

Extraction of Pectin from Citrus Peels - A Review

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Abstract: Pectin is complex polysaccharides contains 1, 4-linked α and β galactosyluronic acid residues extracted using alcohol precipitation method from citrus peels. The color of pectin from orange peel was pale yellow. Pectin is soluble in hot and cold alkaline water. The pectin is polysaccharide use as a stabilizer in foods. The pectin was extracted from various citrus fruit peels and its optimization by varying pH using hydrochloric acid and citric acid. Pectin can be used to improve the mouth feel and the pulp stability in juice-based drinks and as a stabilizer in acidic protein beverages. Extraction of pectin from citrus fruit peels like Orange peels, sweet orange and lemon gives higher yield of pectin. % Yield and rate of extraction for pectin from orange and sweet lemon (Mosambi) peels depends on parameters such as pH, temperature, solvent, time of extraction. Extraction carried out at pH 1.5 at 60 min with 60, 70 & 80 °C temperature. The higher yield of pectin at pH value 1.5 and contact time 60 min. The extraction of pectin such as Citric Acid, Hydrochloric Acid, Sulphuric Acid, Nitric Acid and oxalic acid ($C_2H_2O_4$). The time of extraction increased to an extreme there is less effect on yield of pectin reported and also decreased from maximum level due to thermal degradation of the extracted pectin.

Keywords: Extarcion Pectin, Orange and Sweet Lemon Peels, Acetic and Nitric Acid, Extraction-Precipitation and Drying.

I. INTRODUCTION

Pectin is a family of complex polysaccharides that contains 1, 4-linked α and β galactosyluronic acid residues was extracted using alcohol precipitation method from peels of oranges. The results showed that the color of pectin from orange peel was pale yellow. Pectin is soluble in hot and cold alkaline water. Pectin can be used to improve the mouth feel and the pulp stability in juice-based drinks and as a stabilizer in acidic protein beverages. Pectin also reduces syneresis in jams and marmalades and increases the gel strength of low-calorie jams. Pectin is used in confectionery jellies to give a good gel structure and a clean bite. Pectin is a natural, biocompatible, biodegradable and renewable polysaccharide use as an emulsifier, gelling agent, glazing agent, stabilizer and thickener. Orange peels are a major commercial source of pectin. India is one of the large production countries of orange is about 2.64 millions/year. In Maharashtra Nagpur region is well known in central Asia to produce and market hub for orange. It is also known as the California of India producing excellent quality oranges in large number. This paper reviews the extraction process and its utilization in industry. Orange oil and pectin from orange peel which is the waste of orange juice processing industry. [1]

II. CLASSIFICATION OF PECTIN

Pectin can be classified according to their degree of esterification (DE). The degree of methylation (DM) is defined as the percentage of carbonyl groups esterified with methanol. If > 50% of the carboxyl groups are methylated the pectin are called high methoxy pectin (HMP) and < 50% of the carboxyl groups are called low methoxy pectin (LMP). LM pectin can also be further processed to produce amidated (AM) pectin by de-esterification of the LM pectin in an ammonia medium.

2.1 High Methoxyl Pectin (HMP)

HMP need the presence of a high concentration of solids (> 55%) before they can gel, with sucrose used mainly for commercial pectin. HM pectin to sweetened products.

2.2 Low Methoxyl Pectin (LMP)

LMP can gel in the existence of divalent cations usually calcium. Gelation is due to the arrangement of intermolecular junction zones between homogalacturonic smooth regions of different chains. The arrangement of junction zone is normally attributed to the so called 'egg box' binding process. LMP with a blockwise distribution of free carboxyl groups are very sensitive to low calcium levels. They do not require a low pH, but gel at a pH range of 2-6.

III. LITERATURE REVIEW

The sweet orange peels are good source of orange oil and pectin and does have the potential to become important raw material for food processing industries. The maximum pectin yield is 52.90%. The successful extraction of essential oil and pectin, providing potential benefits for industrial extraction of pectin from an economic and environmental point of view. pH is considered as one of the more crucial parameters affecting the amount and properties of extracted pectin. Pectin yield decreased with increasing pH, highest being 52.90% at pH 1 and 60-mesh size. Soxhlet Extraction method has been adopted ensuring no alteration in the properties of the components and greater yield with appreciable solvent recovery. Strong acids are corrosive and may be a potential threat to health. The liquid waste generated from the industrial processes lead to burden the environment and a high cost might incur for treating the strong acidic waste. [1]

Orange peel converted into the powdered form 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. 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. Ultrasound and citric acid for yield and standard of synthesized pectin and also saves sufficient amount of time and energy. [3]

Experimental study on extract pectin from peel waste of variety of citrus fruit namely lemon (Citrus lemon) and the effect of processing conditions on the process of extraction. Pectin was extracted using nitric acid at three different temperatures (40 °C, 60 °C and 80 °C) and PH (1.0, 2.5 and 4.0). Experiments carried out in water bath for two hours. The variety at various extraction conditions from the analysis the interaction effects were studied and the optimal process conditions, maximizing the percentage yield were found. Using nitric acid the yield of pectin for the variety varies from 4.69 % -20.36 %. As decrease in PH the pectin yield increased and with an increase in extraction temperature the pectin yield also increased. Maximum yield of 20.36% was obtained at PH 1.0 and temperature of 60 °C for the variety. The best condition for extraction using nitric acid was at 60 °C for 2 hours at pH 1.0. The percentage yield was minimum (5.1%) in sweet lime peel at treatment combination of pH 3.0 at 85 °C for 60 min but in case extraction temperature of 40 °C and pH of 4.0 has minimum yields of 5.69 %. [6]

Optimum conditions for the extraction process were established to be pH 3.5, temperature 65°C and time 67.5 min. The interaction effects of these variables were studied using 3-D and contour plots. A 1.5-fold increase in pectin yield was obtained as a result of this experimental design. Analysis of variance indicated the significance of the model. The pectin obtained then subjected to qualitative and quantitative analyses and found to contain desirable methoxyl, hyaluronic acid contents and degree of esterification. Functional groups present in pectin using FTIR spectroscopy. [7]

The extraction of pectin from pumpkin peels Extraction using Soxhlet with two different acids. The influence of time on pectin yield and to characterize the output determinations of methoxyl content, acetyl content, equivalent weight and degree of esterification in a laboratory on a small scale. The higher average yield of pectin obtained by using Soxhlet acid extraction (7.72% for nitric acid and 6.80% for citric acid) while the lower yield was obtained with acid extraction without using Soxhlet (6.24% for nitric acid and 5.36% for citric acid). Equivalent weight and acetyl contain of extracted pectin with both nitric and citric acids were (1250 g/mol and 0.43) respectively while methoxyl content was (6.20% and 7.23 %) degree of esterification was (66.53% and 66.57%) for nitric and citric acid respectively. Pumpkin peels are a promising commercial source of pectin. [8]

The potential of citrus peel as a source of pectin. Pectin was extracted from lemon peel powder using nitric acid and at three different temperatures, time and pH viz (60, 70 & 80 °C), (30, 45 & 60 min), (1.5, 2 & 2.5 pH) respectively. Pectin yield extracted by using citric and nitric acid as reagents medium varied from 15.8% to 67.8% and 13.8% to 44.2% respectively. The best extraction condition by both the extraction reagents showed higher in yield by using citric acid at 80°C, 60 min, 1.5 pH. The isolated pectin using nitric acid as reagents contained 510 equivalent weight

5.45% methoxyl content 65.4% anhydrouronic acid respectively. The degree of esterification of extracted pectin showed low methoxyl pectin. The ash and moisture content of isolated pectin were also determined. [12]

3.1 Applications of Pectin [5]

1. Pectin and its derivatives are used in diarrheal disorder and constipation.
2. Pectin lowers the blood cholesterol level by increasing the fecal cholesterol, fecal fat.
3. In medicine pectin increases viscosity and volume of stool.
4. In cosmetic products pectin acts as stabilizer
5. Pectin is used in confectionery jellies to give a good gel structure and a clean bite.
6. Pectin can be used to improve the mouth-feel.
7. Pectin also reduces syneresis in jams and marmalades.
8. Pectin reduces rate of digestion by immobilizing food components in the intestine.
9. It is used in the hair tonics, body lotions and shampoos.
10. It is also used in the deodorants and tooth pastes.
11. Pectin is also used in wound healing preparations and specialty medical adhesives.
12. Pectin hydrogels have been used in tablet formulations as a binding agent.
13. It has been used in gentle heavy metal removal from biological systems.
14. Pectin is also used in throat lozenges as a demulcent.
15. Pectin acts as a natural prophylactic substance against poisoning with toxic cations.
16. Pectin is used to maintain the viscosity of syrups.
17. Pectin in combination with kaolin is used to prevent ulcerated mouth and throat sores.
18. It is also use for diabetes and gastroesophageal reflux disease.
19. It is used to make artificial cherries.
20. Pectin blocks angiogenesis in cancer cells which results in death of the cancer cell.

IV. METHOD OF PRODUCTION [2]

4.1 Direct Boiling (Hot Water) Extraction Method

A long list of various agents has been reported for the extraction of pectin from plant tissues. Extraction with the hot water is the simplest and oldest method for removing the pectin substances and have very limited amount of produced. Many organic acids and their salts such as oxalic acid, ammonium oxalate, tartaric acid, polyphosphates and many others have been also used. Strong acids are corrosive and may be a potential threat to health. The liquid waste generated from the industrial processes lead to burden the environment and a high cost might incur for treating the strong acidic waste. A very low yield of pectin obtained from dried orange peel was reported using distillation apparatus as extracting at 85-90 °C. Double extraction at 85-88 °C for one hour using a cationic resin for the extraction of pectin from apple pomade has been reported to give higher yields and better gel strength of the product.

4.2 Thermo Mechanical Extraction Method

This method has been used in locations where drying facilities or solvent extraction units cannot be installed. However, these processes have poor yields and frequently require the use of chemical aids. Thermo mechanical extraction method also use high pressure boiler so it needs high steam consumption. This process has poor yields and frequently require the use of chemical aids. Thermo mechanical extraction method also use high pressure boiler so it needs high steam consumption.

4.3 Microwave Extraction Method

During microwave heating considerable pressure builds up inside a material. The high pressure then modifies the physical properties of material tissues, breaking down the cell structure and improving the capillary porous structure of tissues. This feature allows better penetration of extracting solvent into the tissues; improving the subsequent extraction of pectin. Microwave extraction also gave a higher rate and amount of extraction. 0.05 M ethylenediamine tetra acetic

acid and 1 M sodium hydroxide were added into 100 ml of distilled water separately and used to maintain pH up to 1.5. Extraction periods of 5 min, 10 min and 15 mins were used to extract the pectin. After these periods the samples were removed from the microwave filtered and allowed to cool. 25 ml of Alcohol was also added to the samples to enable precipitation of pectin. Further the samples were centrifuged at 4000 rpm for 15 mins and 50 ml of ethanol was then added for overnight precipitation just as in the case of water-based extraction method.

4.4 Acid Extraction Method [2]

The conventional acid extraction involves extracting the pectin using acidified water (pH up to 2) and heat of the solution mixture by using water bath shaker at temperatures of 80 °C. The assembly is run for 1-2 hr. duration and Pectin substances are precipitated using ethanol or isopropyl alcohol. The most commonly used acidifying materials are mineral acids including, hydrochloric, sulfuric and phosphoric acids. The acid extraction method is new technology in addition to simple to construct and operate of minimum cost.

4.5 Pectin Extraction with Combinations of Ultrasound Power

A. Pretreatment of Raw Material

Orange peels were cleaned and washed. Washed and cleaned orange peels were dried in a solar drier at 45°C followed by milling in hammer mill to 1 mm size powdered orange peel, making it convenient for pectin extraction. The powder was packed in a polyethylene bag and stored in the refrigerator until required for the extraction process.

B. Pectin Extraction

Pectin was extracted with combinations of Ultrasound Power 80 to 100 %. The citric acid concentrations at pH 1 to 2 and extraction time 10 to 30 minute. Orange peel powder 10-15 g was mixed with fixed quantity of Citric acid solution (200ml) of pH 1 to 2. Then sonicated for 10, 20 and 30 minutes at the power 80, 90 and 100 %. After Ultrasound treatment the mixture was kept at the room temperature for a while and filtered. The filtrate (containing pectin) was cooled down and then centrifuged at 6000 rpm for 30 min. Propanol was utilized to precipitate the supernatant and left still for an hour with the intention to let pectin flotation. The floating pectin was then separated by filtration. This wet pectin was dried in Tray drier at 45°C. The resulting dried pectin was milled to powdered pectin and stored for analysis. [3]

V. FACTORS AFFECTING PECTIN PRODUCTION [5]

In extraction of pectin from orange peels affect the following parameters such as PH, temperature, solvent used for extraction, time of extraction.

1. **pH:** pH is considered as one of the more crucial parameters affecting the amount and properties of extracted pectin. In literature shows that the pectin yield decreased with increasing the pH value and vice-versa.
2. **Temperature:** At the lower temperature the yield of pectin is low while at high temperature it Combustible. As compared to low and high temperature range the pectin yield is high at moderate temperature. In literature same trend is observed in pectin yield.
3. **Solvent used for Extraction:** In the literature many solvents are used for the extraction of pectin such as Citric Acid, Hydrochloric Acid, Sulphuric Acid, Nitric Acid, and oxalic acid ($C_2H_2O_4$). The high yield is obtained by using Citric Acid as a solvent. The yield of pectin extraction is reported in literature up to 55-60% by using Citric Acid as a solvent.
4. **Time of Extraction:** As the time range of extraction increased the pectin yield increases but up to a limit. The time of extraction increased to an extreme there is less effect on yield of pectin reported and also decreased from maximum level due to thermal degradation of the extracted pectin.
5. **Agitation Rate:** The yield of pectin keeps on increase with increase of agitation rate. This situation is due to the fact that increase stirring rate may reduce the thickness of the diffusion layer which can enhanced the extraction process.
6. **Liquid Solid Ratio (LSR):** The yield of pectin is increased firstly and gradually decline with increasing LSR is reported in literature. This is due to increasing of dissolving capacity when LSR is increased but reduced the

separation ability of pectin from the solution. As for the other reason, when the LSR increase from certain values the degradation of pectin increase with the decrease of pectin concentration in the solution. This was due to the low content of raw material that provided less protection for the dissolved pectin and facilitated to the degradation of pectin.

5.1 Determination of Properties of Pectin [2]

A. Color

This was done by visual observation and pectin is important as it affects appearance of gel produced.

B. Solubility of Pectin in Cold and Hot Water

The pectin samples were separately placed in a conical flask with 10 mL of 95% ethanol followed by 50 mL distilled water. The mixture was shaken vigorously to form a suspension which was then heated at 80°C for 15 min.

C. Sugar and Organic Acids

One gram of the pectin sample was placed separately in 500 mL flask each and moisture with 5 mL ethanol, 100 mL water poured rapidly, shaken and allowed to stand for 10 minutes. To this solution, 100 mL ethanol containing 0.3 mL hydrochloric acid was added, mixed and filtered rapidly, 2.5 mL of the filtrate was measured into a conical flask (25 mL), liquid evaporated on a water bath and the residue dried in an oven at 50°C for 2 hours. It has checked as it is organic compound.

D. PH Determination

The choice of the pH was made by preparing a buffer at pH 7.0 and the temperature adjusted to 28°C, the glass electrode standardized with standard buffer solution with the electrode rinsed with distilled water before inserting into the pectin solution and pH determined read off. It has been found that 5.0 of pH reading.

E. Equivalent Weight Determination

Pectin sample (5 g) was weighed into a 300 mL conical flask and moistened with 5 mL ethanol, was added to the mixture followed by 100 mL distilled water and few drops of phenol red indicator. Care was taken at this point to ensure that all the pectin had dissolved and that no clumping occurred at the sides of the flask before the solution was then slowly titrated.

F. Selected Method [1]

Pectin extraction from orange peels was done in two stages. Oil was first extracted from the orange peel samples after which pectin was isolated with acid hydrolysis technique. The inner part of the peels (albedo) contains the pectin, while the outer part (flavedo) contains d-limonene oil. Simple distillation was employed for essential oil removal from the orange peels. Extraction is the most important process in the pectin production. Pectin extraction in a hot diluted strong mineral acid solution is the most commonly used method. For extraction of pectin from the orange peels Soxhlet apparatus has been used due to low solubility of solute (pectin) in solvent. The orange peels are sun dried till their moisture content is negligible. They are then crushed fed to the Soxhlet apparatus with selected solvent. Temperature maintained at B.P. of solvent for 4-8 hours. The oil dissolved in solvent and collects at the bottom which can be separated by simple distillation. The oil collects at bottom in solvent which can be separated by simple distillation. The powdered peels are collected separately after the solvent extraction is complete. Acid solutions of pH 1.0, 1.5, 2.0 and 2.5 are prepared. Powdered peels collected are heated with the pH solutions prepared for 30 minutes (optimum time) at temperature 65 °C with continuous stirring. After cooling the solution, it was filtered with muslin cloth. The filtrate was added to double amount of ethanol and allowed to precipitate. The jelly-like precipitate formed is nothing but pectin which was subsequently washed with ethanol two times. Pectin was then dried in a hot air oven at 40 °C for 20 minutes.

VI. CONCLUSION

Pectin is soluble in hot and cold alkaline water. The pectin is polysaccharide use as a stabilizer in foods. The pectin was extracted from various citrus fruit peels and its optimization by varying pH using hydrochloric acid and citric acid. Pectin can be used to improve the mouth feel and the pulp stability in juice-based drinks and as a stabilizer in acidic protein beverages. Extraction of pectin from citrus fruit peels like Orange peels, sweet orange and lemon gives higher yield of pectin. % Yield and rate of extraction for pectin depends on parameters such as pH, temperature, solvent, time of extraction. The extraction of pectin such as Citric Acid, Hydrochloric Acid, Sulphuric Acid, Nitric Acid. The time of extraction increased to an extreme there is less effect on yield of pectin reported and also decreased from maximum level due to thermal degradation of the extracted pectin. The various methods are use for extraction of pectin from citrus peels like acid extraction, Hot water extraction method, Thermo mechanical extraction method, Microwave Extraction methods etc. As per literature acid hot water extraction using Soxhlet extraction also give better yield.

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