammonia media de-esterification of low methoxy pectins produces amidated pectins (Noor et al.,2021). For commercial applications, purity required minimun is of 65% GaIA (Robledo et al.,2019). On the commercial basis, production of pectin is done from citrus fruit peels (Twinomuhwezi et al.,2023). Pectin extracted from lime peels gives high yield and is of better quality(Dranca et al.,2018).

II. CITRUS FRUIT WASTE AS A RICH SOURCE OF PECTIN

All over the world, citrus is most popular commercially grown and is the largest fruit crops grown. Huge amount of waste is produced by citrus processing industries that has major impact on enviroment. The citrus waste produced worldwide is more than forty million tons (Russo et al.,2021). Along with soluable sugars, cellulose and hemicellulose; fifty percent of the total

citrus waste by weight is represented by pectin (Zannini. D et al.,2021). Citrus waste is in the form of Rag, peel, and seeds which constitutes fifty percent of waste, which is a rich source of pectin, 54.82% TDS, 25.17% SDF and 29.65% IDF (Maqbool et al.,2023).

Pectin is extracted from waste of citrus fruits such as lemon (citrus limon), tangerine (citrus tangerina) and orange (citrus sinensis) peels (Twinimuhwezi et al.,2023). Scientific name of citrus fruits is Citrus.L, it belongs to the genus citrus, sub-family Aurantioideae and family Rutaceae (Zou, Z. et al.,2016) (Maqbool, Z. et al.,2023). They are nutritionally and commercially crucial fruits throughout the world (Rawat, N. et al.,2015) (Maqbool, Z. et al.,2023). They are considered as staple foods, health promoting, nutrient dense and energy dense fruits (Abirahim, A. et al.,2014) (Maqbool, Z. et al.,2023). In many studies, it is shown that, secondary metabolites and bioactive components present in citrus fruits are used as chemotherapeutics (Abirahim, A. et al.,2014) (Maqbool, Z. et al.,2023). Each year approximately 10 million MT waste is generated by citrus fruit processing globally (Zema, D.A. et al.,2018) (Suri,

S. et al.,2022). Waste generated in the production of citrus fruit juice is 50% of fresh fruit mass and waste of citrus fruit includes peels (50-55% of total fruit mass), seeds (20-40% of total fruit mass), pomace and wastewater; peels, pulp, seeds, spoiled fruits are portions covered under citrus wastewater (Zema, D.A. et al.,2018) (Suri, S. et al.,2022).

III. METHODS FOR THE EXTRACTION OF PECTIN

There are several methods for the extraction of pectin. These methods are classified as conventional methods non-convectional emerging green methods. Convectional methods of pectin extraction involve the presence of acid in the form of catalyst whereas non-convectional emerging green methods involves enhanced heating with or without acid (Kumar. S et al., 2022). There are certain limitations associated with convectional methods of extraction such as longer extraction time, potential impacts on environment, need of large quantity of solvents, and degradation of target pectin (Roman-Benn et al.,2023). Advanced extraction techniques can be used to address these

limitations as these methodologies preserves functional qualities of pectin, by accelerating extracton improves efficiency and minimized the use of solvent (Gavahian et al.,2021). Advanced extraction techniques are MAE (microwave-assisted extraction), UAE (ultrasound-assisted extraction), OHAE (ohmic heating-assisted extraction), EAE (Enzyme assisted extraction) and Supercritical water extraction (SCWE) (Kumar, S et al.,2023).

1.1.MICROWAVE-ASSISTED EXTRACTION

Dielectric heating having above 100 MHz electromagnetic frequency is termed as microwave heating (El khaled, D. et al.,2018). In microwave-assisted extraction, microwave radiation is absorbed by polar solvent which generates heat, mass transfer is accelerated cellulose or hemicellulose network complexes are broken thus protein is released (Guo, X. et al.,2024). Major advantages of MAE are- higher yield, low solvent quantity needed, temperature distribution is homogenous, extraction time is shortened, pectin de-polymerization is reduced (Ling, B. et

al.,2023). In a study by Asma and team conducted at the Collage of Agriculture / University of Tikrit where they had performed microwave assisted pectin extraction from citric acid and ammonium oxalate and studied their physical properties. In the study they concluded that range of viscosity values for two years which were expressed by the time of solution run-off lies between (1.5208 - 1.2161), (1.7250 - 1.6083), (1.6958 - 1.0875), (1.7416 - 1.5941), (2.1208 - 1.7875), (1.3916-1.3041) , whereas the pectin extracted from citric acid requires holding time of (8,7,8,7,8 and 7) minutes, while for ammonium oxalate time required is (10,6,7,8,7 and 6 minutes (Asma et al.)

1.2.OHMIC-HEATING ASSISTED EXTRACTION

ohm's law states that when a voltage is applied, thermal energy is generated, the amount of thermal energy generated is directly proportional to the electrical conductivity of the product (Von Meier et al.,2024). Ohmic heating takes place in a food or biological system when an electric field is applied across it, rapid heating, mass transfer takes place and pectin is released (Jan, B. et al.,2021).

Major advantages are- processing time is reduced, unintentional changes which occur in protein structure due to heat induced are minimized (Niakousari, M. et al.,2019). Limitations associated with ohmic heating is non-uniform conversion of electrical energy into heat (Kumar, T. et al.,2018). Process of ohmic heating is also known as efficient, homogenous and rapid heating (Makroo, H.A. et al.,2020). In a recent study, with the aim of analyzing extraction of pectin from peel of lemon by using ohmic heating, with purpose of evaluating coefficient of specific pectin production (SPPC, mg/KJ) and comparing it with traditional pectin production (TPP) (Cilingir, S. et al.,2021). Three different temperatures (70-90 degree celcius), three different times of holding during extraction (0-30 minutes) , three different ratios of solid:liquid (1:30, 1:40, 1:50) and a constant pH which can be adjusted with citric acid were used while carrying out extraction process; From the study it was found that the pectin yield was affected by extraction conditions; With the increase in temperature and extraction holding time, yield of pectin increases. Under the same conditions, yield of pectin in 30 minutes

by Ohmic heating assisted extraction process was similar to yield of pectin which was obtained from Traditional Pectin Process. The study concluded that ohmic-heating assisted method can be used as an alternative of Traditional Pectin Process (Cilingir, S. et al.,2021) .

1.3.ULTRASOUND-ASSISTED EXTRACTION

For phytochemical extraction, ultrasound in range varying from 20-100 KHz is used (Zabot, G.L. et al.,2021). In a liquid medium when ultrasound waves move, they are compressed and expanded, which results in hydrodynamic cavitation in the liquid or near material surface which is targeted; Hydrodynamics cavitation, resulting in solvent penetration into cell wall, facilitating mass transfer (Picot-Allain et al.,2022) (34). Advantages of UAE are- pectin yield is higher, extraction time is reduced specially when carried out in medium which is acidic (Picot-Allain et al.,2022) (31). Frequency, intensity, solvent type, temperature and sonication period are ultrasonic variables which are considered crucial for mproving the extraction of pectin with increase in sonication frequency, there is reduction

in amount of cavitation thus results in lowering the pectin yield (Picot-Allain et al.,2022) (32). It is also observed that pectin extraction improves with increased efficiency (Gavahian, M. et al.,2021) (33). In a study, aiming to reuse and recycle by-products of citrus, investigated the extraction of pectin from the peels of citrus limetta by employing the technique of ultrasound assisted extraction (Panwar, D. et al.,2023). Under optimum conditions the pectin which was extracted, was compared with commercially extracted pectin and was characterized for its physiochemical, antioxidants, functional, structural, thermal and apparent viscosity; After comparing it was founded that both the pectin from commercial and ultrasound assisted extraction were esterified with $59.71 \pm 0.12\%$ and $55.29 \pm 0.51\%$ degree of esterification; methoxyl content $7.06 \pm 0.10\%$ and $7.19 \pm 0.15\%$; High antioxidants and similar emulsifying properties and water/oil holding capacities were found in ultrasound assisted extracted pectin when compared to commercial pectin; Presence of GalA units were found in both samples which was proved by FTIR

spectra; Ultrasound assisted extracted pectin has high thermal stability when compared with commercially extracted pectin was showed by DSC and TGA; The study concluded that good quality pectin was extracted which can be utilized potentially in food sector as food ingredient (Panwar, D. et al., 2023) .

3.4. ENZYME-ASSISTED EXTRACTION

Release of pectin by the disintegration of plant cell matrix was facilitated by the enzyme's cellulose, pectinesterase, amylase, protease, alcalase, pectin lyase, hemicellulose, and xylanase (Maric, M. et al.,2018) (Kumar, S. et al.,2023). Due to their high specificity towards generation of target polymeric pectin fragments, enzymes degrading polysaccharides such as endo- and exo-rhamnogalacturon hydrolase, rhamnogalacturon lyase, polygalacturonase, endo-xylogalacturonan hydrolase are considered as suitable tools for elucidating structure of pectin (Rodriguez, A.V. et al.,2019) (Kumar, S. et al.,2023). Endo-xylanase and endo-cellulase enzymes at 40 degrees Celsius, pH-5.0 for 10 hours with constant

shaking was used to extract pectin from apple pomace; Endo-xylase gave higher yield (19.8%) when compared to endo-1,4- cellulase with lower yield of (15.2%) (Wikiera, A. et al.,2016) (Kumar, S. et al.,2023). Advantages of EAE are- harmful chemicals are not used during the process, less wastewater generation, highly pure pectin is yielded. Disadvantages are- it is costly and time consuming (Kumar, S. et al.,2023).

3.5 SUPERCRITICAL WATER EXTRACTION

Supercritical Water Extraction is also known as pressurized hot water extraction or superheated water extraction. Without undergoing any phase change, liquid water maintained at critical temperature 374 degree Celsius and critical pressure 1.0-22.1 MPa is known as supercritical/subcritical water (Liew, S.Q. et al.,2018) (Kumar, S. et al.,2023). Supercritical water extraction, uses supercritical water as solvent for extracting pectin, this supercritical water which is used as solvent is bears generally recognized as safe (GRAS) status, is non-toxic and non-flammable, is readily available and cheap (Cui, J. et al.,2021) (Kumar, S. et al.,2023)). As

temperature increases, pH of water drops, which avoids the need for acidic solvent, thus provides high efficacy to SCWE. For extraction of ionic and non-ionic compounds SCWE is regarded as “green technique”. As SCWE is a process of intensive energy and requires extreme temperature and pressure because of this thermal degradation of pectin occurs invariably. Oligomers and sugars are formed when pectin is hydrolysed under certain conditions of SCWE (Benito-Roman, O. et al.,2022) (Kumar, S. et al.,2023).

IV. APPLICABILITY OF PECTIN IN FOOD INDUSTRY

Pectin has a huge range of applications. It can be used as a gelling agent, emulsifier, stabilizer, thickener, and as a fat or sugar replacer in low calorie foods (Himashree, P. et al.,2022)

4.1.GELLING AGENT/ THICKENER

Due to pectin being an essential polysaccharide and its potential of forming gel at low pH, in the presence of Ca^{++} ions or solute; it has applications in food sector, pharmaceuticals and many other more industries (Chandel, V.

et al.,2022). There is no accurate mechanism for formation of gel; Based on the type of pectin, gel formation involves Ca^{++} ions or hydrogen bonding coordinate bonding and hydrophobic interactions (Urbanova, M. et al.,2024). In pectin with low-methoxyl, ionic linkage as a consequence of calcium bridges between two carboxyl functional groups which belongs to two different chains with each other in close contact results in gel formation (Wu, C.L. et al.,2024). Gelation in high-methoxyl pectin results from cross-linking of pectin molecules which involves between the molecules a combination of hydrophobic interactions and hydrogen bonds (Said, N.S. et al.,2023). Factors influencing the formation of gel in pectin are- pH, size of molecule, presence of other solute, density of charge on molecules, number of side chains, arrangement of side chains; Because of the gel formation ability of pectin at food industries it is used in the preparation of jams, frozen foods, jellies and is also used as a replacer of fat or sugar in low calorie foods (Ishwarya S. et al.,2022). Reduction in blood and cholesterol levels and gastrointestinal disorders have come across by the use of

pectins in pharmaceutical industries (Freitas et al.,2021). Pectin is also used in foams, plasticizers, in edible films and etc. In most plants, pectin is found in cell wall, and since the pectin from other sources than apple pomace or orange peel shows poor gelling behaviour, thus the two major sources of pectin used for commercial purpose are apple pomace and orange peels (Chandel, V. et al.,2022).

4.2.EMULSIFIERS

In order to obtain a desirable product in food processing blending of two immiscible liquids is required and to produce such products food additives are used which are known as emulsifiers (Hasenhuettl, G.L. et al.,2019). Properties of attached protein groups and carbohydrate moieties controls the functionalities of pectin (Ropartz, D. et al.,2020). The protein moiety, acetyl groups and feruloyl generally plays a major role in emulsifying activities of pectin (Liu, Z. et al.,2020). In research, it was showed that by replacing pork back fat with 15% insulin and 15% pectin in low-fat meat batter the physiochemical properties and emulsion stability can be maintained (Pollonio,

M.A.R., 2017). Composition of emulsions influences the emulsion-stabilizing and emulsifying properties of pectin (Humerez-Flores, J.N. et al.,2022). Parker, Boulenguer and Kravtchenko (1994) stated that under acidic conditions dairy products protein can be stabilised by the pectin. (Rooker,1927) suggested that in various applications pectin is used as an emulsifying agent such as flavour, vegetable oil emulsions, minerals and mayonnaise.

4.3.PROBIOTICS

Pectin is referred to as a natural prebiotic which has a great potential for encapsulating probiotics for preparation of synbiotics (Sun, R. et al.,2023). Living organisms if administered in adequate amount ($>10^6$ - 10^7 CFU/g of ingested product) promotes health benefits in host are known as probiotics. Health benefits associated with probiotics are reduces inflammation, prevents inflammation, reduction in cholesterol, provides protection against pathogenic bacteria, improves gut barrier integrity, helicobacter pylori inhibition (Das, T.K. et al.,2022). Major groups of probiotics are LAB (Ljungh and

Wadstrom, 2006). Commonly isolated LAB species from dairy products, fruits and vegetables are *Lactobacillus casei*, *L. Plantarum*, *L. Bulgaricus*; the role of these species in human health as probiotic have been shown (Amin, Jarfi, Khosravi, Samarbafzadeh, and Sheikh,2009). In a study, it was investigated that the pectins have ability for improving probiotic species such as *Lactobacillus fermentum*, *L. Paracasei* F-19 survival in stimulated gastric solution. Zeta-potential approach made evaluations of electrostatic interactions between bacteria and pectins. Plate counting and flow cytometry assessed the survival of bacteria (Khodaei, D. et al.,2020).

4.4.EDIBLE PACKAGING

In food industry, food packaging is one of the proliferating segments, generally the increase in functional/active packaging in order to extend the shelf-life and for maintaining the quality of the food product (Kumar, S. et al.,2023). Pectin is an important renewable natural polymer, it has a flexible nature, thus found in biodegradable packaging materials where it acts as a barrier for moisture, oil, aroma, causes reduction in

rate of respiration and food oxidation (Vanitha, T. et al.,2019). Nowadays, a new application has emerged for polymers of pectin i.e., edible film behaves as natural barrier against microbial contamination to protect food and food products and by facilitating exchange of lipids, volatiles and moisture between environment and food (Roy, S. et al.,2023). The edible films incorporated with probiotics are proved to be effective for increasing their survival over dehydration storage and processing (Pop, O.L. et al.,2019), (Romano et al.,2014).

V. CONCLUSION

In food industry, pectin has numerous applications such as it is used as an emulsifier, stabilizer and texturizer, not only in food industry but pectin has various applications in other fields too, due to its gelling and stabilizing property pectin is used in pharmaceutical products. In low calorie foods, pectin is used as a replacement of fat and sugar and because of this the demand for these foods in future is likely to increase; pectin occurs in a number of plant species and there are very limited commercial sources for extracting pectin

thus there is a great need to search for other methods to extract pectin; pectin is also found to have wound healing properties and is effective in cancer therapy. From this paper it is concluded that a lot of citrus fruit waste is generated each day and all over the world which is rich in pectin content. Pectin has huge range of applications in food industry. Sustainable valorization of citrus fruit waste, extracting pectin from it and utilizing it in food industry reduces adverse environmental effects caused by burning and dumping of citrus fruits waste, whereas sustainable valorization will not only protect the environment but will also improve economy.

ACKNOWLEDGEMENT

Authors are thankful to Integral University, Lucknow.

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