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Pectin extraction from cocoa husk of different genotypes and stages of ripeness

Extracción de pectina a partir de cáscara de cacao de diferentes genotipos y estados de maduración

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ABSTRACT

This study evaluates the extraction and physicochemical characterization of pectin obtained from cocoa pod husks (CPH) of two genotypes (CCN51 and IMC-67) at different maturity stages. Multiple extraction variables were optimized to maximize yield and gel quality, including acidulated water concentration, pH, temperature, and exposure time. Results showed that genotype and ripeness significantly affect extraction efficiency and pectin functionality. The highest pectin yield was 77.7% for IMC-67 (ripe) and 72.6% for CCN51 (semi-ripe) at optimal conditions (pH 1.5–1.6, 80 °C, 75 minutes). Physicochemical analysis revealed high galacturonic acid content (up to 82.3%), esterification degree above 50%, and excellent gel consistency, classifying the pectin as high-methoxyl. These findings support the sustainable valorization of cocoa agro-industrial by-products for potential food, pharmaceutical, and packaging applications.

Keywords: circular bioeconomy; biopolymer characterization; high-methoxyl compounds; green extraction technologies; genotypic variability

RESUMEN

Este estudio evalúa la extracción y caracterización fisicoquímica de pectina obtenida de cáscara de vaina de cacao (CPH) de dos genotipos (CCN51 e IMC-67) en diferentes estados de madurez. Se optimizaron múltiples variables de extracción para maximizar el rendimiento y la calidad del gel, incluida la concentración de agua acidulada, el pH, la temperatura y el tiempo de exposición. Los resultados mostraron que el genotipo y la madurez afectan significativamente la eficiencia de extracción y la funcionalidad de la pectina. El mayor rendimiento de pectina fue del 77,7% para el IMC-67 (maduro) y del 72,6% para el CCN51 (semimaduro) en condiciones óptimas (pH 1,5-1,6, 80 °C, 75 minutos). El análisis fisicoquímico reveló un alto contenido de ácido galacturónico (hasta 82,3%), un grado de esterificación superior al 50% y una excelente consistencia en gel, clasificando la pectina como de alto metoxilo. Estos hallazgos respaldan la valorización sostenible de los subproductos agroindustriales del cacao para posibles aplicaciones alimentarias, farmacéuticas y de envasado.

Palabras clave: bioeconomía circular; caracterización de biopolímeros; compuestos de alto metoxilo; tecnologías de extracción verde; variabilidad genotípica

1. INTRODUCTION

Cacao cultivation is a key economic activity in Latin America, contributing to the livelihoods of millions. In 2023, global production exceeded 5.6 million tons, with Ecuador, Brazil, and Peru being notable contributors of 15% (García et al., 2024; FAOSTAT, 2025).

The value of the production in 2023 was US$8,176 million, with an average price of US $1460/ton (FAOSTAT, 2025), which in February 2025 was US$9,783/ton (ICCO, 2025). This highlights the significant global impact of the cocoa agro-industrial sector.

Despite being economically important, cocoa production leads to significant waste. This research examines explicitly cacao pod husks, which are generated following the removal of cocoa beans from fully ripened cacao fruits and constitute the main byproduct of the cocoa and chocolate sectors.

With this considerable cocoa production, disposing of cacao pod husks (CPH) may present serious environmental issues. During cocoa processing, a significant portion of the fruit, about 67% to 76%, ends up as a by-product. About 48 million tons of waste are generated annually (Quiceno Suarez et al., 2024; Campos-Vega et al., 2018). Typically, cocoa pod husks are wasted and left to decompose, leading to environmental problems such as foul odors and the spread of plant diseases, such as pod or bean rot (Meza-Sepulveda et al., 2024).

An alternative and promising solution is transforming this waste into valuable products for different industries. This minimizes environmental problems and promotes the circular economy. For instance, parts od this waste are rich in fiber and pectin, they could be converted into raw material to make biodegradable packaging (Dewan et al., 2024).

With the continuous growth in cocoa production and processing, millions of tons of husks are generated worldwide. In producing countries such as Peru, using this waste could bring economic and environmental benefits to extract valuable compounds, such as pectin, from what is now considered waste. Pectin an interesting option and is widely used in the food and pharmaceutical industry (Chuenkaek et al., 2024; Le et al., 2024).

Pectin extraction from husk uses various techniques, such as the traditional autoclave method, ultrasound-assisted extraction, and microwave-assisted extraction. Among them, the autoclave method has proven to be the most effective, reaching a yield of 26.22% to obtain low-methoxylated pectin, ideal for specific applications (Pinkaew et al., 2024). Ultrasound is a green and novel extraction method for pectin extraction. The pectin produced from CPH contributes to the valorization of waste, an important pillar in the circular economy. This is how scrap materials are reprocessed into new raw materials (Barrios-Rodríguez et al., 2022).

As mentioned previously, pectin presence was confirmed in CPH with previous similar studies (Barazarte et al., 2008; Pinkaew et al., 2024; Mounya & Chowdary, 2024; Wulandari et al., 2023); however, more descriptive information is required.

We also know that the Yield and quality of pectin extracted depend on the cacao pod's maturity. Intermediate and mature pods have a decrease in pectin content as the cell walls tend to mature and rupture, which provides a greater yield during extraction processes. For example, extraction conditions (temperature and pH) can be optimized to obtain the maximum pectin yield from mature pods (Barazarte et al., 2008; Vriesmann et al., 2012).

On the other hand, pectin has different degrees of esterification and methylation depending on the maturity of the cacao pod. The pod may have a higher degree of esterification, especially at maturity, thus affecting the pectin gelling properties (Arlorio et al., 2001; Pranayasa et al., 2022).

Inherent genetic variations among cacao genotypes can produce a wide range of amounts of pectin. These may influence the biochemical process of pectin synthesis and deposition in the pod husk (Blakemore et al., 1966; Barazarte et al., 2008).

Genotypes play a role in the molecular weight and viscosity of extracted pectin, which is important for its functional properties in food and pharmaceuticals (Yapo & Koffi, 2013; Arlorio et al., 2001).

This study focuses on extracting and characterizing pectin from cacao pod husk of two cacao genotypes on green and mature pods.

2. MATERIALS AND METHODS

Fresh cocoa husks (*Theobroma cacao* L.) from immature and mature spontaneous hybrids utilized in this experiment were obtained from the Experimental Station of ICT in Tarapoto. Small pieces of husk pod were obtained from the selected cacao, washed with distilled water, and dried in an oven (Memmert, UFE 500, Germany) with air circulation at 60 °C for 48 h until 6%-7 %. The samples obtained of CPH were ground by using an electric grinder (Bosh, TSM6A013B, Germany) and passed through a 60-mesh sieve. The moisture content of the cocoa husk powder was determined using a moisture meter (DRAMINSKI TwistGrain pro, SN: 29607, Poland). All samples were placed in tightly sealed glass containers and stored at room temperature for later use.

2.1. Pectin extraction

Different essays were carried out with the proposal to optimize the extraction factors

- Determination of optimal relation CPH: Acidulated water volume

The following relations: 1:6, 1:8, 1:10, 1:12, 1:14, 1:16, 1:18, and 1:20 were tested since the extraction process varies according to the raw material studied (Chuenkaek et al., 2024), water retention capacity of the raw material, amount of organic solvent used for precipitation, etc (Maulani et al., 2024). The acidulated water had a pH of 2, a temperature of 80 °C, and a time of 60 minutes, according to Patel (2017) and Baltazar Flores et al. (2013), with slight modifications. The evaluation of this test was carried out according to the performance through the dry pectin from a 100 ml sample, and through a visual appreciation of the gel consistency formed by moist pectin in precipitation before drying.

- Optimal extraction pH

To find the optimal pH of the solution, which will allow the best solubility of pectin in the solution, the pH tested were: 1; 1.3; 1.4; 1.5; 1.6; 1.7; 1.8; 1.9; 2.0; 2.1; 2.2; 2.3; 2.4; 2.5; 2.6; 2.7; 2.8; 2.9; 3.0, 3.0; 3.2, 3.3; 3.5. The constant variables were the best of the previous test, temperature 80°C and time 60 minutes. The best yield in dry pectin and the consistency of the pectin gel were considered for the optimal ph.

- Optimal extraction temperature

To determine the optimal extraction temperature since temperatures higher or lower than the optimal range influence the pectin yield (Anoraga et al., 2024), we tested 65, 70, 75, 80, 85, 90, and 95 °C. In this essay, we consider the constant Relationship with the raw material: the acidulated water volume and the pH of the solution, found in the previous evidence, and the time of 60 minutes. The evaluation was carried out in a similar way to the previous ones.

- Optimal extraction time

Find the optimal contact time between the raw material and the solution since this will influence the yields obtained. The study times were 30, 40, 50, 60, 70, 80, 90, 100, and 120 minutes. The optimal values found

in the previous tests were taken as constants concerning raw material: acidulated water volume, pH of the solution, and temperature. The evaluation was carried out in a similar way to the previous ones.

2.2. Characterization

- Determination of equivalent weight

The equivalent weight of pectin was determined by acid-base titration, following methodologies described by Ranganna (1986) and Stephen and Phillips (2006). The procedure was to dissolve a dry and purified sample of pectin in distilled water, add a few drops of phenolphthalein as an indicator, and titrate with a standard solution of sodium hydroxide (NaOH 0.1 N) to the endpoint, which was signaled by a permanent pink coloration. Equivalent weight was calculated by dividing the sample mass by the base milliequivalents consumed during neutralization of the carboxylic groups present.

- Determination of the galacturonic acid content, the degree of esterification (DE), and the percentage of methoxyl groups

An acid-base titration procedure was used according to the methodology described by Ranganna (1986) and the Food Chemicals Codex (FCC, 2012). To do this, 5 g of pre-dried pectin was weighed and purified by agitation in 5 mL of hydrochloric acid and 100 mL of 60% ethanol, followed by filtration and washing with an ethanol: HCl solution (85:15 v/v) six times. It was then washed with 60% ethanol to remove residual chlorides, and with absolute ethanol to finish drying at 105°C for 2.5 hours. Once cooled in a desiccator, it was weighed again and exactly one-tenth of the net weight (0.5 g) was taken for the assessment.
The sample was dissolved in 2 mL of ethanol and 100 mL of distilled water in a 250 mL volumetric flask. Phenolphthalein was added as an indicator and titrated with 0.1 N NaOH until a persistent pink turn was reached, recording the volume consumed as V1, corresponding to the free acid groups. Next, 20 mL of 0.5 N NaOH was added to saponify the methoxyl groups, shaking and letting stand for 15 minutes. Then, 20 mL of 0.5 N HCl was added to neutralize the excess base. A second titration with 0.1 N NaOH was performed, noting the required volume as V2, corresponding to the acid groups released by saponification.
With the values of V1 and V2, the galacturonic acid content was calculated using the equation:

$$\% \text{ Galacturonic Acid} = [(V1+V2)\times N\times 194.14\times 100)]/ (1000\times W)$$

Where:

- V1: Volume (mL) of 0.1 N NaOH used in the first titration (neutralizing free carboxyl groups).
- V2: Volume (mL) of 0.1 N NaOH used in the second titration (after saponification of methyl ester groups).
- N: Normality of the sodium hydroxide solution (typically 0.1 N).
- 194.14: Molecular weight of D-galacturonic acid (g/mol).
- W: Weight (g) of the pectin sample used for titration (e.g., 0.5 g if using 1/10 of 5 g).
- 1000: Conversion factor from mL to L.
- 100: To express the result as a percentage.

Likewise, the degree of esterification (DE) was estimated using the ratio:

$$\% \text{ DE} = (V2 / V1+V2) \times 100$$

Finally, the percentage of methoxyl groups was determined with the following formula:

$$\% \text{ Metoxilo} = (V2\times N\times 31\times 100) / (1000\times W)$$

Were:

31: Molecular weight of metoxilo group (g/mol)

This parameter is necessary in order to determine if pectin is high (SD ≥ 50%) or low methoxylation (SD=< 50%), which directly affects its gelling properties as well as technological uses (Ranganna, 1986; Food Chemicals Codex - (FCC, 2012)).

- Acetyl Assessment

Pectin acetyl group content ($-COCH_3$) was quantified via alkaline saponification, and subsequent acid-base titration according to the described protocol (Levigne et al., 2002). Methoxyl groups were determined using the same prepared pectin solution. A 20 mL portion of 0.5 N NaOH was added, stirred, and left to stand for 30 minutes to hydrolyze the acetylated groups. The excess base was then neutralized with 20 mL of 0.5 N HCl. Then, the acetic acid originating from the mate acetyl group degradation was titrated with 0.1 N NaOH, and the volume consumed was recorded as V3. The percentage of acetyl was calculated using the following equation:

$$\% \text{ Acetyl} = (V3 \times N \times 4.3 \times 100) / 1000 \times W$$

V3 is the volume (mL) of NaOH 0.1 N used, N is the normality of the base, W is the weight (g) of the pectin sample, and 4.3 corresponds to the molecular weight of the acetyl group. This structural parameter is key to determining the functional behavior of pectin, as a high acetyl content can reduce the ability to form gels, especially in low-methoxyl pectins.

3. RESULTS AND DISCUSSION

In this study, the extraction of pectin from the husks of two cocoa genotypes, CCN51 and IMC-67, was analyzed, considering two stages of maturity and different extraction conditions. The tables presented (Tables 1 to 6) detail the pectin extraction yield (%) and gel consistency based on variables such as acidulated water concentration, pH, temperature, and exposure time. These factors determine both the amount of pectin extracted and its functional properties.

- Pectin extraction yield

The data show that pectin extraction yield varies between genotypes and maturity states. In general, the IMC-67 genotype showed a higher yield than CCN51 under the same concentration of acidulated water and maturity conditions. For example, at a 12 (1:12) concentration, IMC-67 achieved 50.4% yield in the mature state, compared to 46.1% for CCN51 (Table 1).

Table 1. Pectin extraction yield (%) and gel consistency as a function of genotype, maturity stage and acidulated water concentration

Rate		Percentage				Gel Consistency
		CCN51		IMC-67		
Raw material	Acidulated Water	Semi-ripe	Ripe	Semi-ripe	Ripe	
1	6	16.5	13.7	18.3	17.6	Bad
1	8	24.0	21.9	28.6	25.1	Regular
1	10	35.2	32.1	42.6	38.7	Good
1	12	46.1	44.8	50.4	48.7	Very good
1	14	32.5	30.6	46.3	42.1	Very good
1	16	22.6	26.8	42.1	35.8	Good
1	18	16.8	19.3	29.7	24.5	Regular
1	20	12.9	15.6	20.5	16.3	Regular

This result aligns with research suggesting genetic differences affecting pectin production in different cacao cultivars (Sarah et al., 2022; Wulandari et al., 2023).

The variation in pectin extraction yield can be attributed to the inherent chemical composition of cocoa husk, which contains different concentrations of soluble polysaccharides. Pectin extraction from cocoa husk has shown pectin yields ranging from 11% to 15% on a dry basis, indicating that the yield obtained in this study aligns with previous standards (Wulandari et al., 2023).

- Effect of pH and Temperature on Pectin

Yield was also influenced by pH and extraction temperature. An optimal pH was found between 1.5 and 1.6, where yields of up to 49.1% were recorded for CCN51 (Table 2). These observations support studies showing that acidic environments increase the solubility of pectin, favoring its extraction (Pasandide et al., 2018).

Table 2. Pectin extraction yield (%) and gel consistency as a function of genotype, maturity state, and pH

pH	Percentage				Gel Consistency
	CCN51		CCN51		
	Semi-ripe	Ripe	Semi-ripe	Ripe	
1.0	25.1	12.6	27.3	20.2	Bad
1.2	27.1	15.2	32.3	22.1	Bad
1.2	30.1	17.1	36.2	28.5	Regular
1.3	33.6	21.6	38.2	34.6	Regular
1.4	35.2	30.3	44.3	36.1	Good
1.5	37.7	33.9	52.4	38.2	Good
1.6	49.1	37.4	48.3	40.6	Very good
1.7	42.5	40.2	45.4	45.0	Very good
1.8	36.3	35.2	43.2	36.5	Very good
1.9	33.1	30.2	40.1	31.4	Good
2.0	27.6	23.9	36.7	30.5	Good
2.1	24.2	16.4	33.3	29.7	Good
2.2	18.8	12.6	30.1	27.4	Good
2.3	15.3	9.3	27.5	25.4	Good
2.4	11.1	5.1	24.5	23.4	Regular
2.5	9.2	4.2	21.3	20.6	Regular
2.6	7.7	2.8	18.3	17.5	Regular
2.7	6.2	2.5	15.4	16.3	Bad
2.8	5.2	2.3	12.5	13.5	Bad
2.9	4.3	1.9	10.5	10.2	Bad
3.0	3.5	1.3	8.5	9.3	Bad
3.1	3.1	1.1	5.7	7.5	Bad
3.3	3.0	1.0	5.0	6.3	Bad
3.5	2.2	0.9	3.6	4.8	Bad

On the other hand, the extraction temperature showed a positive impact on pectin yield (Table 3). At elevated temperatures of 80 °C, yields of up to 64% were obtained for CCN51 and IMC-67. This is consistent with the literature indicating that increasing temperature may facilitate the breakdown of cell structure and the release of pectin (Nopiani et al., 2024; Wulandari et al., 2023). However, care should be taken with excessive temperatures that could lead to pectin degradation.

Table 3. Pectin extraction yield (%) and gel consistency as a function of genotype, maturity stage, and exposure temperature

Temperature °C	Percentage				Gel Consistency
	CCN51		IMC-67		
	Semi-ripe	Ripe	Semi-ripe	Ripe	
30	18	45	24	16	Bad

40	26	51	29	25	Regular
50	32	57	35	31	Good
60	37	61	38	35	Good
70	45	63	44	44	Very good
75	53	68	49	48	Very good
80	64	70	52	50	Very good
85	52	74	59	53	Good
90	42	52	50	47	Good
100	36	45	30	40	Regular

- Time of exposition in the pectin extraction

Table 4 shows the extraction yield results of two genotypes performed in this study (CCN51 and IMC-67). Such dramatic increases are particularly marked for pectin yield with exposure time, reaching a maximum yield of 72.55% at exposure times of 75 minutes for semi-ripened fruit for CCN51, while IMC-67 achieved a maximum yield of 77.7% for fully ripened fruit at the maximum exposure period.

Table 4. Pectin extraction yield (%) and gel consistency as a function of genotype, maturity stage and exposure time

Time (minutes)	Porcentage				Gel Consistency
	CCN51		IMC-67		
	Semi-ripe	Ripe	Semi-ripe	Ripe	
40	35.8	43.9	21.6	38.2	Regular
50	45.1	50.1	32.4	39.4	Good
60	47.7	55.0	39.6	44.7	Good
70	62.4	65.5	47.9	50.1	Very good
75	72.6	77.7	54.3	52.3	Very good
80	65.4	70.3	62.5	68.5	Very good
85	55.3	58.5	50.3	56.2	Good
90	46.7	51.7	42.3	46.5	Good
100	40.9	48.5	31.6	37.4	Regular

This positive correlation between extraction time and yield is supported by existing literature, which suggests that prolonged extraction periods generally enhance pectin yield due to the hydrolysis of pectin macromolecules and enhanced liberation from cell walls (Mada et al., 2022; Alcantara et al., 2022).

For instance, Khamsucharit et al. (2018) highlighted that the extraction yield diminishes with increasing fruit maturity. Yet, the semi-ripe fruit of certain varieties surpassed the ripe counterparts in terms of pectin yield, aligning with the data provided in Table 4. Additionally, the optimization of extraction conditions, such as temperature and acidity, has been shown to significantly affect pectin yield (Alcantara et al., 2022; Ardiansyah et al., 2021). Arioui et al. (2017) reinforce the yield variations observed and confirm that the extraction conditions and matrix source significantly influence the physicochemical properties.

The gel consistency of pectin extracted varies as a function of maturity stage and exposure time in both genotypes. The results classify gel consistency as "Regular," "Good," or "Very good," with the latter category reserved for higher yields indicative of superior gel-forming capabilities. For instance, the gel consistency was rated "Very good" for CCN51 at 70 minutes (47.9%) and 75 minutes (54.3%), and similarly for IMC-67 at the exact exposure times (50.1% and 52.3%, respectively). This suggests that longer exposure times before gelling lead to optimal pectin characteristics.

In literature, this claim is supported, and it can be observed that with increased yield, the forming ability of gel increases, which is considered a good indicator (Arioui et al., 2017; Tanaid, 2018), as gel formation is associated with higher methoxyl content, which is essential for gel formation. Furthermore, Liew et al., (2016) reported that the longer pectin was exposed to the extraction process, the better the gel properties,

consistent with the results of Table 4. These properties are critical because they define the practical usage of pectin in food systems as thickeners and stabilizers in jellies and jams.

The difference in pectin yield and gel quality between CCN51 and IMC-67 indicates that genotypic differences are important. According to research by Mamiru & Gonfa (2023), different sources of pectin have various chemical compositions, which influence both yield and quality. Thus, the banana genotype must be chosen in terms of pectin extraction for appropriate recycling in the food industry.

In addition, the ripeness of the fruit also plays an essential role. Higher pectin yield from semi-ripe bananas over ripe bananas was also found to be consistent with previous observations, which report more pectin in unripe and semi-ripe fruits than in ripe fruits due to less degradation of the pectin structure with ripening (Shaibu et al., 2022; Khamsucharit et al., 2018). This trend is crucial for those industries seeking to use pectin in food applications, as they will know that finding both the right maturity and the correct genotype becomes an important factor.

- Yield of pectin extraction A

Pectin extraction from cocoa husks analysis, as shown in Table 5. This synthesis investigates the results on the pectin extraction yields from two genotypes of cocoa, specifically CCN51 and IMC-67, at semi-ripe and ripe stages.

Table 5. Pectin extraction yield obtained from cocoa husk

Variables	Percentage			
	CCN51		IMC-67	
	Semi-ripe	Ripe	Semi-ripe	Ripe
Raw material kg	100	100	100	100
Prepared Raw Material kg	34	33.8	33.6	33.5
Dry pectin kg	2.3	2.2	1.9	1.8
Yield %	2.3	2.2	1.9	1.8

Moisture of the husk CCN51: ripe=79.6%, Semi-ripe= 80.2%
Moisture of the husk IMC-67: ripe=85%, Semi-ripe= 81.2%

The results show that CCN51 has the highest yield in the semi-ripe husk stage, reaching 2.3%, and dropping slightly to 2.2% when fully ripe. On the other hand, IMC-67 shows 1.9% in the semi-ripe stage and 1.8% in the ripe stage. Other studies that have been reported similar results in different types of cocoa husk with extraction yields ranging from 1.29% to 4.69%, probably due to extraction methods and the quality of the raw material (Santos et al., 2021; Mollea & Chiampo, 2019). The study of Adi-Dako et al. (2016) indicated that these yields can vary considerably depending on the extraction method used; for example, extractions with hot water and citric acid produce different percentages of pectin.

The moisture content in the cocoa husk is another key factor that directly influences the yield and quality of the pectin extracted. In the case of the CCN51 genotype, it was found that the moisture is 79.6% in the ripe state and 80.2% in the semi-ripe state. Meanwhile, in the IMC-67 genotype, these levels are 85% and 81.2% for the ripe and semi-ripe state, respectively. These values coincide with what has been observed in the literature, highlighting that the amount of water present in cocoa by-products, such as fruit husks, significantly affects the efficiency of extraction and the quality of pectin (Marsiglia-Lopez et al., 2017; Ramli, 2011). In addition, it has been found that parameters of the extraction process, such as temperature and time, significantly affect both the amount of pectin obtained and its degree of esterification (Hennessey-Ramos et al., 2021; Musita, 2021; Barrios-Rodríguez et al., 2022). On the other hand, research indicates that cocoa husks can contain between 11% and 15% pectin on a dry basis (Sarah et al., 2022). This suggests differences in pectin content attributable to process conditions, feedstock type, and potential pretreatment

of the shells (Barrios-Rodríguez et al., 2022; Pinkaew et al., 2024). Optimizing factors such as temperature and extraction duration can significantly increase process efficiency (Ramli, 2011; Yusof et al., 2016).

Physical-chemical characteristics of pectin

Table 6 shows the physical and chemical properties of pectin extracted from CCN51 cocoa husks in two stages of ripeness (ripe and semi-ripe). Knowing how these properties affect pectin's yield is essential, mainly because of its wide variety of uses in different industries.

Table 6. Physical-chemical characteristics of pectin obtained from cocoa husks (CCN51)

Variable	Ripe	Semi Ripe
Humidity	8.5	8.5
Ashes	2.9	3.1
Calcium content	1.4	1.4
Iron Content	0.12	0.1
Magnesium Content	0.05	0.05
Equivalent weight (g)	575	617
Galacturonic acid content	80.2	82.3
Percentage of esterification	50.8	52.6
Percentage of methoxyls	7.96	9.28
Acetyl Assessment	0.7	0.6
Degree of gelling	100	105
Establishment Time (min)	75	75
Establishment Temperature (°C)	70	70
Fineness module	1.3	1.3

Both pectin samples had an 8.5% moisture content. This parameter is pertinent to determining storage stability since high moisture content can compromise gelling ability (Santos et al., 2021).

Ash content, an index of inorganic residues, was slightly higher in pectin from semi-ripe husks (3.1%) than in ripe husks (2.9%). This implies that the mineral content differs with the cocoa husk's maturity stage. Equally, metal analysis indicated that while calcium remained constant at 1.4 g, iron varied slightly, 0.12 g in pectin from ripe husks versus 0.1 g in that from semi-ripe husks, the two metals being relevant to successful pectin gelation (Adi-Dako et al., 2016).

Pectin in semi-ripe husks has higher galacturonic acid (82.3%) than in ripe husks (80.2%). Since galacturonic acid helps form the gel, the increased amount may make the gels more firm. The extent of esterification was also marginally higher in the semi-ripe sample (52.6%) than in the ripe sample (50.8%), further enhancing its gelling potential.

This yield is also reflected in the gelation scores, where pectin from semi-ripe husks gives 105 as opposed to 100 for that from ripe husks. This increase, albeit small, is particularly valued for the food and pharmaceutical industries, where product texture and consistency are paramount (Marsiglia-Lopez et al., 2017). Notably, the extraction process, 75 minutes at 70 °C, yielded replicable results in conformity with previous work (Musita, 2021).

In summary, pectin's physicochemical characteristics are critical for its effective use in various applications. The literature suggests that source material and extraction significantly impact these properties. For instance, cocoa husk pectin has been extensively explored as a pharmaceutical excipient and in controlled drug delivery systems due to its favorable properties (Belkheiri et al., 2021; Adi-Dako et al., 2018).

- Gel Consistency and Physico-Chemical Properties

The gel consistency is a pretty good indicator of pectin yield. By increasing the concentrations of acidulated water and the exposure time, we noticed that the gel quality could be improved from a rating of "bud" to "very good" (Tables 1, 3 and 4). Such a result agrees with previous studies underlining the necessity of an equilibrium among the methoxyl groups and galacturonic acid chains to develop stable pectin gels (Li et al., 2024; Athawale Gauri et al., 2022; Wulandari et al., 2023). To sum up, differences in the chemical structure of pectin are key for gelling and thus crucial for high-quality food products.

Moreover, the pectin obtained in this study contained 20% moisture. The moisture content of this is low, which is beneficial for preserving microbial stability and increasing the material's flow properties (Owusu et al., 2021). These features are important for its applied use as a gelling agent or stabilizer in the food sector.

CONCLUSIONS

The findings of this study show that cocoa husks, particularly from the CCN51 and IMC-67 genotypes, are a noteworthy and feasible source of pectin. Relevant factors, including the genotype, the maturity stage of the fruit, and the extraction conditions, including the concentration of acidulated water, pH, temperature, and exposure time, influence the extraction yield and the quality of the obtained gel. Notably, the IMC-67 genotype produced more when extraction was carried out under acidic conditions (pH 1.5–1.6) and at elevated temperatures (up to 80 °C). In contrast, for the CCN51 genotype, it was better to increase the extraction time, reaching a maximum yield of 72.6% with a very good gel consistency. The ideal conditions for both genotypes indicate that 75–80 °C, pH 1.5–1.6, and 75 min of extraction favor highly eld high-gelling pectin. Furthermore, semi-ripe fruits provided more pectin and better functional characteristics than ripe fruits, which aligns with the literature's note of less structural degradation of pectin in earlier maturity stages. The physicochemical analysis confirmed high galacturonic acid content (up to 82.3%), esterification >50%, and high methoxyl levels, qualifying the extracted pectin as high methoxyl, suitable for applications in food, pharmaceuticals, and other industrial products. These findings support the sustainable valorization of cocoa agro-industrial by-products and contribute to the circular economy. Future studies may focus on industrial-scale optimization and functional validation in food and pharmaceutical systems.

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CONFLICT OF INTEREST

No existe ningún tipo de conflicto de interés relacionado con la materia del trabajo.

AUTHORSHIP CONTRIBUTION

Conceptualization: Arévalo-Gardini, E., Arévalo-Hernández, C.O., Dapeng Zhang and Virupax Baligar.
Formal analysis: Arévalo-Gardini, E. and Arévalo-Hernández, C.O.
Research: All authors.
Methodology: Arévalo-Gardini, E; Arévalo-Hernández, C.O.

Project management: Arévalo-Gardini, E.
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Validation: Arévalo-Gardini, E.
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