

**EXTRACTION AND CHARACTERIZATION OF ORANGE PEEL PECTIN AS A
NATURAL TABLET BINDER**

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ABSTRACT

Orange peel is the substantial pectin source which is considered as the primary component of plant tissues. Pectin is utilized as a gelling, emulsifying and thickening agent in numerous food products. At present, industrial pectin extraction is done by utilizing hot water at low pH for a long duration of time which is a time-consuming process. However, it is verified that Ultrasonication lessens the extraction time of pectin and maximize yield as compared to the traditional Method in use. Identifying the capability of Ultrasound assistance in extraction, this research emphasizes on pectin Extraction using orange peels as raw material. Orange peel converted into the powdered form was utilized for extraction of pectin. Pectin was extracted with different combinations of Ultrasound Power (60, 80 and 100%), citric acid. Concentrations (pH: 1, 1.5 and 2) and extraction time (10, 20 and 30 minutes). As a result, the highest pectin yield of 20.92% was attained at the Ultrasound power of 100 %, Ultrasound Time of 30 Minutes and pH of 1.5 of citric acid Solution. The research confirms that utilizing Ultrasound and citric acid for pH, enhanced yield and standard of synthesized pectin; and also saves sufficient amount of time and energy.

KEYWORDS: orange peel, Pectin, Ultrasound assisted extraction, alcohol precipitation method.**INTRODUCTION**

Pectin has been recognized for at least 127 years back and was originally identified in 1790 in apples by the French chemist Nicholas Vauquelin. It was not until 1824 that further work on pectin was undertaken by Braconid who named the acid, gelling substance pectin acid after the Greek word for gelling or congealing. Later, in the 1920s and 1930s, factories were built that commercially extracted pectin from dried apple pomace. In 1924 Smolensk identified the gelling substance as a polymer of galacturonic acid and later on in the 1937 Schneider and Bock established the basic formula of pectin.^[1,3] Now a day pectin is recognized as a complex polymer that is present in many plants as a component of the middle amella. Pectin is used extensively in the food industry as a gelling agent and is the key gelling agent in jam manufacture which is still one of the biggest markets for pectin. In the past Pectin sold as a liquid extract, but is now most often used as dried powder, which is easier than a liquid to store and handle. Pectin is a naturally occurring biopolymer that is finding increasing applications in the pharmaceutical and biotechnology industry.^[4,5] It is also used in the food and beverage industry as a thickening agent, a gelling agent and a colloidal stabilizer. Pectin occurs commonly in most of the plant tissues, the number of sources that may be used for the commercial manufacture of pectin is limited. The various sources of pectin include citrus peels, sugar beets,

residues of mango, guava, coffee, dried apple pomace, sunflower heads, papaya, and cocoa processing. Currently half of the commercial pectin's used in the food industry are extracted from citrus peels. New application opportunities have emerged and pectin is no longer just a gelling agent but also used in wide applications.^[6] Citrus fruits are at the top not only in total production, but also in economic value. One of them; oranges, specifically, the oranges are a very commonly growing tree fruit in the world. Oranges are widely cultivated in tropical and subtropical climates, which is peeled and eaten whole, or processed to extract orange juice and also for the fragrance. These essential oils (fragrance) are a mixture of volatile compounds as trepan and oxygenated derivatives such as aldehydes (citral), alcohols and esters.^[7] They have a great commercial importance and various applications in many foods industry product because of their aroma and functional properties (antifungal, antimicrobial, etc.) which make them excellent additives. part of something, its end or result. The present work explores the possibility of separation of Pectin is a natural, biocompatible, biodegradable and renewable polysaccharide characterized as an emulsifier gelling agent, glazing agent, stabilizer, and/or thickener in commercial applications; all of which are in fact subsets of the term rheology modifier.^[8,9] Pectin gels are formed when the molecule chains are cross-linked, forming a

three-dimensional network where water and co-solutes are retained.^[10] Consumption of pectin has been shown to reduce blood cholesterol levels. In the large intestine and colon, microorganisms degrade pectin and liberate short-chain fatty acids that have positive influence on health.^[11] The Joint FAO/WHO Expert Committee on Food Additives (JECFA) has recommended pectin as a safe additive with no limit on acceptable daily intake.^[12] Pectin is quite stable under the acidic condition of the stomach, although a slight desferification can occur. Without the fermentation process, pectin would pass almost unchanged through the digestive system.^[13] Increasing consumer awareness of a healthy lifestyle and the emerging trend to produce functional food has made pectin popular. It has been reported that pectin has numerous positive influences on health including improving colonic health, lowering of cholesterol and serum glucose levels, reducing cancer propensity, and stimulating the immune response.^[14] The degree of methyl-esterification (also known as degree of methylation) (DM) of Gal A units is used to classify pectin. DM is a percentage which expresses the molar ratio of methyl esters present to Gal A units (includes both free Gal A and substituted Gal A).^[15] It is the major parameter affecting gelling, influencing surface tension and emulsion formation. The DM percentage above 50% is classified as high methyl ester (HM) pectin while those less than 50% is known as low methyl ester (LM) pectin.^[16] The lower the DM, the faster pectin is hydrolysed, probably due to a lower amount of methyl esterified target groups. This in turn influences gel strength, leading to the formation of a weaker gel.^[17] However, at controlled or reduced temperatures, there is a higher possibility that LMP will be obtained without extensive main-chain breakdown. Enzyme de-esterification has becoming increasingly popular for obtaining LMP in an efficient and environmentally sustainable manner.^[18,19] Rapid-setting High Methoxyl Pectin (HMP) is usually obtained after a short extraction time at temperatures close to boiling.^[20] This is due to short extraction times with high temperature reduces de-esterification. Conversely, long extraction times with low temperature favour slow setting HMP or even LMP.

Therefore, it is important to select suitable extraction conditions to obtain pectin with the desired properties.^[21]

Various Methods of Pectin Extraction

- Ultrasonic assisted extraction method
- Alcohol precipitation method
- Acid extraction of pectin
- Enzymatic extraction
- Cellulase enzyme
- Polygalacturonate enzyme
- Microwave assisted extraction

Applications of Pectin

1. The major application of pectin is as a textural ingredient in food systems, while bit is also used in cosmetics and personal care products.
2. In the pharmaceutical industry, pectin is used in the formulation of controlled-release matrix tablets, for example, as a carrier material in colon-targeted drug delivery.
3. Due to the multifunctional properties of pectin's, they have valuable applications in food industries as thickeners, stabilizers and emulsifiers.
4. Pectin and pectin derivatives are used in diarrheal disorder and constipation. Pectin lowers the blood cholesterol level by increasing the fecal cholesterol, fecal fat, sterols and bile acid.
5. Pectin reduces rate of digestion by immobilizing food components in the intestine.
6. It is used in the hair tonics, body lotions and shampoos.
7. It is also used in the deodorants and tooth pastes.
8. Pectin is also used in wound healing preparations and specialty medical adhesives, such as colostomy device.^[26]

MATERIALS AND EQUIPMENTS

The chemicals used were of analytical reagent grade. Material and equipment's used for the extraction and characterization of pectin from orange peel are shown in Table No. 1 and 2.

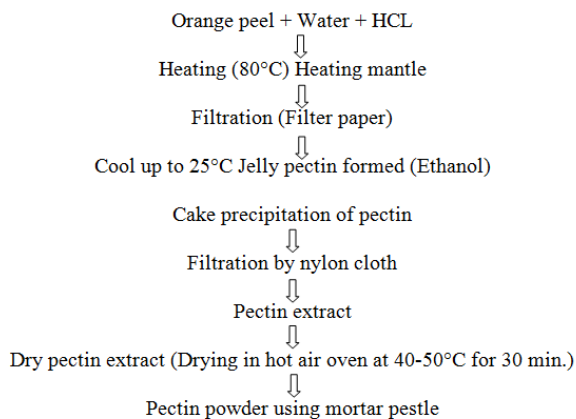
Table 1: List of Materials and respective suppliers.

Sr. No.	Chemicals	Suppliers
1	Conc. Sulfuric Acid	Molychem, Mumbai
2	Ethanol	Loba, Mumbai
3	Hydrochloric acid	Molychem, Mumbai
4	Ibuprofen	Valona pharma private Ltd. India
5	Talc	Prominence health care private Ltd.
6	Magnesium stearate	Sankalp organics pvt. Ltd, Mumbai

Table 2: List of Equipment's.

Sr. No.	Equipment	Make
1	Bulk density apparatus	Indosathi, Ambala
2	Digital weighing balance	Wesnar, Mumbai
3	Digital pH meter	Labrotonics, Mumbai
4	Hot air oven	Indosathi, Ambala
5	Stalagmometer	Indosathi, Ambala

Extraction of Pectin: Mature Orange fruits were collected and orange fruits were cut into four parts and the peel was removed (a soft white substance inside the skin of citrus fruits), then the peels were further cut into smaller pieces for easy drying and washed with large quantity of water to remove the glycosides the bitter taste of the peels and then weighed with a digital weighing balance and air dried.



Evaluation Test of Pectin

Physicochemical characterization of pectin

Organoleptic evaluation

Sample of pectin was visualized for organoleptic properties such as Color, Odor, Taste, Texture etc.

Solubility of pectin

The solubility of the pectin was identified by taking one part of dry pectin powder and it was shaken with different solvents and the solubility was determined.

pH of pectin

The pH of 1% w/v solution of pectin was measured using digital pH meter.

Swelling index of pectin

Transfer 1gm of Pectin to a 25 ml stoppered measuring cylinder. Fill the cylinder up to 20 ml mark with water. Agitate gently occasionally during 24 hour and allowed to stand. Measure the volume occupied by the swollen and final result was calculated using formula.

$$\text{Swelling weight} = \frac{\text{Final weight} - \text{Initial weight}}{\text{Initial weight}} \times 100$$

Surface tension of pectin

Surface tension of 1% w/v solution of pectin was measured using a stalagmometer.

Loss on drying

The test was carried out according to the procedure described in Indian Pharmacopoeia. One gram of hydrogel powder was weighed accurately in a tarred glass Stoppard bottle and was dried in a hot air oven at

105°C and the weight was checked at intervals of 1 h, until a constant weight was obtained. The percentage of weight loss by the powder was calculated.

Powder flow property

Flow property of pectin powder was calculated by measuring angle of repose. Angle of repose was calculated thrice by using formula,
 $\theta = \tan^{-1} h/r$

Powder Compressibility

Compressibility of powder is determined by Carr's Index. Powder. 5g of powder was transferred into a measuring cylinder and using the bulk density apparatus calculations were done.

Particle size analysis

The particle size of powdered pectin was determined using Optical Microscopy.

Ash value^[46]

Ash value was calculated by weighing 2gm of pectin powder in a tared silica crucible. It was then incinerated in a muffle furnace up to 450 °C till the powder completely changes to ash. The crucible was then kept in desiccator after complete incineration. Weight of ash was noted and total ash was calculated in terms of percentage.

Preparation of Granules

Weighted quantity of ibuprofen, orange peel pectin, lactose, talc and magnesium stearate were added according to formula of four batches in dividually. According to formula all the ingredients were mixed properly with the help of mortar and pestle and using distilled water for forming a wet mass. The wet mass was passed through sieve 20 to prepare granules. Granules of all batches were dried at 450 C for 5 hours and stored in air dried packets for further evaluation study.

Properties of Granules

Granules were evaluated for all pre-compression parameters like angle of repose, bulk density, tapped density, bulkiness, Hausner's ratio and compressibility index etc.

RESULT AND DISCUSSION**Phytochemical Screening of Orange Peel Powder****Table 3: Phytochemical Screening of Orange Peel Powder.**

Test	Observation	Inference
Molisch's Test Sub. + Water + 2 drops of Molisch's reagent. shake well and add 2 ml Conc.H ₂ SO ₄ along the side of the test tube.	Formation of a reddish violet at the junction of two liquids. which on shaking produces deep violet solution	carbohydrates present
Confirmatory Test Osazone Test	Needle Shaped Crystals	Carbohydrates confirmed

Physicochemical Characterization of Pectin**Organoleptic Evaluation**

The observed organoleptic properties of pectin are shown in Table No. 4.

Table 4: Organoleptic properties of pectin powder.

Sr. No.	Organoleptic properties	Observations
1	Colour	Pale yellow white
2	Odour	Fresh fruity orange peel
3	Taste	Sweet
4	State	Solid
5	Texture	Rough and irregular

Solubility Profile of Pectin

Table No. 5 displays the pectin's solubility profile. According to a solubility investigation, pectin is soluble in hot water, expands to form gel in cold water, and is insoluble in the majority of organic solvents.

Table 5: Solubility Profile of Pectin.

Sr. No.	Solvent	Solubility
1	Cold Water	Swells To Form Gel
2	Hot Water	Soluble
3	Methanol	Insoluble
4	Ethanol	Insoluble
5	Acetone	Insoluble

pH

The pH of pectin powder was determined and it is observed as 2.2 prepared by Ultrasonic assisted method and 3.1 prepared by Alcohol precipitation method.

Swelling Index

The Swelling Index of pectin powder was found to be 150 % prepared by Ultrasonic assisted method and 160 % prepared by Alcohol precipitation method.

Surface Tension

The Surface Tension of pectin powder prepared by Ultrasonic assisted method was found to be 75 Dyne/cm and 70 Dyne/cm by Alcohol precipitation method.

Loss on Drying

The Loss of Drying of pectin powder was found to be 5% and 4% by Ultrasonic assisted method and Alcohol precipitation method respectively.

Particles Size Analysis

The particle size of powdered pectin was determined using optical microscopy. The value observed was 1.76 µm.

Ash Value

The Ash Value of pectin powder prepared by Ultrasonic assisted method and Alcohol precipitation method was found to be 3.7% and 3.4% respectively.

Bulk Density

The Bulk Density of pectin powder prepared by Ultrasonic assisted method and Alcohol precipitation method was found to be 15 g/ml and value is 10 g/ml respectively.

Tapped Density

The Tapped Density of pectin powder prepared by Ultrasonic assisted method and Alcohol precipitation method was found to be 11 g/ml and 7 g/ml respectively.

Carr's Index

The Carr's Index of pectin powder prepared by Ultrasonic assisted method and Alcohol precipitation method was found to be given 12% and 17% respectively.

Angle of Repose

The Angle of Repose of pectin powder prepared by Ultrasonic assisted method and Alcohol precipitation method was found to 40° and 45° respectively.

SUMMARY AND CONCLUSION

Orange peel is a significant source of pectin, which is regarded as the main building block of plant tissues. Pectin is used in many culinary items as a gelling,

emulsifying, and thickening ingredient. Currently, industrial pectin extraction involves using hot water with a low pH for an extended period of time, which is a labor-intensive method. However, it is confirmed that when compared to the currently used traditional method, ultrasonication shortens the time required for pectin extraction and increases yield. This study focuses on pectin extraction utilizing orange peels as the raw material to determine the extraction capabilities of ultrasound. Pectin was extracted from orange peel that had been ground into a powder. It is concluded from the result that the extracted pectin from orange peels showed presence of carbohydrates, hexose sugar. All the organoleptic properties evaluated were found to be acceptable. The pH was found to be acidic. Swelling Index reveals that the pectin swells well in water. Total ash value was in the limits. The values of angle of repose and Carr's Index of powdered pectin showed excellent flow property. The ultrasound assisted extraction and alcohol precipitation extraction applied for the extraction of pectin. The ultrasound assisted extraction were found to be strongly influenced the yield. However, ultrasound power was highly significant compared to alcohol precipitation extraction.

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