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Green extraction and characterization of Durian (*Durio zibethinus* Murray cv. 'Puyat') rind pectin

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Abstract

The durian industry generates a significant amount of waste. In this study, durian rind was used for the extraction of pectin using citric acid. Extraction temperature was varied at 80°C, 90°C, and 100°C. The optimum extraction was 90°C, with a yield of 27.99%. The moisture and ash content of durian rind pectin were 10.45% and 4.43%, respectively, which are within the limit set by the International Pectin Producers Association. A low Water Activity (Aw) (0.57%) means it is shelf-stable. Pectin was characterized in terms of equivalent weight and methoxy content which were 412.39 g/mol and 8.26%, respectively. The anhydrouronic acid content of 89.56% indicates a high-purity pectin. Its 52.35% degree of esterification classifies it as high methoxyl. The Fourier Transform Infrared Spectroscopy (FTIR) spectra had characteristic peaks at 3493.48, 3288.57 and 2957.56, and 1712.35 cm^{-1} corresponding, respectively, to –OH, –CH, and C=O, which was comparable to commercial pectin. The findings of this study align with the SDG 12 on responsible consumption and production by reducing waste generation through the valorization of agricultural waste products.

Keywords: Citric acid, Durian rind, Green extraction, High-methoxyl, Pectin

1. Introduction

Fruits are good sources of vitamins, minerals, and phytochemicals that promote good health in humans. These are either consumed raw or processed to preserve color, texture, and taste while extending their shelf life. Over the years, the production and processing of fruits have significantly increased due to the growing population and changing diet habits [1]. However, these generate a significant amount of waste. Around 25–30% of the total fruit weight, including pomace, peels, rind, and seeds are in landfills [2]. These can be sources of bioactive compounds, hemicelluloses, cellulose, starch, pectin, and dietary fibers that can be used in various products [1].

Durian (*Durio zibethinus*), known as the "King of Fruits," grows abundantly in the Davao Region of the Philippines. Annual durian production in Davao City typically reaches up to 12,000 tons [3]. The durian rind or husk, which is the outer shell of the fruit, accounts for 60% of the fruit weight and mostly ends up in the landfills [4]. However, durian rind contains a high amount of cellulose which is used as an alternative raw material for making paper and hydrogel bandages [5,6]. Aside from cellulose, durian rind also contains pectin (18.6%) which has industrial applications [7], and the yield varies differently on the variety, maturity, extraction solvent and method.

Pectin is a heterogeneous polysaccharide class containing mainly 1,4-linked α-D-galacturonic acid residues and is present in plant cell walls [8]. Its functionality is strongly influenced by its structural characteristics. The degree of esterification, which refers to the proportion of the methyl esterified galacturonic residues, is a key determinant of the gelling behavior. High methoxyl pectins (>50%) gel is found in the presence of high sugar and low pH, where low methoxyl pectin (<50%) requires calcium ions for gelation. According to established quality standards, good-quality pectin typically exhibits an anhydrouronic acid (AUA) content of at least 65%, a methoxyl content above 7% for high methoxyl pectin, and an equivalent weight ranging from 400 to 1000 g/mol. It is primarily obtained from apple pomace and citrus peels [9]. Agricultural wastes such as banana peels, mango peels, durian rind, and jackfruit peels are also considered sources of pectin [10–12]. This polysaccharide can be utilized as a thickener, gelling agent, stabilizer, emulsifier, fat replacer in low-calorie foods, and excipient in the pharmaceutical and food industries [13,14].

Acid extraction under hydrothermal conditions is the most widely employed method for extracting pectin from plants. Traditionally, mineral acids such as hydrochloric, sulfuric, and nitric acid have been utilized for pectin extraction. However, the solvents' toxicity and environmental risks have raised concerns about their continued used [15]. Moreover, the addition of mineral acids can introduce toxic components into the end product. In contrast, organic acids such as citric, tartaric, and lactic acids, though weak acids, are non-toxic and environmentally friendly. Recently, organic acids have gained wide attention for pectin extraction from agricultural by-products [16,17]. Notably, citric acid is classified as Generally Recognized as Safe (GRAS) for human use as a direct food ingredient by the Food and Drug Administration. These are very promising materials, and could lead to further investment in clean technologies, resource efficiency, recycling, and reuse of waste [18].

The global pectin market has seen substantial growth, with projections ranging from USD 1.1–1.5 billion in 2024–2025, and forecast estimating a rise to USD 2.0–3.0 billion by 2030–2037 [19,20]. This expansion is driven by the increasing demand for natural, clean-label ingredients across food, pharmaceutical, and care industries. Pectin multifunctionality is widely used in food and pharmaceutical industries, commonly sourced from citrus peels and apple pomace. However, there is limited research on the extraction of pectin from underutilized tropical fruit by-products such as durian rind, particularly using food-grade citric acid as an alternative to strong mineral acid. This study investigates the potential of durian rind, a commonly discarded waste material. The use of citric acid provides a sustainable alternative and eco-friendly approach to the production of pectin from durian rind. This research aligns with current targets on Sustainable Development Goal (SDG) 12 that aims for responsible consumption and production, as well as the application of principles of green chemistry, which utilizes renewable resources. This study aimed to extract pectin from durian rind using citric acid. Specifically, it aimed to determine the yield of pectin at varying temperatures and assess the physicochemical properties of the extracted pectin.

2. Materials and methods

2.1 Preparation of durian rind

Durian (Puyat) rinds were obtained from Belvis Farm, Calinan, Davao City. Samples with no discoloration or foul odor were selected and cleaned with tap water to remove any dirt. The white portion of the rind was removed and crushed using a blender. Crushed samples were stored in the freezer for further use.

2.2 Bacterial isolates

Pectin was extracted using citric acid based on the methods described by Gragasin et al. [21] and Jong et al. [22] with some modifications. Durian rind (250 g) was placed in a beaker with 1000 mL distilled water that was acidified to pH 2.0 using citric acid and a solid-to-liquid ratio of 1:4g/ml. The mixture was incubated at 80°C, 90°C, and 100°C for 60 minutes on a hot plate. The mixture was cooled to 60°C and filtered in a 200 mm mesh nylon sheet to obtain the pectic liquor. Afterwards, absolute ethanol (1:1) was added to the extract. The mixture was left for 30 min to allow complete precipitation of pectin and was separated by filtration. The resulting pectin was washed twice with 95% ethanol and was oven-dried at 60°C for 24 h until constant weight was obtained. The pectin was pulverized and stored in an airtight container. The percent recovery of pectin was calculated using the formula:

$$Yield(\%) = \frac{weight\ of\ dried\ durian\ pectin\ (g)}{weight\ of\ durian\ rind\ (g)} x100 \quad (1)$$

2.3 Moisture content and water activity (A_w)

The moisture content and Aw of the sample were determined using an infrared moisture analyzer (Shimadzu, Japan) and water activity meter (Aqualab Lite, USA), respectively.

2.4 Ash content

In a pre-weighed crucible, 2 g Durian Rind Pectin (DRP) was weighed and placed in a muffle furnace at 600°C for 6 hours. The ash content of the sample was calculated using the formula:

$$Ash(\%) = \frac{weight\ of\ ash\ (g)}{weight\ of\ pectin\ (g)} x100 \quad (2)$$

2.5 Equivalent weight

The method used in this study was based on the procedure of Jong et al. [23]. DRP (0.5 g) and 5 mL of 95% ethanol were placed in an Erlenmeyer flask. A 100mL of 1% NaCl and 6 drops of phenol red as an indicator were added to the mixture. The mixture was titrated with 0.1 N NaOH until a shade of light pink persisted for 30 seconds (Solution A). The equivalent weight (EW) was calculated using the formula.

$$EW = \frac{Weight\ of\ the\ pectin\ sample\ (g)}{ml\ of\ NaOH\ x\ Normality\ of\ NaOH} x1000 \quad (3)$$

2.6 Methoxyl content

Twenty-five milliliters of 0.25 N NaOH were added to Solution A and allowed to stand for 30 min. After which, 25 mL of 0.25 N HCl was added. The solution was titrated with 0.1 N NaOH until a brownish purple color was reached (Solution B). The methoxyl content (MeC) was calculated using the formula:

$$MeC(\%) = \frac{ml\ of\ titrated\ NaOH\ x\ Normality\ of\ NaOH\ x\ 3.1}{weight\ of\ the\ pectin\ sample\ (g)} \quad (4)$$

2.7 Total anhydrouronic acid content

Estimating anhydrouronic acid (AUA) content is essential to determine the purity and degree of esterification and evaluate the extracted pectin's physical properties from durian rind. Total AUA content was calculated using the formula:

$$Total\ AUA = \frac{176\ x\ 0.1z\ x\ 100}{W\ x\ 1000} + \frac{176x\ 0.1y\ x\ 100}{W\ x\ 1000} \quad (5)$$

Molecular unit of AUA (1 unit) = 176 g

z: mL of NaOH from equivalent weight determination

y: mL of NaOH from methoxyl content determination

W: weight of the sample

2.8 Degree of esterification

The degree of esterification (DE) of DRP was calculated using the formula:

$$DE(\%) = \frac{176\ x\ \%\ MeC\ x\ 100}{31\ x\ AUA(\%)} \quad (6)$$

where,

% MeC: methoxyl content

% AUA: anhydrouronic acid content

2.9 Fourier Transform Infrared Spectroscopy (FT-IR) analysis

DRP with the highest degree of esterification (DE) and commercial pectin was submitted to the Technology Transfer Business Development Office (TTBDO), University of the Philippines Mindanao for Fourier transform infrared spectroscopy (FTIR) analysis.

2.10 Statistical Analysis

Data were analyzed using one-way analysis of variance (ANOVA) and Tukey's Honest Significant Difference (HSD) at $p < 0.05$ level of significance to determine if there were significant differences among treatments. These tests were performed using SPSS software (version 29).

3. Results and discussion

3.1 Extraction of Durian Rind Pectin (DRP)

Pectin yield depends on factors such as the plant material, the acid used, and the extraction time and temperature. The percent yield of extracted pectin from durian rind using citric acid at pH 2.0 for 60 min at 80°C, 90°C, and 100°C ranged from 23.19–29.65%. In the case of Jong et al. [23], lower pectin yield (6.96–9.49%) was observed using the same solvent at 0.1 mM, 90°C for 60 minutes. Spinei and Oroian [24] added that the use of citric acid gave higher pectin yield (6.01) compared to sulfuric acid (5.80%) and nitric acid (5.84%). The utilization of mineral acids (sulfuric, nitric, hydrochloric, etc.) for pectin extraction has been associated with having detrimental environmental consequences. They are also costly [24,26]. On the other hand, organic acids (acetic, citric, etc.) provide a greener method for extracting pectin [24].

As shown in Table 1, extraction temperature had an effect on the pectin yield. Higher extraction temperatures resulted in a higher pectin yield. At 90°C, the pectin yield was 27.99%. Increasing the extraction temperature to 100°C did significantly affect the yield. It was reported that 90°C is the optimum temperature for the extraction of pectin from watermelon by-products and mango peels [27,28]. The results indicated that the extraction time of 90°C gave a maximum pectin yield mainly due to practical considerations, such as reduction of energy consumption, lower risk of thermal degradation and preservation of functional properties. Extraction at high temperatures initiates the hydrolysis of the protopectin, leading to the cleavage of glycosidic bonds between sugars and the cell wall matrix, thereby facilitating the release of pectin into the extraction medium [29]. Figure 1 shows the DRP extracted at different temperatures.

3.2 Moisture content

The moisture content of food products is an important parameter because it is related to microbial growth. As shown in Table 1, the moisture content of DRP did not significantly change regardless of the extraction temperature. Similarly, Hasem et al. [30] obtained a moisture content of 11.53 % from durian rind extracted using hydrochloric acid. According to the International Pectin Producers Association (IPPA), the maximum moisture content for dry pectin should not be more than 12%. Thus, the moisture content of the DRP meets the quality standard, indicating good stability and suitability for food and pharmaceutical applications. Reports on pectin from jackfruit and dragon fruit peel also show moisture content ranging from 9-12%, aligning with the present findings and supporting the feasibility of fruit waste as a reliable pectin source [31-32]

3.3 Ash content

Ash content can be used to estimate the material's mineral content and determine pectin's purity. The higher the ash content, the lower the pectin purity [33]. It serves as a key quality parameter of a good-quality pectin at the initial assessment of pectin. In this study, the ash content of pectin extracted from DRP ranged from 4.43% to 4.67%, with no significant differences observed across different extraction temperatures, indicating that the pectin extracted under all three extracted temperatures was of good quality. These values are similar to those reported by Ceylan et al. [34] and Hamed et al. [35] with 5.65% in artichoke waste and 7.1% in pumpkin peel, respectively. In general, the ash content of pectin extracted from the DRP was within the IPPA standard (≤ 10%).

Table 1 Yield and proximate analysis of durian rind pectin and commercial pectin.

Extraction Temperature/ Sources	Parameters			
	Pectin Yield (%) (wb)	Moisture Content (%)	Ash Content (%)	A_w
80°C	23.19±1.02^a	10.38±0.25^a	4.44±0.44^a	0.56±0.01^a
90°C	27.99±0.42^b	10.45±0.18^a	4.43±0.09^a	0.57±0.01^a
100°C	29.65±0.70^b	10.57±0.21^a	4.67±0.16^a	0.56±0.01^a
Commercial Pectin	-	10.90±0.19	2.74±0.16	0.55±0.01
IPPA Standard	-	max. 12%	max. 10%	-

3.4 Water activity

Water activity (A_w) represents the amount of free water that is available to support microbial growth. In this study, the A_w of DRP ranged from 0.56–0.57, indicating that the extracted pectin is microbiologically stable. A low A_w is less prone to microbial growth and, hence, more shelf-stable. Conversely, food with a higher A_w (>0.85) tends to have a shorter shelf-life due to the growth of bacteria, molds, or yeast. In the study of Srikamwang et al. [36], they reported an A_w of 0.56 for mango peel pectin, while Chan et al. [37] obtained Aw around 0.48-0.52 in jackfruit rind pectin. These comparable values suggest that DRP derived pectin obtained a similar microbiological stability to other fruit waste derived pectin and met necessary quality requirements for safe storage and utilization, where low A_w is advantageous for extending product shelf life.

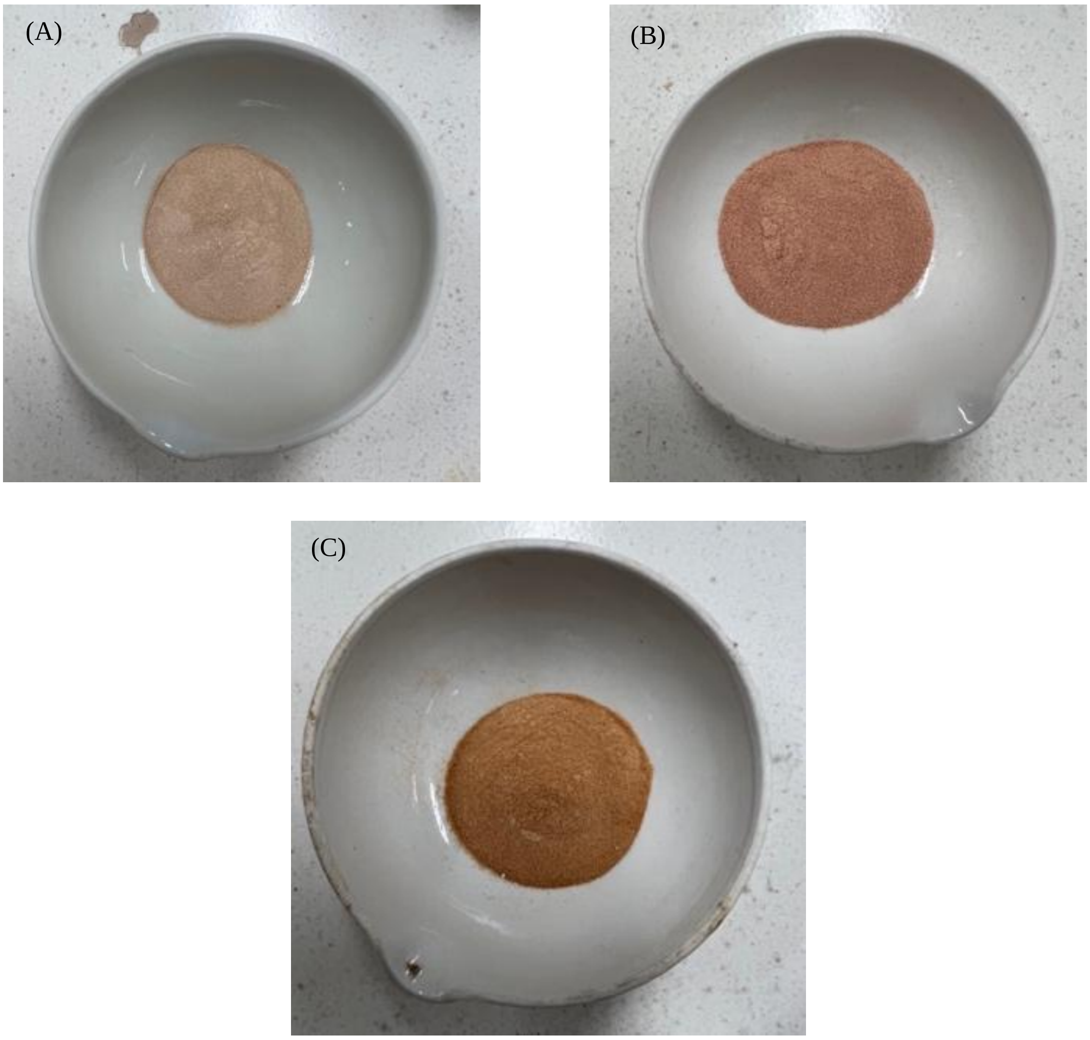

Figure 1 Dried durian rind pectin extracted at different temperatures (A) 80°C, (B) 90°C and (C) 100°C.

3.5 Equivalent weight

The EW of this study ranged from 395.51- 412.39 g/mol. Highest temperature obtained the lowest EW. The extraction temperature significantly affected the equivalent weight of the DRP (Table 2). In comparison to the commercial pectin, the EW obtained in this study is lower, probably due to the source and the extraction method. However, DRP's highest value obtained was 412.39 g/mol at 90°C as the extraction temperature, higher than the minimum value (400 g/mol) recommended by the IPPA. However, at 100°C the EW decreased, likely due to the partial degradation of pectin chains, resulting in reduced molecular weight. These results align with the finding of Dranca et al. [38], who reported that high temperature extraction could degrade pectin polymers, lowering their EW and structural integrity.

3.6 Methoxyl content

The methoxyl content of extracted DRP at varying temperatures is shown in Table 2. The results showed that the higher the extraction temperature, the greater the methoxyl content. Extraction of DRP at 100°C resulted in a methoxyl content greater than 80°C. The increase in methoxyl content is attributed to higher temperatures. Higher temperatures cause a faster protopectin hydrolysis reaction and break the bonds of the pectin galacturonic acid units and other compounds that can subsequently undergo esterification, thereby increasing the methoyl content. This process increases the availability of carboxyl groups, which subsequently react with alcohol to form esters [39]. A similar observation was reported by Obodo-Ovie et al. [40], that higher extraction temperatures enhance the methoxylation of pectin due to increased solubilization and structural modification of protopectin. In general, pectin can be classified into low-methoxyl pectin (<LMP, <7% MC) and high-methoxyl pectin (HMP, >7% MC), which determines its gelling behavior. The methoxyl content obtained in this study are within the acceptable range of HMP, suggesting potential application in traditional sugar-acid gels such as jams and jellies. Similar findings were reported by Setyajati et al. [41], who demonstrated that pectin extracted from tropical fruit wastes exhibited higher methoxyl contents at higher temperatures, making them suitable alternatives to commercial citrus pectin.

Table 2 Characterization of durian rind pectin and commercial pectin.

Extraction Temperature/ Sources	Parameters			
	Equivalent Wt. (g/mol)	Methoxyl Content (%)	Anhydrouronic Acid (%)	Degree of Esterification (%)
80 °C	400.27 ± 1.63^a	8.09 ± 0.06^a	89.93 ± 0.47^a	51.10 ± 0.11^a
90 °C	412.39 ± 5.99^b	8.26 ± 0.05^b	89.56 ± 0.77^a	52.35 ± 0.36^b
100 °C	395.51 ± 3.07^a	8.29 ± 0.03^b	91.90 ± 0.41^b	51.21 ± 0.11^a
Commercial Pectin	948.64 ± 16.96	6.46 ± 0.24	55.20 ± 1.71	66.37 ± 0.45
IPP Standard	min. 400	LMP 2.5-7.12 HMP >7.87	min. 65	-

3.7 Anhydrouronic acid

The anhydrouronic acid (AUA) values indicate the purity of the extracted pectin. The AUA of DRP from this study ranged from 89.56-91.90% (Table 4). The total AUA showed that the different extraction temperatures were significantly different. The significant differences across extraction temperatures suggest that thermal conditions influenced the release and solubilization of galacturonic acid units from protopectin. The highest AUA value was obtained at 100°C. The AUA values are higher compared to commercial pectin. The value was observed to be >65% regardless of the temperature used. Akhter et al. [42] reported AUA values of 66.09-67.19% for pectin extracted from citrus peels, while higher AUA (>82%) was obtained from non-traditional sources such as watermelon rind [43], making them suitable alternatives to conventional sources. A minimum AUA content of 65% is recommended by the Food and Agriculture Organization of the United Nations (FAO), Food Chemical Codex (FCC), and European Union (EU) for pectin intended for use as food additives or in pharmaceutical use [44].

3.8 Degree of esterification

The DE of pectin plays a critical role in determining its gelling mechanism and industrial application. In this study, the DE of the extracted pectin ranged from 51.10% to 52.35%, which classify them as high-methoxyl pectin. The DE extracted from DRP was found to be significantly different among the three extraction temperatures. The extraction conditions influenced DE values of durian rind pectin produced in this study. The highest DE was obtained at an extraction pH of 2.0, heating time of 60 min, and temperature of 90°C. These conditions influence the esterification status of galacturonic acid unite. A comparable result was reported by Seixas et al. [45], which obtained DE values between 50-64% in pectin extracted from passion fruit peel. Thermal treatment during extraction affects the pectin degrees of esterification. Differences in cell wall structure and protopectin composition across plants tissues directly affect the DE, which explains the variations between DRP and pectin extracted from other fruit by-products.

3.9 FTIR analysis

The FTIR spectra of the DRP extracted at 90°C is shown in Figure 2. The absorption band at 3493.48 cm^{-1} corresponds to the O-H stretching due to intra-and-intermolecular hydrogen bonding of the galacturonic acid polymer in pectin samples [46]. The bands around 3288.57 and 2957.56 cm^{-1} correspond to C-H stretching and bending vibrations [47].

The strong band at around 1712.35 cm^{-1} is attributed to the C=O stretching vibration of free carboxyl groups present in both the methyl esterified group ($-COOCH_3$) and the carboxylic acid group (-COOH) [48]. The FTIR spectra of the commercial pectin sample had characteristic peaks at 3287, 2940, and 1753 cm^{-1}, which correspond to –OH, -CH, and C=O of ester and acid, respectively.

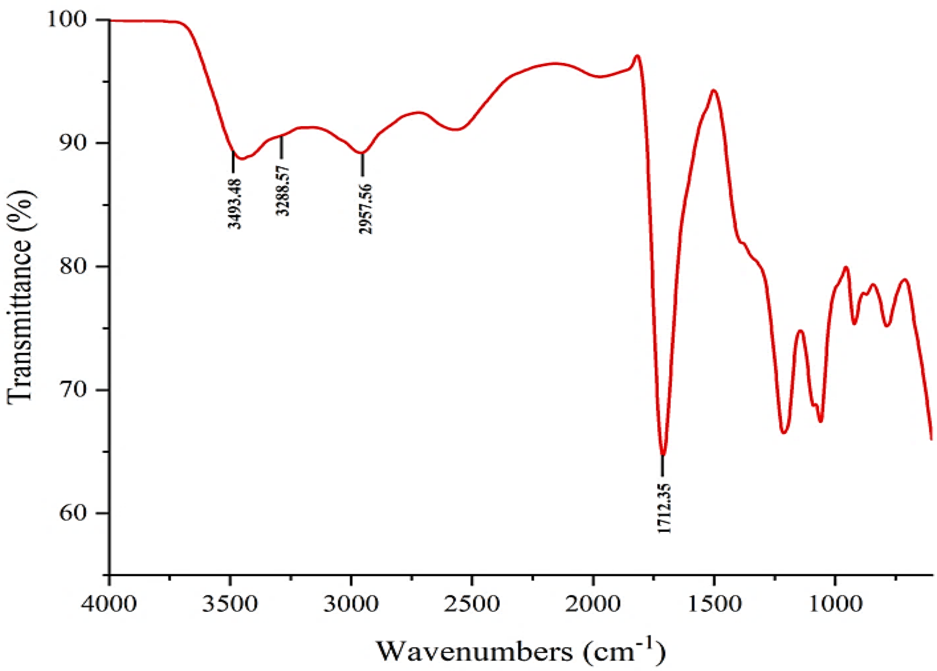

Figure 2 FTIR spectra of durian rind pectin.

4. Conclusions

The results of this study not only demonstrate the feasibility of extracting pectin from durian rind using citric acid but also contribute to the growing interest in valorizing agricultural waste into high-value food ingredients. In this study, the optimum temperature for extraction was 90°C and the obtained pectin had an anhydrouronic content of 89.56%, It was classified as high-methoxyl pectin (8.26%), degree of esterification of 52.35%, and 412.39 g/mol equivalent weight. The extracted pectin had a desirable physicochemical property, including low moisture, ash content, and water activity. FTIR analysis confirmed its similarity to commercial pectin. The method is an environmentally conscious and economically viable alternative to conventional pectin sources. These results underscore the potential of durian rind, an abundant and often discarded by-product that can serve as a functional ingredient in food systems, thereby supporting circular economic initiatives and promoting sustainable food processing innovations.

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6. Conflicts of interest

The authors declare that there is no conflict of interest.

7. Author contributions

Bitoon, JI: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing; Fundador, NG: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing; Alviola, JN: Conceptualization, Methodology, Resources, Validation, Writing – review & editing; Lopez, R: Conceptualization, Methodology, Writing – review & editing

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