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RECENT STRATEGIES FOR CITRUS FRUITS VALORIZATION: A COMPREHENSIVE OVERVIEW OF INNOVATIVE APPROACHES AND APPLICATION

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ABSTRACT

Citrus, the most extensively cultivated and consumed fruit globally, yields significant byproducts in the processing industry approximately 50% juice and 50% waste. Fruit juice concentrate enhances shelf life and enables reconstitution. Processing generates substantial citrus peel, juice sacs, and seeds. Citrus peel is rich in bioactive compounds, phytochemicals, nutrients, and minerals. Peel waste is utilized for essential oil, pectin, and modified citrus pectin production. Modified citrus pectin offers medicinal benefits, potentially inhibiting cancer and heart disease by inactivating galectin-3.
***Keywords* :** Valorization, Citrus, Pectin, Citrus juice and Zero wastage

Introduction

Citrus is one of the most predominantly grown crop in tropical and subtropical parts of the world. It believed that citrus originated in the warm Southern Himalayan slopes in Northeastern India and Northern Myanmar. A member of the Rutaceae family, the Citrus L. genus is most widely cultivated and consumed plants on earth belong torutaceae family. *Citrus reticulata* (mandarin), *Citrus aurantifolia* (lime), *Citrus tangerine* (tangerine), *Citrus limon* (lemon), *Citrus limetta* (sweet lime), *Citrus sinensis* (orange), *Citrus maxima* (pummelo), grapefruit (Citrus × paradisi) and *Citrus junos*(yuzu) are the most common fruits in the citrus family (Benavente Garcia and Castillo, 2008; Economos and Clay, 1999; Gmitter and Hu, 1990; Nangia Makker *et al.*, 2002; Liu*et al.*, 2012; Okwiand Emenike, 2006 and Ziegler and Wolfe, 1975).

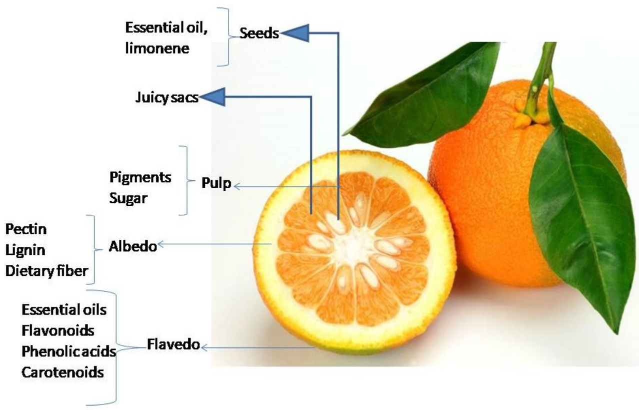


Fig. 1 : Anatomy of citrus fruit

Citrus fruit typically generates at least 45–50% waste and 50–55% juice (Fig. 1). Numerous byproducts, comprising segment wall, flavedo/albedo, seeds, and pith residue, constitute up to the 50%

wastage of citrus mentioned (Gmitter and Hu, 1990). Citrus pulp, peel, pith, pomace, and seeds are highly valued for mitigating pollution and environmental issues.

Citrus byproducts are rich reserves of phytochemicals, bioactive compounds, phenols, dietary fibre, tannins, ascorbic acid, antioxidant activity, as well as vitamins and minerals crucial for human health, development, and nutrition. Citrus fruit bioactive compounds are highly therapeutic and have anti-inflammatory, anticancer, antitumor, and antioxidant activities (Czech *et al*., 2021). These also have a history for possessing anti-inflammatory, anti-platelet aggregation, and antiviral properties. Valorization is a method for using citrus waste in the food sector to produce pharmaceutical, cosmetic, and nutraceutical products (Bakkali *et al*., 2008; Crawshaw, 2004; Lertsatitthanakorn *et al*., 2006; Luangnarumitchai *et. al.,* 2007 and Marin *et al.,* 2007). Valorization is process of giving or enhancing value of product or value addition. Citrus valorization is process of utilization of pulp, peel, pomace and seed for the production of various products. Some of the nutrient compounds in citrus peel, pulp and fruit were reported in Table no 1(Czech *et al*., 2021).

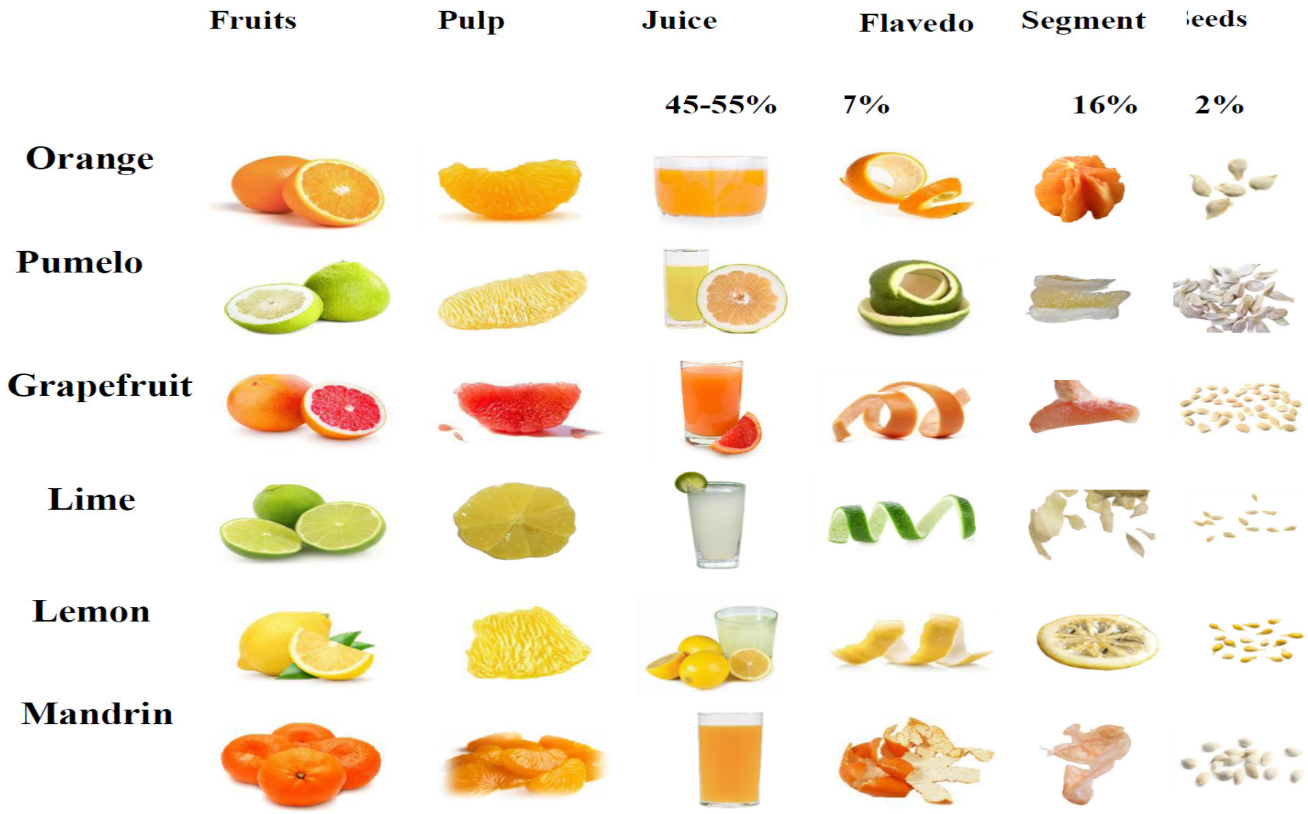


Table 1 : Content of dietary fibre and phytochemicals in citrus pulp, peel and whole fruits

Fruit/Index	Part	Orange	Pumello	Mandarin	Lemon	Lime	Grape fruit
Dietary fibre (g/100 g)	**Pulp**	28.40	33.11	26.32	28.29	28.85	32.96
	Peel	63.77	61.85	62.53	64.64	63.89	61.22
	Whole	48.27	52.62	37.99	37.88	34.55	55.05
Phenolic compounds (mg/100 g)	**Pulp**	164.37	63.07	99.53	78.59	105.10	169.60
	Peel	312.20	645.00	204.30	251.10	282.70	208.70
	Whole	284.68	466.50	158.70	174.40	169.30	238.30
Tannins (µgm/100 g)	**Pulp**	10.13	4.49	9.74	8.590	13.54	14.11
	Peel	22.80	31.50	29.90	28.04	34.81	34.91
	Whole	16.21	16.15	20.38	19.42	22.83	25.75
DPPH (mmol Trolex/g)	**Pulp**	99.14	297.10	448.90	444.80	173.10	170.00
	Peel	73.35	121.4	16.16	12.250	18.99	48.96
	Whole	91.89	219.6	213.40	223.70	96.89	109.70

Medicinal benefits of citrus fruits

- Prevents kidney stone formation: Citrus juice produces calcium citrate, which dramatically reduces the incidence of stone formation by calcium oxalate. Supports the production of hemoglobin and rich in iron content.
- Reduces the high blood pressure.
- Citrus fruits were rich in calcium, iron, and other mineral components, which helps to support healthy body tissues.
- Prevents constipation and promotes proper digestion: Modified citrus pectin produced from pectin and it acts as prebiotic. So it promotes the multiplication of beneficial microorganisms in gut by that it prevents the constipation and promotes proper digestion.
- Cancer prevention: liminoid fight against various cancers diseases like breast cancer, blood cancer and lung cancer
- Cholesterol: alkaloid synephrine present beneath the citrus peel can reduce cholesterol
- Peels are good ant-allergic for skin
- Build good immune system by enhancing the production of white blood cells

Citrus processing plant

A sequence of key steps were typically carried out at citrus processing plant, , including fruit unloading at the receiving site, grading, storage, washing, and manual grading based on fruit diameter (Fig. 2). After that, the fruits transported to the machine which extracts juice. succeding pulpy juice passed to the finisher where it centrifuged to remove remaining solid suspended elements. Further juice pasterized at 85 °C for 25–30 seconds to maintain its microbiologic safety. Natural water content from juice partially evaporated by process of evaporation under vacuum during this process TSS (Total soluble solids) of the product increased and moisture content partially removed. Final concentrate cooled at -18 °C and stored in frozen storage containers. Centrifuged juice passed through pasterizer and vaccum pump inorder to remove the air and stored as Not From Concentrate in aseptic refrigerated condition at - 4 °C tempreture through second line. Second process line pulpy juice passed through defect removal, double finishers and pastrization process and pulp stored in refrigerated condition at - 4 °C. Water and oil emulsion produce during extraction of juice this can be purified by passing through centrifugation, polisher and oil conveyed to winterization process inoreder to remove the wax which was included in oil by keeping oil at 4 °C. Final purified oil stored in frozen drum storage.Discarded fruits, peel, pomace, seeds and rag were utilized for pectin extraction and animal food by pressing and drying process (Ringbloom, 2017 and Kimball,1999).

Citrus fruit juice is a comercial major product produced from citrus fruits. Several bevearges like syrup, squash and RTS were produced from citrus fruits from many years ago. Now a days citrus fruit juice concentrate is emerging product which was produced by various methods.

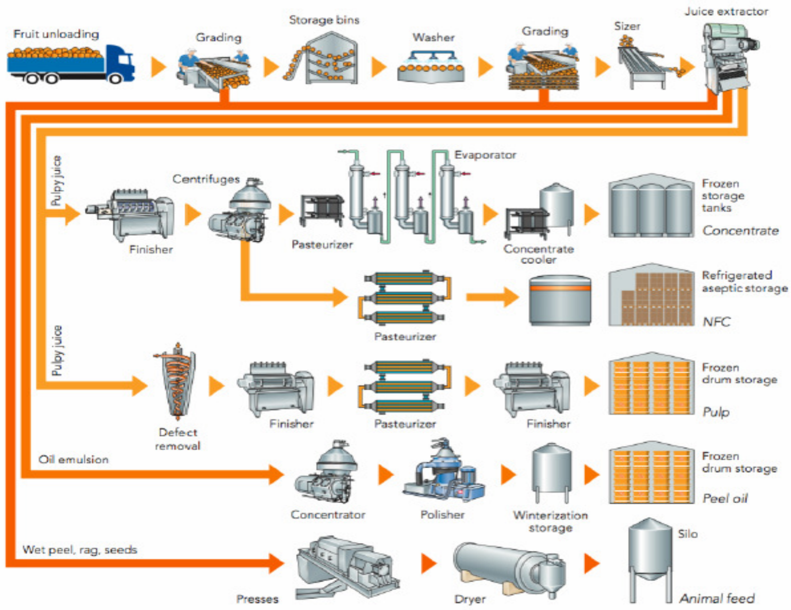


Fig. 2: Flow chart showing typical processing steps in citrus processing plant

Fruit juice concentrates

"Juice concentrate" is the term used for the process of extracting a specific portion of the natural water content present in fruit juices. This procedure involves reducing the juice volume while intensifying its natural sugars, flavors, and other components. The outcome is a thick, syrupy liquid with a higher concentration of the essential elements found in the original fruit juice. Juice concentrate serves as a valuable ingredient in various food and beverage applications and can be reconstituted by adding water to restore the original fruit juice before consumption. Some commercial products utilize concentrated juice as an ingredient without further reconstitution, such as frozen fruit juice concentrates used for making fruit drinks or frozen treats (Petzold *et al.,* 2019 and Medina and Garcia, 2018).

Advantages

- Lowering the water activity (a_w) of the juice product increases its shelf life
- Reduce packaging, storage, and transportation expenses
- Makes handling of the finished juice product more stable or easier

Methods of juice concentration

1. Thermal evaporation under vacuum
2. Cryoconcentration
3. Membrane concentrate

1) Thermal evaporation under vacuum

Principle:

➢ The high vacuum condition maintained in the evaporator, approximately 29 inches of Hg, which facilitates the evaporation of water from juice at significantly lower temperatures (around 58-60 °C) than its boiling point.

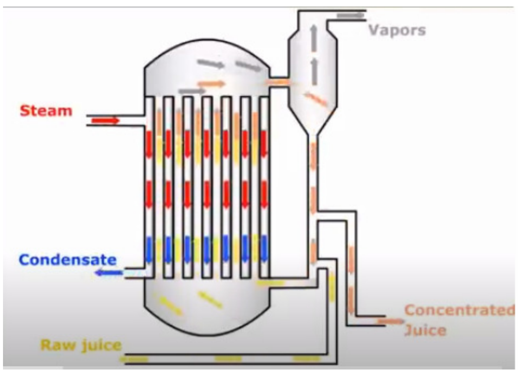


Fig. 3: Thermal evaporation under vacuum

Process: Here the raw fruit juice feed from the bottom and steam used as heating medium which enters from the top of the column. As soon as steam moves towards down of the column the moisture content in the juice evaporate and juice become concentrate. The concentrate juice collected from middle vent (Fig. 3). Vapors which produced due to the heating of water were used as heating medium for further columns. Pressure and tempreture in the colums should maintain based on the type of raw juice used (Deshpande *et al.,*1982; OrellanaPalma*et. al.,*2019 and Petzold *et al.*, 2019).

Advantages

- Economical method of fruit juice concentration
- Comercial method used for production of fruit juice concentrate

Disadvantages

- Loss of more volatile flavoring compounds as well as nutrients due to heat
- Fining agents, enzymes and centrifugation are needed for the clarity of juice
- The application of high temperatures facilitates the oxidation of compounds
  Ex: Ascorbic acid
- High temperature can lead to the chemical alteration of aroma and flavor compounds (Aider and Ounis, 2012; Deshpande *et al*., 1982; Orellana Palma *et al.,* 2019 and Petzold *et al*., 2019).

2. Cryoconcentration

- The process involves removing pure water from the fruit juice by forming ice crystals at sub-zero temperatures.
- The product is cooled below its freezing point, causing water to crystallize, and these crystals are subsequently separated from the concentrate through a centrifugation process (Deshpande *et al*.,1982 and Orellana Palma *et al.,*2019).

Preservation principles :

- Reduction in water activity
- High retention of volatile aroma compounds
- Nutritional qualities and sensory characteristics are maintained

Advantages

- Low temperature
- High retention of volatile aroma

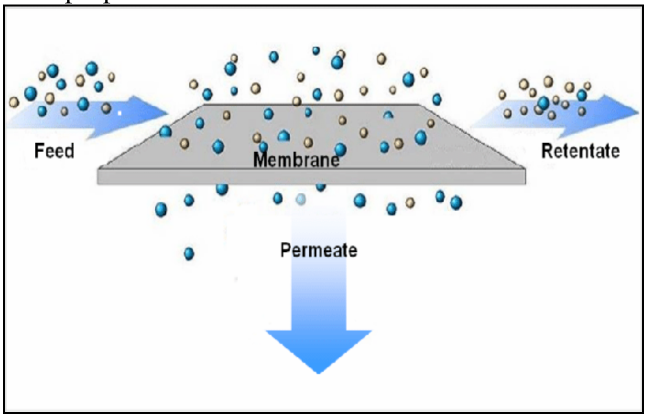
Disadvantages

- The concentration achieved up to (40-45 °brix) lower than the evaporation method
- Separation of small ice crystals is difficult
- High refrigeration cost
- Low production rate(Jiao*etal.,* 2004)

Membrane concentration

- Membrane is placed between two phases i.e., feed and permeate

- Membranes are materials that have voids in them allowing some molecules to pass more conveniently and retain some other molecules
- Retained the most of the sensory and nutritional properties of the food



Advantages

- There is no application of heat to the product
- Negligible losses of volatiles/ nutritional and eating quality
- This technique uses energy more effectively by avoiding the phase transformation.
- Require easy installation with less expensive labour and running costs

Disadvantages

- The product flow rate varies due to fluctuations in the concentration of the feed juice.
- The concentration achieved atmost 30 °brix
- Fouling membranes (deposition of polymers) reduce efficiency(Khan *et al.,* 2010; Rikraj *et al.*, 2019; Singh *et al.,*2020 and Singh *et al.*, 2021).

Pectin

➢ Pectin is a structural acidic hetero-polysaccharide found in the primary and middle lamella of plant cell walls. It is a heterogeneous polysaccharide composed mainly of α (1-4) -D galacturonic acid units. The unique structure and properties of pectin make it an essential component in various industries, including food, pharmaceuticals, and cosmetics, due to its gelling, thickening, and stabilizing properties (Eliaz *et al.,* 2006; and Nangia-Makker*et al.*, 2002).

Classification based on degree of methoxylation

1. **High methoxylpectin:** Pectin with more than 50% of methyl-esterified carboxylic groups is known as high methoxy pectin. This type of pectin can form gels with the help of a cosolute, such as sugar, in an acidic environment with a pH lower than 3.5. When the pH is reduced, the carboxylic groups of pectin become protonated, and the pectin molecules can interact and bind together, resulting in the formation of a gel structure. This gelation process is essential in the production of jams, jellies, and other food products that require a stable and consistent texture. The degree of esterification of pectin plays a significant role in its gelling properties, and high methoxy pectin is commonly used for gelling applications in acidic conditions.
2. **Low methoxyl pectin:** Pectin with less than 50% of methyl-esterified carboxylic groups is known as low methoxy pectin. This type of pectin can form gels in the presence of calcium ions. When calcium ions are present in the solution, they bind to the carboxyl groups of the pectin molecules, cross-linking them and causing the pectin to form a gel network. This gelation process occurs in the presence of divalent cations like calcium, and it is commonly used in the production of fruit preserves, dairy products, and other food applications that require gelling without the need for acidic conditions. The ability of low methoxy pectin to form gels with calcium ions makes it a valuable ingredient in various food and pharmaceutical products.

Modified citrus pectin (MCP)

MCP is more digestible form of pectin. It is obtained from commercial pectin. Commercial citrus pectin has molecular weight 50-300 kDa (kilo Daltons) with esterification more than 70%. MCP has molecular weight less than 13 kDa with esterification less than 10%. However optimal biological activity of pectin is more with molecular weight less than 10 kDa and less esterification which allow easier absorption by the small intestinal epithelium. Therefore, MCP has more health benefits than unmodified pectin. Modified citrus pectin also known as depolymerized pectin, fractioned pectin (Alvarez *et al.,* 2000; Eliaz *et al.*, 2006; Fang *et al.*, 2018; Gunning *et al.,* 2011; Gunning *et al.,* 2013; Maxwell *et al.,* 2012 and Patil *et al.*, 2006).

Production of modified citrus pectin

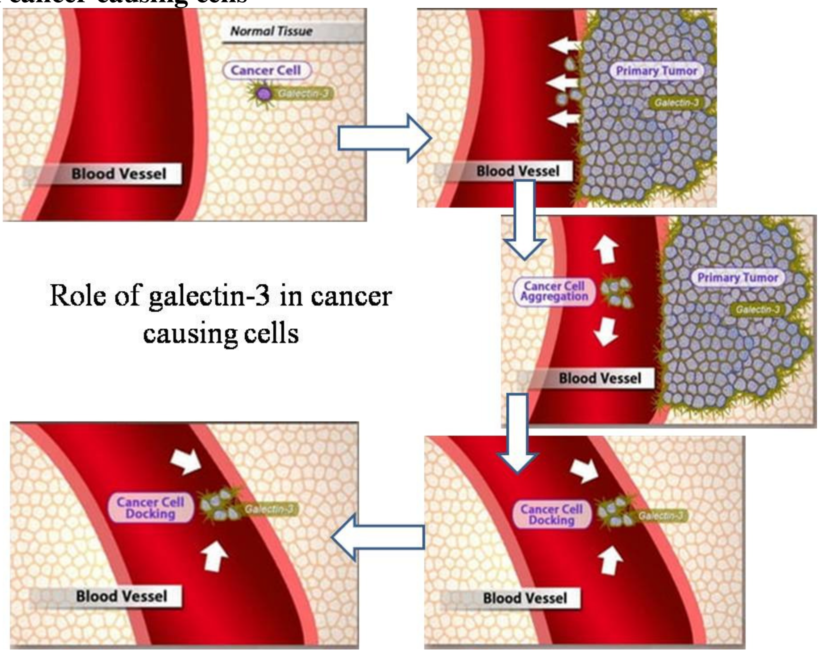
Citrus pectin was dissolved in a 1.5% solution of distilled water. A 3 N solution of NaOH was used to modify and keep the solution pH at 10.0. The combination was then incubated at a temperature of 50 to 60 °C for an hour. 3 N solution of HCl was used to reduce the pH to 3.0 once it had reached room temperature. The samples were left overnight, and the next day, they were precipitated with 95% ethanol. The precipitate was incubated for two hours at 20 °C. The material was then filtered, cleaned with acetone, and dried in a vacuum at 25 °C to complete the procedure (Eliaz *et al.,*2006).

Health benefits of MCP

- Supports gut health
- Prevents constipation
- Improves immune function
- MCP inactivates galactin-3 a marker for heart failure and cancer

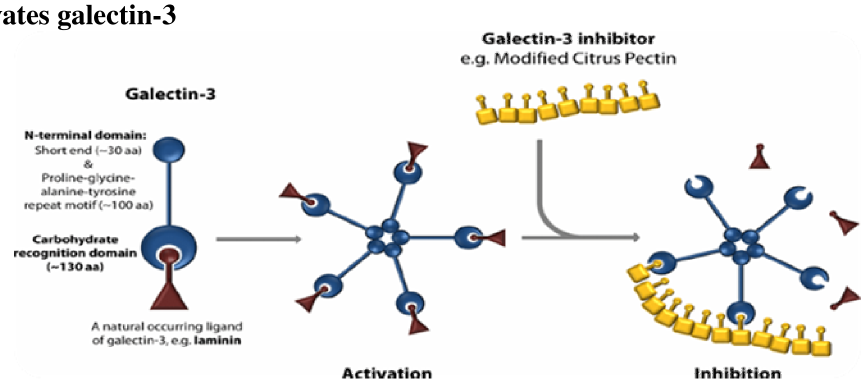
Inhibit tumor growth by increasing autophagy and apoptosis(Alvarez *et al.,* 2000; Eliaz *et al*., 2006; Fang *et al*., 2018; Gunning *et al.,* 2011; Gunning *et al.,* 2013; Maxwell *et al.,* 2012 and Patil *et al*., 2006).

➢Role of glaectin-3 in cancer causing cells



- If any oncogenic cells present in body in that situation galectin binds to that.
- It starts multiplication and primary tumor grows and spreads to the other tissues
- Some of the cells may aggregate inside the blood tissues and blocks the blood vessels
- Metastasis taken place
- Cancer cell and galectin docking to the laminin which is present in basal part of blood vessels and it enhance the multiplication of cancer cells (Fang *et al.,* 2018).

How MCP inactivates galectin-3



When galectin-3 binds to the naturally occurring ligand laminin it activates galectin-3 but modified citrus pectin has more affinity and very specific than laminin consistently demonstrated by using a combination of fluorescence microscopy, flow cytometry, and force spectroscopy (Fang *et al.,* 2018 and Fox, 2009). Therefore, it replaces the laminin and deactivates the galectin-3 and it restricts the spreading of cancer cells.

Extraction of high-value compounds from citrus peel waste

Extraction is regarded as the most important stage in the recovery of these high-value chemicals. In recent years, extensive research has been conducted on 'green extraction techniques' such as microwave-assisted, ultrasound-assisted, supercritical fluid, and enzyme-assisted extraction for the effective valorization of citrus by-products particularly peel, which are known to be eco-friendly, sustainable, safe, energy- and time-efficient. Researchers are increasingly interested in ionic liquids and deep eutectic solvents for extraction operations due to their biodegradability, low volatility at ambient temperature, high miscibility in water,

and higher solvent ability. However, the sort of extraction procedure used may vary based on the component to be extracted and the type of citrus fruit from which it must be removed. To maximize the yield of the necessary chemical, a suitable solvent must be chosen, as well as operational parameters such as time, temperature, pH, solute-to-solvent ratio, and others. Numerous studies have been conducted regarding the technical and economic aspects of extracting different high-value compounds from citrus peel waste using green techniques (Table 2), which have been listed below:

Table 2: Utilizing citrus peel waste for production of high-value compounds using green techniques

Substrate	Method of valorization	Bioproduct obtained from citrus waste	References
Citrus reticulata peel waste	Fermentation via *Trichoderma sp, E. coli*	Mucic acid	Jeong *et al*., 2021
Citrus sinensis peel waste	Enzymatic hydrolysis and fermentation	Bioethanol	John *et al.,* 2017
Citrus maxima peels waste	Pulse Electric Field Processing	Naringin	Kaur *et al*., 2021
Citrus medica peel waste hydrolysates	Fermentation via *Saccharomyces cerevisiae*	Bioethanol	Sharma *et al.,*2017
Persian lemon waste	Ultrasound Assisted Processing (UAP)	Catechin, hesperidin, and naringenin	Sharma *et al.,*2019
Citrus reticulate peel waste	Ultrasound Assisted Processing (UAP)	Lutein	Saini *et al.*, 2021
Citrus maxima peel waste	Solvent-free microwave processing (SFMP)	Essential oil	Celia *et al.,*2013
Citrus sinensis peel waste	Ensiling	Lactic acid, acetic acid, and ethanol	Ruiz *et al.,* 2016
C. aurantifolia peel waste	Enzyme-assisted extraction (Cellulase, Xylanase)	pectin	Naghshineh *et al.*, 2013
C. sinensis peel waste	Steam explosion technique	Dietary fibre	Wang *et al.,* 2015
C. limon peel waste	Solvent free microwave extraction	Limonene	Golmakani *et al.,* 2016

Essential oil

Plant-derived essential oils are highly prized in today's society because of their importance as key ingredients in a variety of goods. The food, aromatics, medical, and agricultural industries all use these oils for different purposes (Aggarwal *et al*., 2022).

The volatile substances that constitute the essential oils are generally found in oil sacs within citrus peels and cuticles. Most of the terpenic hydrocarbons in these oils, such as monoterpenes and sesquiterpenes, as well as their oxygenated derivatives such alcohols, ketones, aldehydes, and esters, account the overall oil's predominant chemical composition. These elements help the oils powerful therapeutic capabilities, which include anaesthetic, analgesic, sedative, anti-inflammatory, and antibacterial actions (Xu *et al*., 2011 and Ghafoor *et al.,* 2012).

The most popular extraction techniques used to obtain essential oils from plant sources are cold expression and distillation (including water, steam/ water, and steam distillation). Although these traditional methods are frequently utilised, they have a few disadvantages, most notably a high time and energy cost. Actually, the energy used by these conventional extraction methods accounts for about 70% of the overall process energy. The emphasis on creating ecologically friendly methods has grown more alluring in recent years. The adoption of the latest innovations has shown encouraging results in terms of cost savings, reduced energy use, and improved entirety of the product and process quality. Researchers are currently keen on optimizing innovative processes, including ultrasound-assisted extraction, supercritical fluid extraction, and microwave-assisted extraction (Table 3). These advanced techniques hold great potential for enhancing the efficiency of extracting essential oils and other valuable compounds from natural materials (Table 4) (Fox, 2009; Fracasso *et al*., 2018; Glinsky and Raz, 2009 and Zhang *et al*., 2016).

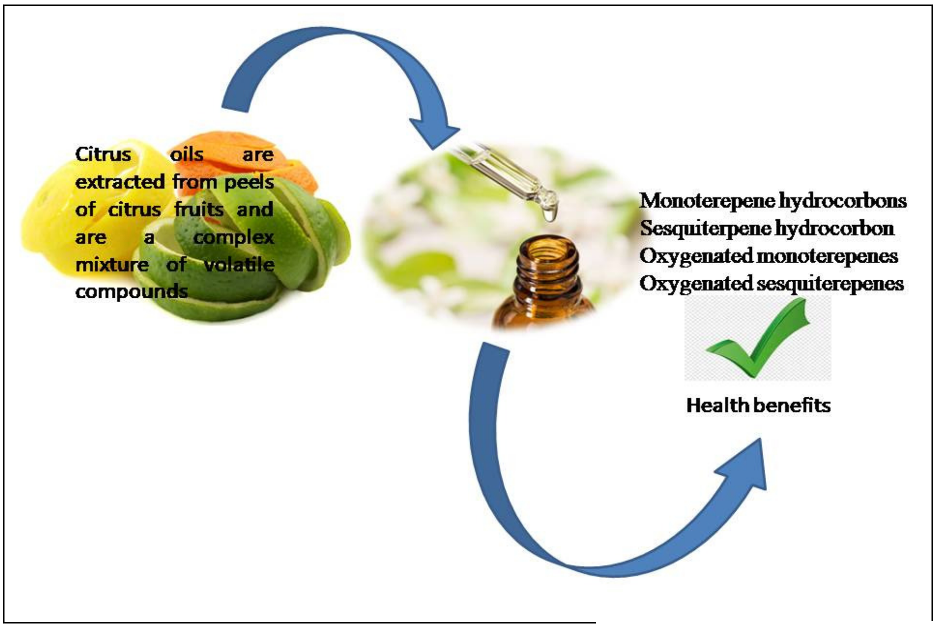


Table 3 : Methods for extraction of essential oil (EO) (Fox, 2009; Fracasso *et al*., 2018; Glinsky and Raz, 2009 and Zhang *et al*., 2016).

Extraction method	Advantages	Disadvantages
Conventional methods		
Hydrodistillation	Simple approach of extraction Easy essential oil isolation Low equipment cost	Less recovery Require more energy
Steam distillation	Reduce extraction time Reduce chemical alterations Reduce loss of polar molecules	Several hours of heating; Degradation of thermolabile compounds
Solvent extraction	Overcomes modifications and chemical residues (cold extraction)	Extraction time is long Large volume of solvent required More energy required
Innovative techniques		
Supercritical fluid extraction (SFE)	Produces EO of higher grade with better biological and functional activity	High process cost Necessity of CO_2 purity
Ultrasound-assisted Extraction (UAE) of essential oil	Simple and affordable Increases the pace of EO release Increased extraction rate and efficiency Lower temperature during extraction	Lack of uniform distribution of ultra-waves
Microwave assisted extraction (MAE)	Most effective way of extracting EOs Greater reproducibility in less time a minimal energy footprint Less use of solvents	Filtration and centrifugation are necessary

Table 4: Composition and potential food applications of citrus fruits

Citrus Source	Part	Composition	Food application	References
Citron	Peel, pulp	Antioxidants, vitamin C, phenols, flavanols	Carbonated drinks, alcoholic beverages, jams, syrup	Chhikara *et al.,* 2018
Lemon	Peel, pulp	Vitamin C, pectin, phenols, carotenoids	Edible coatings, use as film matrixes	Jiang *et al.,* 2022
Sweet orange	Peel, pulp	Narirutin, naringin, quinic acid, sakuranetin	Enhance vegetable oil oxidative stability, candied orange peel, salad dressings, desserts	Shehata *et al*., 2021
Grapefruit	Peel, pulp	Naringin, naringenin, flavonoids, phenol	Jam making, sauces, dessert recipes	Khan *et al.,* 2021

Conclusion

Citrus processing industries produce huge byproducts which are causing environmental issues. Citrus byproducts rich in bioactive compounds, phyto-chemicals, vitamins, minerals, essential oil and dietary fibers (modified citrus pectin). Therefore in order to avoid the environmental burden a way to utilize the citrus waste in to potential products like citrus juice concentrate, essential oil, pectin and modified citrus pectin, cosmetics and pharmaceutical industries. Which were having the great health benefits to control the cancer, heart diseases and it also act as prebiotics. Valorization strategies were enhance the farmer income and depletes the cost incurring on pollution management by industries by making way for zero wastage and also meet the need of hunger for growing population.

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