



## RESEARCH ARTICLE

## PHYTOCHEMISTRY OF THE EXTRACTED PECTIN FROM *CITRUS SINENSIS* FRUIT PEELS

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### Abstract

**Aim and objective:** Pectin, a natural polymer, is often extracted from *Citrus sinensis* fruit peels under slightly acidic circumstances. It is a multipurpose pharmaceutical excipient that has been researched for its possible use in oral solid dosage forms. The study's goal was to describe the pectin that was extracted from *Citrus sinensis* fruit peels.

**Methods:** Pectin was produced using acidified water and a Soxhlet apparatus. Following phytochemical screening, the extracted pectin was calculated for its micromeritics properties, flow behavior, viscosity, and swelling index, utilize an acidified water-based extraction technique.

**Results:** Total 8.00% yield was achieved when producing pectin. The phytoconstituents of the extracted pectin include carbohydrate, protein, alkaloids, and mucilage. The organoleptic properties of the extracted pectin include; brownish, odorless, orange juice taste, amorphous powder with melting point of 151°C. The extracted pectin from *C. sinensis* fruit peel showed good flow properties (angle of repose: 29±02), an ash value of 2.03% by weight, and a loss on drying of 0.70% was discovered. The pH was found to be 3.5, suggesting that it may be consumed orally without any irritation. Extracted pectin was soluble in methanol, hydrochloric acid, and warm water. It formed lumps in cold water and was insoluble in certain organic solvents including ethanol, acetone, and alkali.

**Conclusion:** The outcomes of the evaluated parameters showed that pectin from the *C. sinensis* plant's peel might be used as a pharmaceutical excipient to make solid oral dosage forms such tablets and powder for oral suspension.

**Keywords:** *Citrus sinensis*, extraction, pectin, phytochemistry.

## INTRODUCTION

Pectin is a very valuable dietary component with several functions that is also a part of cell walls. Citrus fruits are often extracted for commercial manufacture of it and turned into a white to light brown powder. D-galacturonic acid is a linear chain that is (1, 4)-l linked and makes up the backbone of pectin<sup>1</sup>. Natural polysaccharides offer a number of advantages over semi-synthetic and synthetic polysaccharides, including low toxicity, biodegradability, biocompatibility, affordability, and ease of availability<sup>2</sup>. One such example is the pectin that has been isolated from *Citrus sinensis* fruit peels. Pectin may, however, also serve as a thickener, water binder, and stabilizer<sup>2,3</sup>. Most often, pectin is used as a gelling agent. Methoxy groups have

an impact on pectin's gelling activity, which is the main quality that makes it a useful excipient in pharmaceutical formulations. In the presence of adequate (for example, 65% by weight) carbohydrates like sucrose and at a low pH (3.5), high methoxyl pectins quickly produce thermally irreversible gels; the lower the methoxyl concentration, the slower the set<sup>4,5</sup>. In the presence of calcium ions and at low pH (3-4.5), low methoxyl pectin (50% esterified) creates thermos reversible gels.

Pectin was extracted from *C. sinensis* fruit peels and employed in the study to examine its micromeritics characteristics and physicochemical capabilities as a pharmaceutical excipient. Some of the parameters that were looked at were particle size analysis, bulk density, tapped density, actual density, angle of repose, Carr's

index, bulkiness, and evaluation of the viscosity and swelling index of *C. sinensis* fruit peels pectin. Isolated pectin from *C. sinensis* makes a fantastic tablet stabilizer and binder, respectively.

## MATERIALS AND METHODS

### Sample collection/preparation

*C. sinensis* fruits were purchased from Obla orchard in Ajobe, Otukpo LGA, Benue State. Professor G.E. Omakhua of the Faculty of Agriculture at the University of Port Harcourt's Department of Forestry and Wildlife Management certified that these fruits are of the species *C. sinensis* (L). Family Rutaceae; specimen stored as a source of information in the university herbarium under accession number UPHRO477. Osbck. Peels were removed and cleaned properly. They underwent a 14-day period of shade drying and then an additional 30–40°C oven drying time to attain a consistent weight. The dried fruit's skins were crushed and the pulverized using an electric grater. After being further filtered via sieve #20, the powdered peel was then placed in an airtight container for storage.

Pectin extraction required two stages.

#### Step 1: Extraction of Pectin

Water that had been hydrochloric acid-acidified to a pH of 2.2 was used to extract the pectin under reflux in a condensation machine for 60 minutes, with the extraction medium maintained at a temperature of 120 °C. A Whatmann cellulose thimble measuring 33 mm on the inside and 80 mm on the outside made up the extractor. *C. sinensis* fruit peel that had been dried and pulverised into a powder<sup>4,5</sup>.

#### Step 2: Isolation of Pectin

The concentrated juice was cooled to 4°C after being extracted from the hot acid in a cheese-cloth bag. Pectin was precipitated after 15 minutes of continuous stirring using a 2:1 (v/v) alcohol (ethanol) to juice ratio. The mixture was given an extra 2 hours to settle in order to enhance pectin precipitation. Before being cleaned with alcohol (95%) and crushed, the flocculating pectin coagulate was filtered through cheesecloth. The crushed pectin was further dried to a uniform weight in a hot air oven at 35–45°C. Hard pectin cake was pulverised and sieved through sieve #20 in preparation for future use<sup>4,5</sup>.

### Physico-chemical analysis of the extracted pectin

#### Test for carbohydrate

##### Molisch's test

The test was performed on the resultant 1% w/v extracted pectin-solvent system, which was created by dissolving 500 mg of the extracted pectin in 50 mL of filtered water. To 2 mL of the extracted pectin-solvent system, two drops of the  $\alpha$ -naphthol solution were added. The 1% w/v extracted pectin-solvent solution was placed in an inclined test tube, and concentrated sulfuric acid was introduced in drips. The color change was seen and noted.

#### Test for protein

##### Million's test

A 2 mL of the extracted pectin-solvent system received a few drops of Million's reagent before being inspected. The precipitate developed was noticed<sup>6</sup>.

#### Tests for alkaloids

##### Dragendorff's test

A 2 mL volume of the extracted pectin-solvent mixture was combined with a few drops of Dragendorff's reagent (Bismuth and potassium iodide), and the color change was observed and recorded<sup>6</sup>.

##### Wagner's test

Wagner's reagent, a solution of sodium and potassium iodide, was added to a 2 mL sample of the extracted pectin-solvent combination, and the resulting color change was observed<sup>6</sup>.

##### Hager's test

A little amount of Hager's reagent (saturated picric acid solution) was added to 2 mL of the extracted pectin-solvent mixture to observe and record the color change<sup>6</sup>.

#### Test for saponins

##### Frothing test

A 2 mL of freshly prepared distilled water and 2 mL of the 1% by volume extracted pectin-solvent system were added to a test tube, and the mixture was vigorously agitated. After that, the test tube was examined, and the observation was noted for future use<sup>6</sup>.

#### Test for flavonoids

##### Ammonium test

A 2 mL of diluted ammonia solution were mixed with around 2 mL of the extracted pectin-solvent system. A few drops of strong sulfuric acid were added, and the consequences were immediately apparent<sup>6</sup>.

##### Aluminum chloride test

Results were seen after adding a few drops of a 1% w/v aluminum chloride solution to a 2 mL sample of the extracted pectin-solvent combination<sup>7</sup>.

#### Test for tannins

##### Ferric chloride test

Two drops of a solution containing ferric chloride at 0.1% by volume were added to about 2 mL of the extracted pectin-solvent system, and the result was reported<sup>6</sup>.

### Determination of ash values of *C. sinensis* fruit peels

#### Total ash

Three completely dry and clean nickel crucibles were heated for about 15 minutes at 35°C, then allowed to cool for almost an hour in a desiccator. Then the crucibles were weighed and given a W0 tag. Each nickel crucible W1 received a weighted transfer of 2 g of the drug substance. These were slowly heated up until all the drug ingredients were burned, the carbon had evaporated, the residue was carbon-free, and the ash was white. Two drops of a solution containing ferric chloride at 0.1% by volume were added to around 2 mL of the extracted pectin-solvent system, and the result was reported<sup>7</sup>.

$$\text{Total ash} = \frac{W2 - W0}{W1}$$

Where W0 represents the weight of the empty crucible, W1 represents the initial weight of the drug before burning, and W2 represents the weight of the crucible and sample after cremation.

**Water soluble ash**

The whole ash was heated for 5 minutes with 25 mL of filtered water. Whatmann's No. 1 filter paper, famed for its weight and lack of ash, was used to filter this mixture (W0). After washing the residue with hot water, the filter paper was then dried in an oven at 105 °C until a constant weight (W2) was obtained. The amount of water soluble ash was determined using the formula shown below:

$$\text{Weight of residue} = W_2 - W_0$$

$$\text{Water soluble} = \frac{\text{Weight of total ash} - \text{weight of residue}}{\text{Weight of dried drug material}} \times 100$$

Where W0 represents the weight of an empty crucible, W1 represents the weight of the drug material prior to incineration, and W2 represents the combined weight of the crucible and the ash<sup>8</sup>.

**Organoleptic examination of the extracted pectin**

The color, odor, taste, form, and texture of the extracted pectin powder were examined, and these characteristics were noted.

**Moisture absorption/hysteresis of the extracted pectin**

The experiments were conducted at various relative humidity levels, including 52% RH (potassium nitrate), 75% RH (sodium chloride), 84% RH (potassium chloride), and 96% RH (potassium sulfate). These salts were completely dissolved in some recently distilled water and then allowed to sit for seven days. After weighing the empty containers (W0), the extracted pectin (0.5 g) was added to the different previously weighed containers and reweighed (W1). These containers containing the extracted pectin W1 were placed for 72 hours in various desiccators with varying relative humidity. Each container was reweighed together with its contents after 72 hours for the final weight W2.

Where the extract weight is W1-W0, Change in weight of extract is W2-W1

$$\% \text{ Moisture absorption} = \frac{W_2 - W_1}{W_1 - W_0} \times 100$$

**Swelling Index of the extracted pectin**

Total 1 g of the extracted pectin was accurately weighed and transferred into a 50 mL graduated measuring cylinder and tapped to obtain the tapped volume, v<sub>t</sub>. Then, a dispersion of the powdered pectin extract was made in 40 mL of distilled water and thoroughly agitated. The volume was made to 50 mL with more distilled water and allowed to stand undisturbed for 24 h on a flat surface and the volume of the sediment formed, v<sub>s</sub> noted. Triplicate determinations were done, and the swelling index for the extracted pectin was calculated as a percentage using the formula<sup>9</sup>.

$$\text{S. I.} = \frac{V_s - V_t}{V_t} \times 100$$

Where v<sub>s</sub> is the volume of sediments, v<sub>t</sub> is the trapped volume.

**pH determination of the extracted pectin**

The extracted pectin-solvent system included 2% w/v of the extracted pectin after being prepared up to 50 mL in distilled water with 1 g of the powdered

extracted pectin and vigorously shaken for 3 minutes. Using a pH meter from Hanna® USA, the pH was measured<sup>10</sup>.

**Solubility of the extracted pectin**

After a few drops at a time of distilled water were added, a piece of the extracted pectin weighing 0.5 g was submerged, vigorously swirled, and the results were observed. The identical procedure also included the use of acetone, methanol, alkali, and 0.1 N HCl solution<sup>11</sup>.

**Relative viscosity of the extracted pectin-solvent system**

Total 4% w/v Weighing and dissolving 2 g in distilled water to a volume of 50mL produced the stock dispersion of the extracted pectin-solvent system. Distilled water was used to create successive dilutions of the dispersion that produced corresponding concentrations of 0.05, 0.125, 0.5, 1.0, 1.5, 2.0, and 2.5% w/v. The average flow time of the several serial dilutions of the dispersion at room temperature was determined using a U-tube viscometer (PSL, England). Additionally, it was established what the mean flow rate of cleansed water was.

The pectin extract dispersion's viscosities at various concentrations were calculated using the equation.

$$\text{Relative viscosity, } n = k \rho t$$

Where n is the solvent's viscosity, ρ its density, k its viscosity constant, and t its period of flow for the extracted pectin<sup>12</sup>.

**Intrinsic viscosity of the extracted pectin-solvent system dispersion**

In the determination of intrinsic viscosity, the least concentration of the dispersion which is the concentration closest to that of the solvent was used, and in this case 0.05% W/V.

$$N_{rel} = t/t_0$$

Where t and t<sub>0</sub> are the time of flow for the pectin extract-solvent system and distilled water respectively.

Then, using Bill Meyer's equation, the intrinsic viscosity, N<sub>int</sub> could be calculated;

$$\tilde{N}_{int} = \frac{0.25 \times [N_{rel} - 1 + 3 \times \ln(N_{rel})]}{C}$$

Where N<sub>rel</sub> =relative viscosity, C=concentration of the extracted pectin-solvent system. N<sub>int</sub>=intrinsic viscosity.

**Estimation of the molecular weight of the extracted pectin**

Mark and Houwink independently linked the intrinsic viscosity and molecular weight in 1938 and 1940, respectively.

$$[N_{int}] = kM^a$$

The Mark-Houwink constants, k and a, were determined empirically by measuring the inherent viscosity of several pectin polymer samples, for which the molecular weightiness was confirmed using an independent approach such osmotic pressure or light scattering<sup>13</sup>. M is the molecular weight.

**Loss on drying**

In order to achieve a consistent weight, one gram of hydrogel powder was accurately weighed in a tared glass stoppered container, dried at 105°C in a hot air oven, and the weight was checked every hour. The powder's contribution to weight loss was calculated<sup>5,6</sup>.

**True density**

The current study made use of the liquid displacement technique, one of numerous options for figuring out true density. Considering that pectin is both insoluble and heavy in n-hexane, n-hexane was selected as the liquid for displacement. This technique has been embraced by numerous authors<sup>5,10,11</sup>.

**Angle of repose and the flow rate**

The angles of repose of the extracted pectin were discovered by modifying the static approach somewhat. The top end of a plastic pipe with an internal diameter of 4 cm and a length of 13.5 cm was filled with 50 g of the extracted pectin. The pipe was open at both ends. The extracted pectin powder was discharged through an elevated pipe to produce a heap of the appropriate height, h, and the diameter of the heap's edge could be measured precisely without distorting it.

**Bulk density**

A 25 mL glass measuring cylinder was dried, cleaned, and filled with the ground-up, 20 g amount of extracted pectin before being placed on a level, flat surface. It was possible to see how much space the grains occupied.

**Tapped density**

A 25 mL measuring cylinder that was clean, dry, and set on a level, smooth surface was filled with a 20 g amount of the extracted pectin powder after it had been precisely weighed using an electronic digital scale. Once a steady volume was attained, the measuring cylinder was tapped repeatedly on the flat, smooth surface at a height of roughly 4 cm.

**Melting point determination of the extracted pectin powder**

The melting point of the extracted pectin powder at room temperature was determined using the open capillary technique and the Stuart melting point instrument (Bibby Scientific Ltd., UK)<sup>15</sup>.

**Scanning electron microscopy (SEM)**

SEM was used to clarify the morphologic characteristics of the powdered pectin that had been extracted. A glass rod was used to apply a 5 mg sample of the powdered extract that had been dissolved in distilled water on a microscope slide. An automated imaging device called a motic image analyzer that was connected to a polarized photomicroscope (Hind

Weltzar Germany) and operated at a magnification of X100 was used to observe the combination while it was covered with a cover slip<sup>5</sup>.

**Powder x-ray diffraction (PXRD) of the extracted pectin**

By employing a Panalytical Xpert Pro Diffractometer to analyze the powder at room temperature for x-ray diffraction patterns, the extracted pectin's patterns were determined (PANalytical J B Eindhoven, Netherlands). Cu target metal, K filter, 40 kV voltage, and 40 mA current made up the test circumstances<sup>16</sup>.

**RESULTS AND DISCUSSION**

**Yield of the pectin extracted from *C. sinensis* fruit peel**

An alcoholic extraction of *C. sinensis* fruit peels was carried out in acidic medium and pectin extract was obtained. The percentage yield was 8% w/w as shown in Table 1. This confirms earlier report, though, lower value as earlier report ranges from 12-14%<sup>17</sup>. This may be due to the differences in the working temperatures. For their work, the temperature was 95°C and the period of extraction was 105 min., and the pH used was 1.5. Whereas, in this case the working temperature, extraction period and pH were 120°C, 45 min. and the pH 2.2 respectively. The higher temperature of 120°C may have resulted in the degradation of the extracted pectin. For maximum extract pectin yield; the pH should be lower than 2, the extraction period must be long enough, about 2 h and the temperature should be lower than 100°C. This agrees with the earlier findings<sup>18,19</sup>. The yield also decreases with increase in fruit maturation<sup>20</sup>.

**Phytochemical evaluation of the extracted pectin**

When the extracted pectin's components were examined, it was found to include mucilage, alkaloids, protein, and carbohydrates, as indicated in Table 1. A drug substance's ash values, which indicate its level of purity and/or adulteration, were assessed. 3.68, 1.65, and 2.03%, respectively, were the results for total ash, water insoluble ash, and water soluble ash. The extracted pectin was deemed to be pure based on the low amounts of ash<sup>21</sup>.

**Table 1: Physicochemical characteristics of the extracted pectin.**

Parameter	Result
Extraction yield (%)	8.0
Loss on drying (%)	0.70
Carbohydrate	+
Protein	+
Alkaloids	+
Mucilage	+
Gum	+
Saponins	-
Flavonoids	-
Tannins	-
Total ash	3.68±0.17
Acid insoluble (%)	2.43±0.00
Water insoluble (%)	1.65±0.01
Water insoluble (%)	2.03±0.00
Surface tension (dynes/cm <sup>2</sup> )	35.20±0.15

KEY: + Presence, - Absence.



The extracted pectin's phytochemical components are consistent with research on *C. sinensis* peels<sup>22</sup>. The calculated ash levels also accord with previously reported values, which range from 2 to 6%<sup>23</sup>.

**Organoleptic properties**

Organoleptic evaluations are sensory characterization that helps in the identification of a crude drug material and/or excipient. Organoleptic evaluation of *C. sinensis* alcoholic pectin extract was carried out using sensory organs to evaluate the texture, color, odor, and taste of the extracted pectin<sup>24</sup>. The result revealed that it was a rough and irregular, brownish and odorless

amorphous powder with orange juice taste, amorphous powder as shown in Table 2. This is in agreement with earlier reports on extracted pectin<sup>25</sup>.

**Moisture absorption capacity**

Moisture absorption capacity of a material gives an idea on its ability to absorb and retain moisture, thus, aids dosage formulators on choice of packaging materials and storage conditions. The extracted pectin had different moisture absorption capacity depending on the relative humidity; at 54, 75, 84, and 96 % RH, the moisture absorption capacity was determined to be 20, 08, 20, and 24 % respectively.

**Table 2: Physico-chemical evaluation of the extracted pectin.**

Parameter	Extracted pectin	Parameter	Extracted pectin
Color	Brown	Solubility;	
Odor	Odorless	In cold water	Insoluble/ lumps.
Taste	Orange fruit juice.	In hot water	Suspension/gel.
Texture	Rough, irregular	Acetone	Insoluble
52 % RH	20±0.01	Ethanol	Insoluble
75 % RH	08±0.00	Alkali	Insoluble
84 % RH	20±0.00	Methanol	Soluble
96 % RH	24±0.00	Dil. HCl	Soluble
Swelling index (%)	2.30±0.01	Relative viscosity (cP) 0.05 %w/v	3.73
pH	3.5±0.2	Intrinsic viscosity (cP) 0.05 %w/v	33.45
		Estimated molecular weight (g/mol)	1.44 x10 <sup>5</sup>

This result indicated that the extracted pectin was most stable at 75 % RH as it absorbed the least quantity of moisture at this relative humidity as shown in Table 2. The implication of the findings is that hygroscopic and/or deliquescent drugs should not be formulated into dosage form using pectin extract as an excipient; a binder, a suspending agent, and/or carrier. However, if used in such formulations, the medicament must be packaged in air-tight containers<sup>26</sup>.

**Swelling index**

The swelling index measures the volume (in mL) that 1 g of test material takes up as it swells under certain circumstances. The swelling index of a substance controls how the granules break apart during dissolution, how medications are released from their dosage form, and how quickly the medication is absorbed. According to data in Table 2, the extracted pectin's swelling index was 2.30±0.01. This indicates that the extracted pectin retains almost twice as much water compared to the amount that was retained in its dry powder. This is a sign that if any of these excipients were utilized in the formulation of granules and/or tablets, they would probably operate as effective disintegrates, releasing the active medicinal components quickly for dissolving and subsequent absorption<sup>14</sup>.

**pH determination of the extracted pectin**

The point of release/dissolution of a formulation's medication and subsequent absorption are largely influenced by its pH. The pH of the gastrointestinal system that an oral dose form travels through affects how quickly the medicine is released. According to Table 2, the isolated pectin-solvent-system had a pH of 3.50±0.2, which is close to the stomach's pH. It

improves oral formulations and makes it safe and pleasant for the patient<sup>10</sup>.

**Solubility tests for the extracted pectin**

One of the requirements for an oral formulation is the solubility of the medication substance and/or excipient since this determines how easily the dosage form will dissolve and how quickly the patient will absorb it. Insoluble and lumpy in cold water, the extracted pectin was thoroughly dissolved in hot water. It was soluble in methanol and hydrochloric acid but not in alkali, acetone, or ethanol. The outcome is shown in Table 2<sup>25</sup>.

**Relative viscosity and intrinsic viscosity**

Thickeners and viscosity boosters are substances that cause suspension. For 0.05% w/v dispersion, the extracted pectin's relative and intrinsic viscosities were 3.73 and 33.45 cP, respectively. At room temperature, the viscosity was around 0.91cP<sup>10</sup>. It was discovered that the extracted pectin has a greater viscosity than water, reconstituting it tends to make the solution more viscous. These calculations were done at a concentration of 0.05% w/v, while the extracted pectin concentration needed to provide suspending action was 3.0% w/w. This suggests that viscosity improvement would be more effective at this greater concentration. The extracted pectin-solvent system is non-Newtonian (dilatant) at this greater concentration, however, and it expands when agitated<sup>25</sup>.

**Estimated molecular weight of the extracted pectin.**

The pectin that was extracted is a polymer made up of many monomer units fused together. It has a large molecular weight as a result. According to Table 2, the computed estimated molecular weight was 1.44 x 10<sup>5</sup> g/mol. This study's estimated molecular weight falls between the 5.0x10<sup>4</sup> to 1.8x10<sup>5</sup> g/mol range described

by other researchers<sup>26,27</sup>. As a result, adding the extracted pectin to the dispersion medium will undoubtedly make it more viscous and/or thicken the vehicle, making it an excellent suspending agent.

#### Flow properties of the extracted pectin

Table 3 displays the findings of a micromeritics analysis on the bulk density, bulkiness, true density, total porosity, and powder flow behavior of pectin. The bulkiness grade identifies powder as having a "heavy"

composition. Pectin's flow characteristics were acceptable.

#### Angle of repose

The computed range of the angle of repose for the extracted pectin is  $29.00 \pm 0.02$  and is shown in Table 3. A substantial difference, though ( $p < 0.05$ ), was seen. This suggests that there will be excellent flow into the end container, the extracted pectin powder would not cling together, and the tablets will likely produce well with little fluctuation in tablet weight<sup>28</sup>.

**Table 3: Micromeritics properties of the extracted pectin powder.**

Parameter	Result
Bulk density (g/mL)	$0.35 \pm 0.01$
Tapped density (g/mL)	$0.42 \pm 0.00$
Angle of repose ( $^{\circ}$ )	$30 \pm 0.04$
Flow rate (g/sec)	$5.46 \pm 0.01$
Carr's (%)	$15 \pm 0.01$
Hausner's quotient	$1.2 \pm 0.00$
Particle density (g/mL)	$1.57 \pm 0.01$
Melting point	$151 \pm 0.33$

#### Bulk and Tapped density

The bulk and tapped densities of the extracted pectin powder are shown in Table 3 after analysis. The bulk densities of the extracted pectin powder were consistently lower than those of the tapped powder, indicating that the powder's volume was decreased during tapping. The calculated bulk density for the extracted pectin was  $0.35 \pm 0.01$  and the tapped density was  $0.42 \pm 0.00$  g/mL. The bulk densities did not greatly alter ( $p > 0.05$ ). The tapping densities were similar as well.

#### Hausner's quotient and Carr's index

These positive flow indices suggest that the extracted pectin will be present as loose powder in the final pack/containers or tableting. Good flow-ability is preferred in order to effectively fill dies and create tablets with little weight variation<sup>28</sup>.

#### Flow rate of the extracted pectin powder

The extracted pectin's computed flow rate was  $5.46 \pm 0.01$  g/min, which is an excellent flow property for filling final containers and would be suitable for making tablets and powder for suspension with good physical qualities, including consistent weight and content, hardness, and friability. Although there was a significant difference ( $p < 0.05$ ).

#### Particle density

According to Table 3, the calculated particle density was  $1.57 \pm 0.01$ . This is the density of the tiny particles that make up the powder when there is no space, liquid, or air present<sup>29,30</sup>.

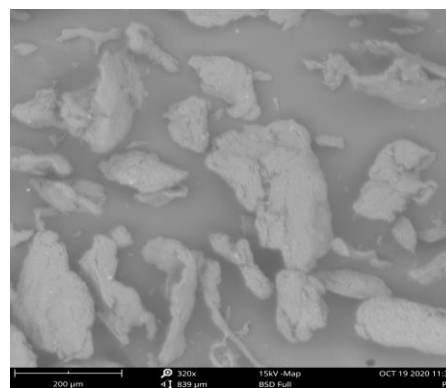
#### Melting point determination of the extracted pectin

Table 3 displays the determined melting point for the pectin powder. A melting point of  $151^{\circ}\text{C}$  was reached. Due to this, the extracted pectin is stable and able to remain in a solid powder state at temperatures lower than  $151^{\circ}\text{C}$ . The finding, which ranges between  $150$  and  $154^{\circ}\text{C}$ <sup>31</sup>, is consistent with earlier studies. Because the extracted pectin has a high melting point, care should be made to prevent melting point depression when combining it with active pharmaceutical

ingredients<sup>32</sup>. This will prevent the product's melting point from unduly falling below that of its constituents.

#### Scanning electron microscopy (SEM) of the extracted pectin

The surface morphology of the produced bio-based material, which included pectin derived from *C. sinensis* fruit peels, was shown in a micrograph, and it had a rough, uneven appearance with some fibers in it. Furthermore, the size and form of the polysaccharide particles varies. The forms of the particles vary and they might be smooth, very irregular, or seem as unequal pores. Since various polymers were included in the film, it is evident that this had an impact on the particles' morphology. It's possible that drying in shade is to blame for this rough and uneven look. With past findings<sup>33</sup>, this outcome is consistent.



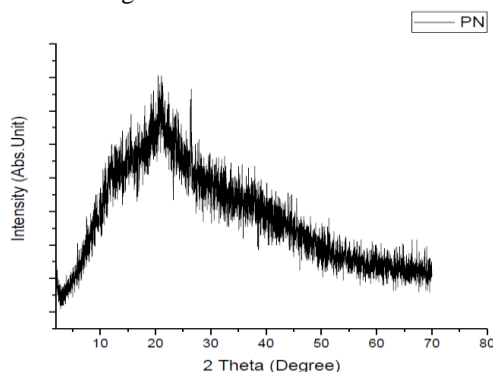
**Figure 1: Micrograph of pectin.**

#### X-Ray diffraction for pectin powder

The examination of the crystal structure of crystalline materials for powder is done using X-ray diffraction. Additionally, it displays the sample's chemical make-up for analysis. It has the ability to identify the main, minor, and trace chemicals in a sample.

The Worldwide Centre for Diffraction Data, or ICDD, international database is used for the X-ray diffraction analysis, which also contains the mineral name of the material, chemical formula, crystalline system, and

reference pattern number. When looking at the x-ray diffractogram of the pectin extracted from *C. sinensis* fruit peels, it is possible to see a large humped peak at 2 degree that is about 15°, 19.5°, 20°, and 27°, revealing the amorphous nature of the powdered pectin as shown in Figure 2<sup>33,37</sup>.



**Figure 2: X-Ray diffraction for pectin powder.**

The following elements are present in the chemical composition: graphite (0.708), quartz (0.876), brushite (1.169), hydrophilite (0.708), pyrochlore (0.916), lime (1.121), calcite (1.195), and calcium silver; silicate of aluminum, hydration 3.088<sup>38</sup>.

#### Limitations of the study

Detection of the constituents of the extracted pectin from *C. sinensis* which include, but, not limited to carbohydrate, mucilage, gum, alkaloids, and proteins.

#### CONCLUSIONS

Previous researchers showed that the yield of pectin extracted from *C. sinensis* fruit peels was 8% weight-for-weight. The result for the organoleptic properties revealed rough, irregular, brownish and odorless amorphous powder with orange juice taste. On the basis of evaluated parameters present study concludes that pectin extracted from the *C. sinensis* plant's peel might be used as a pharmaceutical excipient to make different solid oral dosage forms like tablets, and powder for oral suspension.

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#### AUTHOR'S CONTRIBUTION

**Ubieko EA:** writing original draft, conceptualization, methodology, investigation. **Onugwu AL:** Writing, review, and editing. **Ogbonna JDN:** writing, review, analysis. **Okoye E:** writing, review, and editing.

**Nwakile CD:** methodology, investigation, formal analysis. **Attama AA:** conceptualization, methodology, investigation. All authors read and approved the final manuscript for publication.

#### DATA AVAILABILITY

Data will be made available on request.

#### CONFLICT OF INTEREST

No conflict of interest associated with this work.

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