RESEARCH PAPER

Purification, Characterization, and Physicochemical Evaluation of Natural Polymer Isolated from Orange Peel

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ABSTRACT

The present study aimed to isolate, purify, characterize, and evaluate the physicochemical properties of pectin obtained from orange peel, an agro-industrial waste material. Pectin was extracted by aqueous extraction at different temperatures (40°C, 70°C, and 100°C) followed by acetone precipitation. The percentage yield of isolated pectin was found to be 15%. Phytochemical screening confirmed the presence of carbohydrates, while proteins, tannins, fats, volatile oils, and glucose were absent, indicating the purity of the isolated polymer. Organoleptic evaluation revealed a brown-colored powder with characteristic odor and taste. Solubility studies showed that pectin was soluble in hot water, formed a gel in cold water, and was insoluble in organic solvents. Physicochemical characterization demonstrated pH values ranging from 4.36 to 4.56, swelling index from 77.26% to 188.18%, viscosity from 8.43 to 10.18 poise, and ash content from 5.6% to 9.0%. The P3 batch (100°C) exhibited the highest swelling capacity, whereas the P1 batch (40°C) showed superior flow properties and viscosity. FTIR analysis confirmed the characteristic functional groups of pectin. The results indicate that orange peel-derived pectin possesses desirable physicochemical properties and can serve as a promising natural polymer for pharmaceutical and biomedical applications.

Keywords: Orange peel, Pectin, Natural polymer, Purification, Physicochemical characterization, Swelling index, FTIR, Pharmaceutical excipient.

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Purification of Plant Material

Fresh orange peels were collected, washed thoroughly with distilled water to remove extraneous matter, and shade-dried for a period of 12–24 hours to prevent thermal degradation of pectin. The dried peels were pulverized, sieved through mesh number 20, and used as crude plant material. The powdered material was subjected to aqueous extraction by boiling for 5 hours at different temperatures, namely 40 °C (Batch A), 70 °C (Batch B), and 100 °C (Batch C). After boiling, the extract was filtered and squeezed through a muslin cloth to separate marc from the filtrate.

To precipitate pectin, an equal volume of acetone was added to the filtrate with continuous stirring. The precipitated pectin was collected, dried in a hot-air oven at approximately 45 °C, powdered, and stored in a desiccator until further characterization and use **[1]**.

Physicochemical Characterization of Isolated Pectin

Identification Tests for Carbohydrates, Proteins and Tannins:

A 1% (w/v) aqueous solution of the isolated pectin was prepared and subjected to qualitative chemical tests to identify the presence or absence of carbohydrates, proteins, starch, fats, and tannins. All tests were performed according to standard pharmacognostical procedures to confirm the purity and nature of the isolated polymer **[2]**.

5.2.2 Solubility Behaviour

The solubility characteristics of isolated pectin were determined by dispersing one part of dry pectin powder in various solvents, including water, alcohol, and organic solvents. The mixture was shaken thoroughly, and solubility behavior was observed and recorded to evaluate the hydrophilic nature of the polymer **[3]**.

5.2.3 Organoleptic Evaluation of Isolated Pectin

The isolated pectin was evaluated for organoleptic properties such as color, odor, taste, and texture. Visual observation and sensory evaluation were carried out to assess the physical acceptability and consistency of the isolated polymer **[4]**.

5.2.4 pH of Pectin

The pH of a 1% (w/v) aqueous solution of the isolated pectin was measured using a calibrated digital pH meter. The measurement was conducted at room temperature to ensure accuracy and reproducibility **[5]**.

5.2.5 Swelling Index of Pectin

The swelling index of pectin was determined to evaluate its hydration capacity. A piece of butter paper (2 × 2 cm) was weighed, and 0.1 g of pectin powder was placed inside it. The sample was immersed in a petridish containing 15 ml of distilled water. Swelling was measured at specific time intervals (15, 30, 45, 60, 120, 240, 300, and 1440 minutes). The swelling index was calculated using the following equation **[6]**:

$$\text{Swelling weight} = \frac{\text{Initial weight - Final weight}}{\text{Initial weight}} \times 100 \quad \text{....................Equation 5.1}$$

5.2.6 Viscosity of Pectin

The viscosity of 1% (w/v) aqueous solutions prepared from different batches of isolated pectin was determined using an Ostwald viscometer. Viscosity measurements were conducted at controlled temperature conditions to assess batch-to-batch consistency **[7]**.

5.2.7 Surface Tension of Pectin

Surface tension of a 1% (w/v) pectin solution was measured using a stalagmometer. The measurements were carried out using distilled water as reference, and the results were used to study the surface activity of the polymer **[8**.

5.2.8 Bulk Density and Bulkiness

Bulk density and tapped density were determined by transferring accurately weighed pectin powder (5 g) into a graduated cylinder. The powder was tapped using a bulk density apparatus until constant volume was achieved. Bulk density was calculated using the following equation **[9]**:

$$\text{Bulk density} = \frac{\text{weight of powder}}{\text{Weight of apparent volume}} \quad \text{…… Equation 5.2}$$

Bulkiness was calculated as the reciprocal of bulk density.

5.2.9 Powder Flow Properties

Powder flow characteristics were evaluated by measuring the angle of repose using the fixed funnel method. The angle of repose was calculated using the equation:

$$\tan \theta = h/r \quad \text{....Equation 5.3}$$

where h is the height and r is the radius of the powder heap **[10]**.

5.2.10 Powder Compressibility

Compressibility of pectin powder was determined using Carr's Index. Bulk and tapped densities were measured, and Carr's Index was calculated using the equation **[11]**:

$$\text{Carr's index} = \frac{\text{tapped density-bulk density}}{\text{Tapped density}} \times 100 \quad \text{…… Equation 5.4}$$

5.2.11 Particle Size Analysis

Particle size of powdered pectin was determined by optical microscopy. Multiple readings were taken to calculate the average particle size distribution **[12]**.

5.2.12 Ash Value

Ash value was determined by accurately weighing 2 g of pectin powder in a silica crucible and incinerating it in a muffle furnace at approximately 450 °C until carbon-free ash was obtained. The residue was cooled in a desiccator and weighed to compute total ash content **[13]**.

5.2.13 Infrared Spectral Analysis of Isolated Pectin

Infrared (FTIR) analysis was carried out to identify functional groups present in isolated pectin. A mixture of pectin

(100 mg) and potassium bromide (400 mg) was compressed into a pellet under 15-ton pressure using a hydraulic press. The pellet was scanned in the range of 4000–400 cm^{-1} using a Shimadzu FTIR spectrophotometer **[14]**.

Characterisation of Pectin:

After isolating pectin from orange peel using acetone, the percentage yield of pectin was found to be 15%. Phytochemical investigation showed the presence of carbohydrates and hexose sugar but glucose, tannins, proteins and polysaccharides were absent. Results after phytochemical test are summarized in Table 5.1.

Table 5.1: Chemical characterization of isolated pectin

S. No.	Test	Batch A	Batch B	Batch C
01.	Carbohydrates	+	+	+
02.	glucose	-	-	-
03.	Hexose Sugar	-	-	-
04.	Tannins	-	-	-
05.	Proteins	-	-	-
06.	Volatile oils	-	-	-
07.	Fats	-	-	-

+ Present; –Absent

Organoleptic properties of pectin were observed and found to be acceptable. The colour of pectin powder was brown. The odour and taste was found to be characteristic and agreeable. The fracture was rough and solubility profile of pectin is shown in Table 5.2

Table 5.2: Solubility profile of pectin

Solvents	Solubility
Cold water	Swells to form gel
Hot water	Soluble
Methanol	Insoluble
Ethanol	Insoluble
Diethyl ether	Insoluble
Petroleum ether	Insoluble
Acetone	Insoluble

Solubility analysis has showed that pectin was soluble in hot water, swells to form gel in cold water and insoluble in most of the organic solvents.Different parameters of pectin were evaluated and are shown in Table 5.3.

Table 5.3: Parameters of Pectin

Parameters	P1(40 °C pectin)	P2(70 °C pectin)	P3(100 °C pectin)
pH	4.56 ± 0.05	4.56 ±0.05	4.36 ±0.05
Swelling Index (%)	77.26±35.53	104.56±49.31	188.18±81.21
Viscosity (poise)	10.18± 0.35	9.39 ±0.26	8.43 ±0.47
Surfacetension (dyne/cm)	65.14± 0.78	63.86±0.35	67.3 ±0.44
Bulk density (g/cm^3)	0.75 ± 0.06	0.79 ±0.06	0.79 ±0.06
Tappedde nsity (g/cm^3)	0.88 ± 0.09	0.94 ±0.09	0.94 ±0.09
Bulkiness (cm^3/g)	1.33 ±0.11	1.26 ±0.11	1.26 ±0.11
Hausner's ratio	1.17 ±0.01	1.26 ±0.11	1.18 ±0.01
Carr's index (%)	15.81±1.39	20.66±6.87	15.89±1.39
Angl	13.7	14.6	14.8

e of repose (°)	3±2.43	2±2.01	2±1.54
Particle size (µm)	80.30± 34.27	79.30±26.64	73.23±25.87
Total Ash (%)	5.6 ± 0.76	6.3± 0.28	9±0.5

Value with "±" shows standard deviation of triplicate study.

Swelling Index:

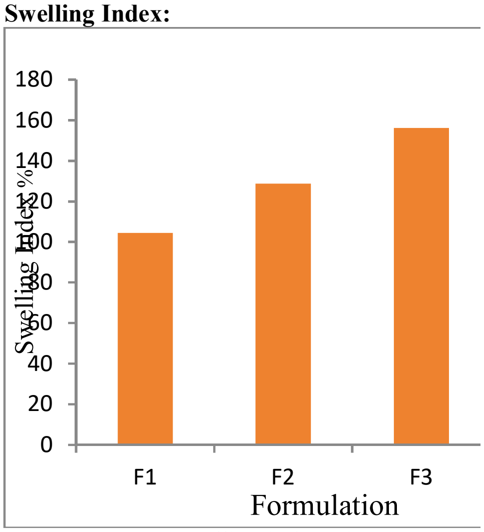


Figure 5.1: % Swelling index of pectin batches

The ash content of the P3 batch of pectin was observed to be higher compared to the other batches. The pH of the 1% solution of the P3 batch indicated greater acidity. The bulk density, tapped density, Carr's index, and Hausner's ratio of the P1 batch were lower than those of the other batches; however, the bulkiness of the P1 batch was comparatively higher, indicating improved compressibility. The angle of repose for the P1 batch was the lowest, demonstrating excellent flow characteristics. Although P2 and P3 batches also exhibited good flow properties, they were inferior when compared to P1. The P3 batch showed the highest swelling capacity among all batches. The lowest surface tension was recorded for the P2 batch of pectin. In contrast, the P1 batch exhibited the highest viscosity relative to the other batches.

FTIR Spectroscopy:

FTIR Spectroscopy of pectin at 40°C

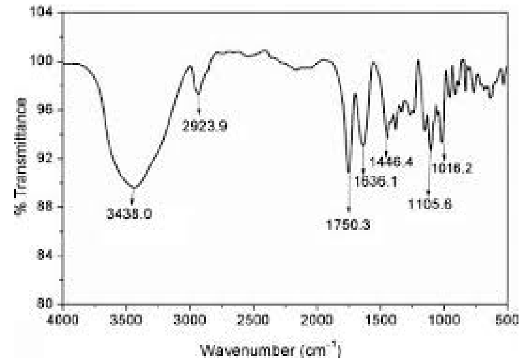


Figure 5.2: FTIR Spectroscopy of pectin at 40°C temperature

During the spectral analysis of the extracted orange peel pectin, the presence of major functional groups such as O–H, C–H, N–O, and C–O, as listed in Table 5.4, was identified. These functional groups were observed as prominent and intense peaks in the spectrum, confirming their presence. The detection of these characteristic groups provides valuable insight into the structural features of pectin extracted at 40 °C.

Table 5.4: Infra-red assignment of pectin at 40°C

Wavenumber (cm^{-1})	**Functional Group**
3612	O-H stretch
1772.82	C=O stretch
1700	COOH
732.17	C-X

FTIR Spectroscopy of pectin at 70°C

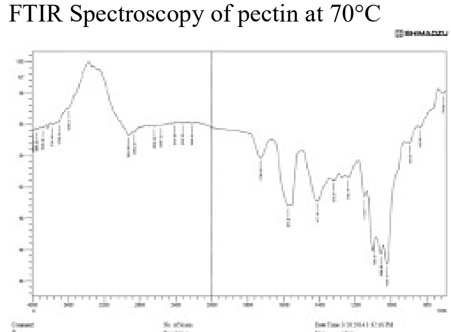


Figure 5.3: FTIR Spectroscopy of pectin at 70°C temperature

Spectral analysis of the extracted orange peel pectin revealed the presence or absence of several key functional groups, as detailed in Table 5.5, including O–H, C–H, N–O, and C–O. These functional groups appeared as distinct and intense peaks in the spectrum, confirming their characteristic signatures. The identification of these groups provides deeper insight into the structural composition of pectin extracted at 70 °C.

Table 5.5: Infra-red Assignment of pectin at 70°C.

Wavenumber (cm^{-1})	**Functional Group**
3649	O-H stretch
2922	C-C stretch
1772.82	C=O stretch
1700	COOH
732.17	C-X

FTIR Spectroscopy of pectin at 100°C

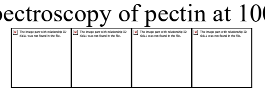
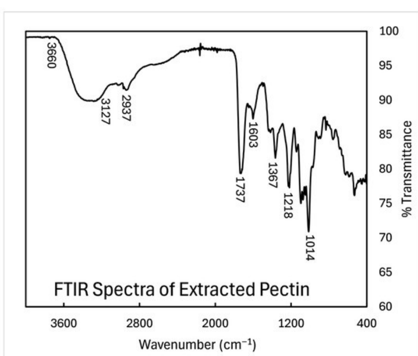


Figure 5.4: FTIR Spectroscopy of pectin at 100°C temperature

Spectral analysis of the extracted orange peel pectin indicated the presence or absence of key functional groups, as presented in Table 5.6, namely O–H, C–H, N–O, and C–O. These functional groups were represented by prominent and intense peaks in the spectrum, confirming their characteristic nature. The identification of these groups enhances the understanding of the structural properties of pectin extracted at 100 °C.

Table 5.6: Infra-red assignment of pectin 100°C.

Wavenumber (cm^{-1})	**Functional Group**
3648	O-H stretch
2304	N-H stretch
1772.82	C=O stretch
1700	COOH
1395	C-O stretch
1338	C-N
732.17	C-X



Conclusion

Pectin was successfully isolated and purified from orange peel using aqueous extraction followed by acetone precipitation, yielding 15% pectin. Phytochemical studies confirmed the presence of carbohydrates and the absence of proteins, tannins, fats, volatile oils, and other interfering constituents, indicating the purity of the isolated polymer. The extracted pectin exhibited desirable organoleptic and physicochemical properties, including good swelling ability, acceptable flow characteristics, suitable viscosity, and acidic pH. Solubility studies demonstrated its hydrophilic nature, with solubility in hot water and gel-forming ability in cold water. FTIR analysis confirmed the characteristic functional groups of pectin, validating its successful isolation. Among the batches studied, pectin extracted at 100°C showed the highest swelling capacity, while pectin extracted at 40°C exhibited better flow properties and viscosity. Overall, the findings suggest that orange peel is an economical and sustainable source of pectin, and the isolated polymer possesses properties suitable for use as a natural pharmaceutical excipient in controlled-release, matrix-forming, and other drug delivery systems.

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