



Optimization of microwave-assisted extraction of pectin from cupuassu (*Theobroma grandiflorum*) pod husk as an alternative for waste valorization of Amazonian fruits

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Abstract

This study aimed to optimize the extraction process of pectin from cupuassu pod husk powder by microwave-assisted extraction method, using response surface methodology (RSM). The effects of three variables: irradiation time, pH and solvent ratio, as well as their interactions, were analyzed to determine the optimal extraction conditions with the best process yield. The results indicated that the optimum conditions were pH 1.8, irradiation time of 20 min and solvent ratio of 20 mL/g, to obtain 10.82% yield. Characterization of the extracted pectin showed that obtained pectin is classified as high methoxyl (HMP), which highlights its applicability in the food industry as a gelling, thickening, emulsifying and stabilizing agent in products that require the presence of high levels of soluble solids, such as sugar, and under acidic pH conditions. Key parameters such as equivalent weight of 741.55 ± 1.73 mg/mol, moisture of $9.8 \pm 0.19\%$ and ash content of $10.19 \pm 0.25\%$, key characteristics to evaluate the properties of pectin, were also determined. Finally, fourier-transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR), thermal analysis (TGA and DSC), and scanning electronic microscopy (SEM) revealed structural characterization, thermal stability and morphology of the obtained pectin.

Highlights

- First report on pectin extraction from *Theobroma grandiflorum* pod husk.
- High methoxyl pectin obtained from *Theobroma grandiflorum* pod husk via microwave-assisted extraction.
- pH and irradiation time significantly influenced the yield of *Theobroma grandiflorum* pectin in microwave-assisted extraction.

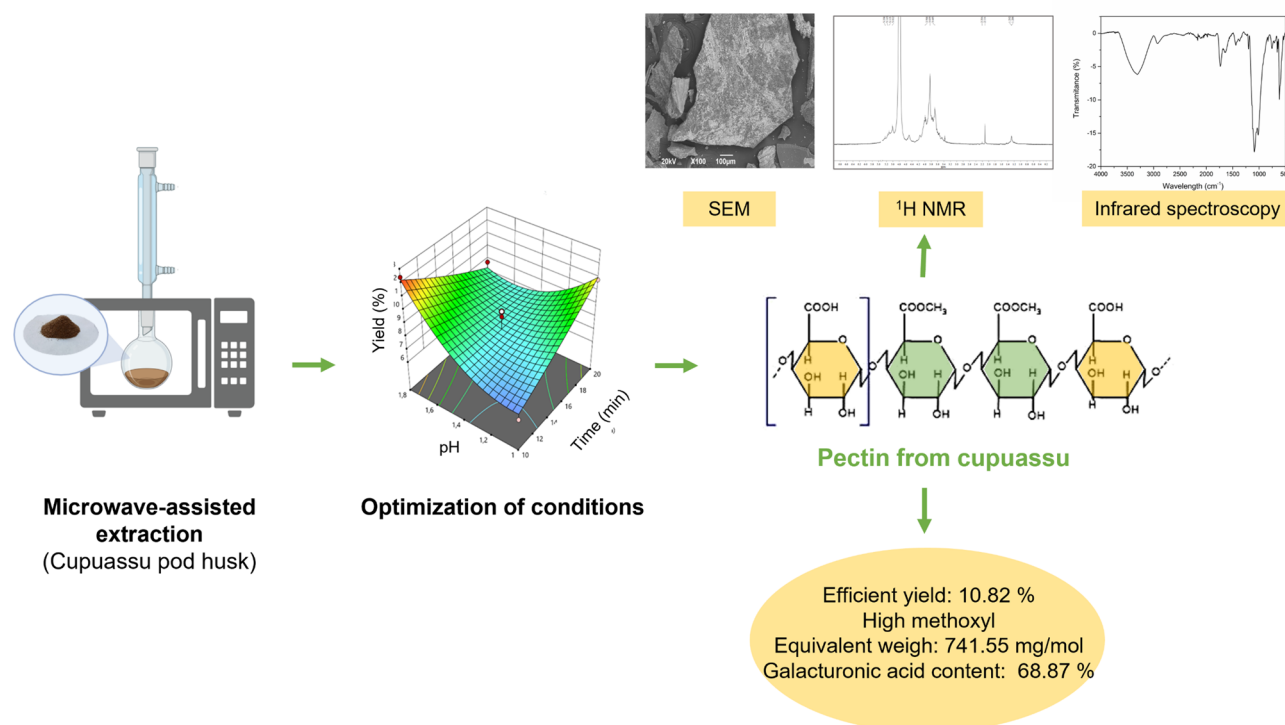
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Graphical Abstract



Keywords FTIR · High methoxyl pectin · Response surface methodology · Thermal stability

Introduction

Pectin, a complex carbohydrate consisting of long chains of sugar molecules, such as galacturonic acid, glucose and rhamnose [1], is a type of soluble fiber found mainly in fruits and vegetables. When heated with sugar and acid, pectin forms a gelatinous substance that gives these products the desired consistency or rheological properties according to the type of food product [2].

In addition to its wide range of uses in the food industry, pectin has been studied for its potential health benefits. It is known to help lower cholesterol levels, improve digestive health and even reduce the risk of certain types of cancer [3–5]. Pectin is also commonly used as a treatment for constipation, as it can help regulate bowel movements [6]. This makes the extraction and marketing of pectin an attractive investment, commercial pectin is extracted from citrus peel and apple pomace [7].

Theobroma grandiflorum, known as cupuassu, is a tropical rainforest tree species native to South America, specifically the Amazon Basin, and belongs to the same family as cocoa; its fruit is similar in appearance to the cocoa pod, but larger and with a thicker rind. The fruit of the cupuassu is highly valued due to its nutritional and medicinal properties.

It is rich in antioxidants, vitamins B1, B2 and C, and minerals such as calcium, phosphorus and iron. The pulp of the fruit is used to make a variety of products, including juices, ice cream, yoghurt, and sweets [8].

According to data from the Food and Agriculture Organization of the United Nations (FAO), the total world production of cupuassu fruit in 2019 was around 13,500 metric tons, with Brazil being the largest producer. Cupuassu fruit typically comprises around 20.0% seeds, 35.0% pulp, and 45.0% pod husk by weight [9]. Pod husks are the most abundant waste and could cause environmental pollution if they are not properly managed. However, they also present an opportunity for the extraction of valuable compounds such as pectins. The valorization of agro-industrial waste for pectin extraction has several advantages. It provides a sustainable solution as it contributes to the management of these wastes, reduces the demand for pectin from traditional sources and generates additional sources of income for agro-industrial companies [10].

Currently, pectin extraction has three main challenges for the implementation of new methodologies: (i) extracting pectin without damaging the side chains, (ii) reducing the environmental impact through less energy use, less toxic solvents and lower amounts of solvents, and (iii) extracting

pectin from a wider range of biomass feedstocks, such as food and agricultural wastes and co-products [11, 12].

Traditional pectin extraction from plant materials is generally carried out through prolonged heating in an acidic medium, with acid hydrolysis being the most employed method in the industry [11, 13]. Nevertheless, this process is associated with several drawbacks, including high energy consumption, long processing times, equipment corrosion, the use of aggressive chemical reagents, and negative environmental impact [11, 13]. To address these issues, several emerging green extraction methods have been developed such as ultrasound-assisted extraction (UAE), supercritical fluid extraction, the use of deep eutectic solvents (DES), and microwave-assisted extraction (MAE) [14].

MAE is a promising technique that employs microwave irradiation to rapidly heat extraction medium (solvent) and biological material, enhancing mass transfer and facilitating the release of intracellular compounds such as pectin [15, 16]. This technique offers advantages over traditional acid hydrolysis, including higher yields, shorter processing time, enhanced pectin quality, and lower solvent quantities [17–19]. Moreover, the rapid heating in MAE minimizes thermal degradation, such as depolymerization, by avoiding prolonged exposure to high temperatures, thereby preserving the structural integrity and functional properties of the extracted pectin [20]. Owing to these strengths, MAE has been effectively used to extract pectin from a variety of vegetables and fruit sources [11] such as pomelo peel [21] apple pomace, black carrot pomace, red and white dragon and passion fruit peels, mango peel, carrot pulp, lemon peel [22], pineapple peel, orange peel, walnut husks, melon peels, banana peel [23], lime peel, cocoa pod husk [24], among others [11]. According to reported studies, the total pectin content and composition vary according to biomass, variety, ripening stage and even within the same source of the same variety. Yields can range from 2.0 to 42.0%, and optimal extraction times vary between 2.5 and 120 min, with applied power typically ranging from 50 to 600 W. However, even for MAE technology, pectin yields usually depend on variables such as time, pH, and the solid/solvent ratio, that have a great influence over the solubility of pectin in the extraction media [19].

Despite its benefits, MAE still faces limitations that hinder industrial application. scaling up this method is particularly complex due to the limited penetration of radiation into the sample, which depends on the relationships between the solvent and the solid and reduces both the speed and uniformity of heating, which in turn affect the yields and quality of the extracted polymer. As a result, MAE extraction becomes more technically demanding and more costly to scale up, because it requires specialized equipment, process optimization, and precise control over operational parameters,

especially when compared to advanced techniques [25]. A few recent reports indicate the potential feasibility of MAE at industrial scale under controlled conditions. For example, Arrutia et al. 2020 [26], carried out the development of a semi-continuous process for the extraction of RG1 (ramnogalacturonan-I)-rich pectin from potato pulp, with the aim of maximizing heating speed and selective heating using MAE, in order to provide homogeneous heating to the extraction matrix. On the other hand [27], pectin was extracted from orange peel in a circulating batch system, obtaining high methoxyl pectin, with yields of 50 g/kg compared to the conventional method with yields of 30.5 g/kg. This was achieved by maintaining a constant temperature through constant modulation of microwave power.

According to the information presented above, MAE can be selectively applied to different raw materials, making it a versatile and effective technique for pectin extraction [11]. Nonetheless, no studies have reported the use of MAE for extracting pectin from cupuassu peel, which limits the potential for valorizing this agro-industrial residue. Given the need to create value-added products from cupuassu waste and the strong potential of MAE to obtain high-quality pectins, this research aimed to optimize experimental conditions, such as pH, extraction time, and solid-to-solvent ratio, using response surface methodology to achieve the highest pectin yield. The extracted pectin was also subjected to structural, physicochemical, and morphological characterization.

Materials and methods

Raw materials and reagents

Cupuassu pod husks (Fig. 1b) used as raw material were supplied by growers from the Balcanes Experimental Farm of the Universidad de la Amazonia, located in Florencia, Caquetá, Colombia. Geographical coordinates are 01° 25' 5.75" N latitude and 75° 30' 9.72" W longitude. The site has an average temperature of 28 °C, relative humidity between 80.0 and 85.0%, and an average annual precipitation of 3600 mm. For the pectin extraction process, husks from 20 physiologically mature fruits harvested in March 2023 were used. The husks were dried in a hot-air oven with forced convection (vacuum oven Jeiotech, model OV-11, South Korea) at 60 °C to avoid carbonization of the material, until a constant weight was achieved. Subsequently, the dried material was ground and sieved through a 40-mesh screen to obtain a homogeneous powder. The resulting powder (with a moisture content of 9.8% on a dry basis) was stored in polyethylene bags at room temperature and kept in a desiccator until use.



Fig. 1 Step-by-step photographic overview of the microwave-assisted extraction procedure for obtaining pectin from cupuassu pod husks: (a) cupuassu fruit, (b) Pod husk, (c) drying stage, (d) milling and sieving, (e) MAE-treated samples, (f) filtration of the extract, (g) pectin product

Table 1 Experimental design for microwave assisted pectin extraction from Cupuassu (*Theobroma grandiflorum*) pod husk with the independent variables and the corresponding experimental yield results

Run	Time-A (min)	pH B	Solvent ratio C (mL/g)	Extraction yield (%)
1	20 (+1)	1.4 (0)	40 (+1)	9.34
2	20 (+1)	1.0 (-1)	30 (0)	10.83
3	15 (0)	1.0 (-1)	20 (-1)	9.59
4	20 (+1)	1.4 (0)	20 (-1)	8.85
5	15 (0)	1.4 (0)	30 (0)	6.63
6	15 (0)	1.4 (0)	30 (0)	7.01
7	15 (0)	1.4 (0)	30 (0)	8.77
8	15 (0)	1.8 (+1)	20 (-1)	12.25
9	15 (0)	1.4 (0)	30 (0)	9.17
10	10 (0)	1.8 (+1)	30 (0)	12.4
11	15 (0)	1.4 (0)	30 (0)	7.48
12	20 (+1)	1.8 (+1)	30 (0)	8.74
13	15 (0)	1.0 (-1)	40 (+1)	10.38
14	10 (-1)	1.0 (-1)	30 (0)	6.1
15	15 (0)	1.8 (+1)	40 (+1)	9.4
16	10 (-1)	1.4 (0)	20 (-1)	8.13
17	10 (-1)	1.4 (0)	40 (+1)	10.05

(+1) indicates the highest value (level) that the variable can take within the experimental region. (-1) indicates the lowest value that the variable can take within the experimental region. (0) value at the center point

All solvents and chemicals used in this study were of analytical grade. Absolute ethanol (99.8%; CAS No:64-17-5) and hydrochloric acid (37.0%; CAS No:7647-01-0) was purchased from PanReac (ITW reagents, Darmstadt, Germany), Sulfuric acid (95.0–97.0%; CAS No:7664-93-9) was purchased from Scharlau (Scharlab, Barcelona, Spain) and sodium hydroxide (98.0%; CAS No:1310-72-2) was supplied by Loba Chemie (Loba Chemie Pvt Ltd, Mumbai, India).

Extraction of pectin from dried pod husk using MAE

The MAE of pectin from cupuassu was performed in a conventional, domestic microwave oven (LG, MS1536GIR, South Korea) at a working frequency of 2450 MHz. Approximately 1 g of dry powdered cupuassu pod husk was placed in a beaker and different amounts of distilled water (20–40 mL) and pH values (1–1.8, adjusted with concentrated sulfuric acid) were used. Each solution was exposed to microwave irradiation (e) at 290 W power for a specified irradiation time (10–20 min). Prior optimization data from pectin extraction in *Theobroma cacao* guided the selection of values for each variable in this study [18, 24]. A total of 17 experimental runs were conducted by varying the extraction parameters, as detailed in Table 1. These runs were performed as part of the experimental design (Sect. [Extraction of pectin from dried pod husk using MAE](#)) aimed at determining the optimal extraction conditions.

After each irradiation, mixtures were allowed to cool at room temperature and filtered through a Whatman No 1 filter paper (Fig. 1f). Then, a volume of 1.5 times (respecting the volume of the filtered extract) of 95.0% ethanol was added, in order to precipitate the pectin. Pectin was washed with 95.0% (v/v) ethanol to remove monosaccharides and finally dried at 35 °C, until constant weight (Fig. 1g).

Extraction yield

The extraction yield was calculated as the ratio of weight of dried pectin obtained and the initial weight of cupuassu pod husk, it is expressed as a percentage, as shown in Eq. (1).

$$\text{Extraction yield (\%)} : m_1/m_0 \cdot 100 \quad (1)$$

Where m_1 (g) is the weight of the extracted and dried pectin and m_0 (g) is the weight of dried powdered cupuassu pod husk used for extraction.

Experimental design and statistical analysis

A RSM was developed by applying a Box-Behnken design (BBD) with 17 experimental trials, including 5 replicates at the central point for the optimization of time (A: 10, 15, 20 min), pH (B: 1.0, 1.4, 1.8), and solvent ratio (C: 20, 30, 40 mL/g) employed in the extraction process of pectin from cupuassu pod husk. Extraction yield (%) was measured as the response variable and modeled through a second-order polynomial equation, represented by the corresponding surfaces. Analysis of variance (ANOVA) allowed the evaluation of both significance and lack of fit ($\alpha=0.05$), and confirmation of the model under optimal conditions suggested by the software (97.0% of desirability) was performed [28–31]. All the aspects related to experimental design and its evaluation were carried out with Design Expert 11 software.

Pectin characterization at optimal conditions

Methoxyl content (MC)

The MC determination was carried out by mixing dried pectin (0.5 g) and NaOH (25 mL at 0.25 N). The mixture was stirred and allowed to stand for 30 min at room temperature. Subsequently, HCl (25 mL at 0.25 N) and 3–5 drops of phenolphthalein were added and titrated with NaOH (0.1 N) until the color changed (pH 7.5) and persisted for 30 s. The data obtained were calculated using the Eq. (2):

$$MC (\%) = (V_1 \times 31 \times 0.1) / m \quad (2)$$

Where m: weight (mg) of the pectin sample; 0.1: N of NaOH; V_1 : volume (mL) of NaOH; 31: weight of methoxyl (CH_3O) group [18].

Equivalent weight (EW)

The EW was determined by mixing dry pectin (0.5 g), ethanol (5 mL), distilled water (100 mL), sodium chloride (1 g), and 3–5 drops of phenolphthalein in an Erlenmeyer flask. The mixture was slowly titrated with NaOH (0.1 N) until the color changed (pH around 7.5) and persisted for 30 s. The obtained data were calculated using the Eq. (3):

$$\text{Equivalent Weight (g/mol)} = m / (V_2 \times 0.1) \times 100\% \quad (3)$$

where m: weight (g) of the pectin sample; 0.1: N of NaOH; V_2 : volume (mL) of NaOH [18].

Galacturonic acid content (GAC)

The GAC was calculated using the following Eq. (4):

$$\text{GAC (\%)} = (176 \times 0.1V_2 \times 100) / m + (176 \times 0.1V_1 \times 100) / m \quad (4)$$

where m: weight (g) of the pectin sample; V_1 : volume (mL) of NaOH from the methoxyl content determination; V_2 : volume (mL) of NaOH from the equivalent weight determination; 176 is molecular weight of galacturonate [18].

Degree of esterification by Titration (DE)

The DE was determined following the methodology described in ref [32]. A 1.0% pectin solution was first titrated with 0.1 N NaOH, using 1.0% phenolphthalein as indicator (titration A). To de-esterify the pectin, 20 mL of 0.5 N NaOH was then added, followed by neutralization with 20 mL of 0.5 N HCl. A second titration was subsequently performed using 0.5 N NaOH (titration B). The following Eq. (5) was used to calculate the (DE):

$$DE \% = B / (A + B) \times 100 \quad (5)$$

Where A: volume (mL) of 0.1 N NaOH spent in titration A; B: volume (mL) of 0.1 N NaOH spent in titration B.

Infrared spectrum and degree of esterification of pectin by fourier transform infrared spectrometry

FTIR spectra were recorded at ambient conditions in the range of 500–4000 cm^{-1} using a Nicolet iS50R Thermo-Scientific spectrometer. Each spectrum represents the average of 32 scans collected with a resolution of 4 cm^{-1} . Measurements were performed using an ATR accessory. This parameter was determined according to Eq. (6) and Eq. (7) [33].

$$DE = 124R + 2,2013 \quad (6)$$

$$R = A_{1735} / A_{1735} + A_{1633} \quad (7)$$

$$A = 2 - \log\%T \quad (8)$$

Where A_{1735} and A_{1633} were presented as the absorbance intensities of the bands for methyl-esterified and non-methyl-esterified carboxyl groups at 1740 cm^{-1} and 1630 cm^{-1} of the infrared spectrum, respectively. Absorbances were obtained from the transmittance percentages, using Eq. (8). These mixed peaks represent the ester and carboxyl vibrations [33].

Nuclear magnetic resonance spectroscopy

Nuclear magnetic resonance spectroscopy was performed on the obtained pectin to determine its structural characteristics. The sample was dissolved in D_2O , and the ^1H NMR

spectrum was recorded at 25 °C using a Bruker AVANCE NEO 400 MHz spectrometer.

Scanning electron microscopy

Scanning electron microscopy images were acquired using a JEOL-JSM-6490LV instrument operating at 20 kV to study the surface microstructure of pectin using three magnifications: 100x, 500x, and 1000x. The sample was attached to carbon tape and coated with a thin layer of gold. Analysis was performed under a high vacuum.

Thermal analysis

Differential scanning calorimetry (DSC) and thermogravimetry (TG/DTG) were performed simultaneously using a thermogravimetric analyzer, model STD 650 (TA Instruments, USA). Thermal analyses were carried out with a 10 °C min⁻¹ constant heating rate. The heating was from room temperature until 800 °C in nitrogen (N₂), a reactive gas, with a mass flow of 60 mL min⁻¹. Also, a flow of 40 mL min⁻¹ of N₂ was used as protection gas into the electronic balance; around 3–5 mg of pectin was placed into a Pt crucible for each analysis.

Results and discussion

Microwave assisted extraction of pectin from cupuassu pod husk

A total of 17 experimental runs were conducted to evaluate the influence of extraction time, pH, and solvent-to-solid

ratio on pectin yield. These variables were analyzed through response surface methodology, which enabled the assessment of both individual effects and interactions among factors, as well as the identification of optimal extraction conditions [29].

Table 1 shows the values of experimental yield obtained from the microwave assisted extraction of pectin from cupuassu pod husk, together with their respective experimental conditions. From this point forward, the independent variables are identified as follows: extraction time (A), pH (B), and solvent ratio (C). These designations are used consistently in the following tables and discussion.

Table 2 displays ANOVA results that were used to verify the significance of the model and factors, as well as the lack of fit and the determination coefficient. The analysis enabled fitting the experimental data to a reduced quadratic model (Eq. 9) that was statistically significant ($p=0.044$), indicating that it adequately describes the relationship between the experimental variables and the pectin yield.

$$\begin{aligned} \text{Yield} = & -4.24880 + 1.49525 [A] - 0.715551 [B] \\ & - 1.04875 [AB] - 0.289823 [BC] \\ & + 9.63647 [B^2] + 0.006895 [C^2] \end{aligned} \quad (9)$$

The determination coefficient ($R^2=0.833$) reflects a good fit, suggesting that 83.3% of the variability in pectin yield can be explained by the model. Among the factors, only pH showed a moderately significant effect ($p=0.098$), whereas time and solvent ratio were not statistically significant ($p>0.7$). In contrast, the interaction between time and pH (AB) was highly significant ($p=0.006$), showing a strong dependence between these two factors. Regarding the quadratic factors (Table 2), the factor B² (pH) was significant (0.025), confirming the presence of curvature in the response surface and demonstrating that pH is a relevant factor in the pectin extraction process. It is important to note that the Lack of Fit was not significant ($p=0.503$), suggesting the model fits the data well and that unexplained variability due to experimental error (pure error).

Figure 2 illustrates the response surface plots for the effects of key extraction parameters, pH and extraction time (Fig. 2a), extraction time and solvent ratio (Fig. 2b), and pH and solvent-to-solid ratio (Fig. 2c on pectin yield).

Figure 2a an upward trend is observed with increasing extraction time, particularly at intermediate pH values around 1.8. In contrast, a noticeable depression is seen at the lower end of the pH range (pH 1.0–1.2), indicating that overly acidic conditions negatively affect the yield. The highest point on the response surface suggests that the optimal conditions occur at approximately pH 1.8 and an extraction time of 20 min. This behavior aligns with previous reports suggesting the implementation of moderate

Table 2 Analysis of variance (ANOVA) for the microwave-assisted extraction of pectin from Cupuassu pod husk. Acquired values of the design expert 11 software

Source	SS	DF	MS	<i>p</i> -value	<i>R</i> ²
Model	41.710	9	4.630	0.044	0.833
A - Time	0.145	1	0.145	0.736	
B - pH	4.340	1	4.340	0.098	
C - Solvent Ratio	0.015	1	0.0153	0.913	
AB	17.600	1	17.600	0.006	
AC	0.511	1	0.511	0.533	
BC	3.310	1	3.310	0.139	
A ²	0.162	1	0.162	0.722	
B ²	9.590	1	9.590	0.025	
C ²	4.950	1	4.950	0.081	
Residual	8.350	7	1.190		
Lack of Fit	3.440	3	1.150	0.503	
Pure Error	4.910	4	1.230		
Cor Total	50.060	16			

SS: sum of squares; DF: degrees of freedom; MS: mean square; *R*² determination coefficient

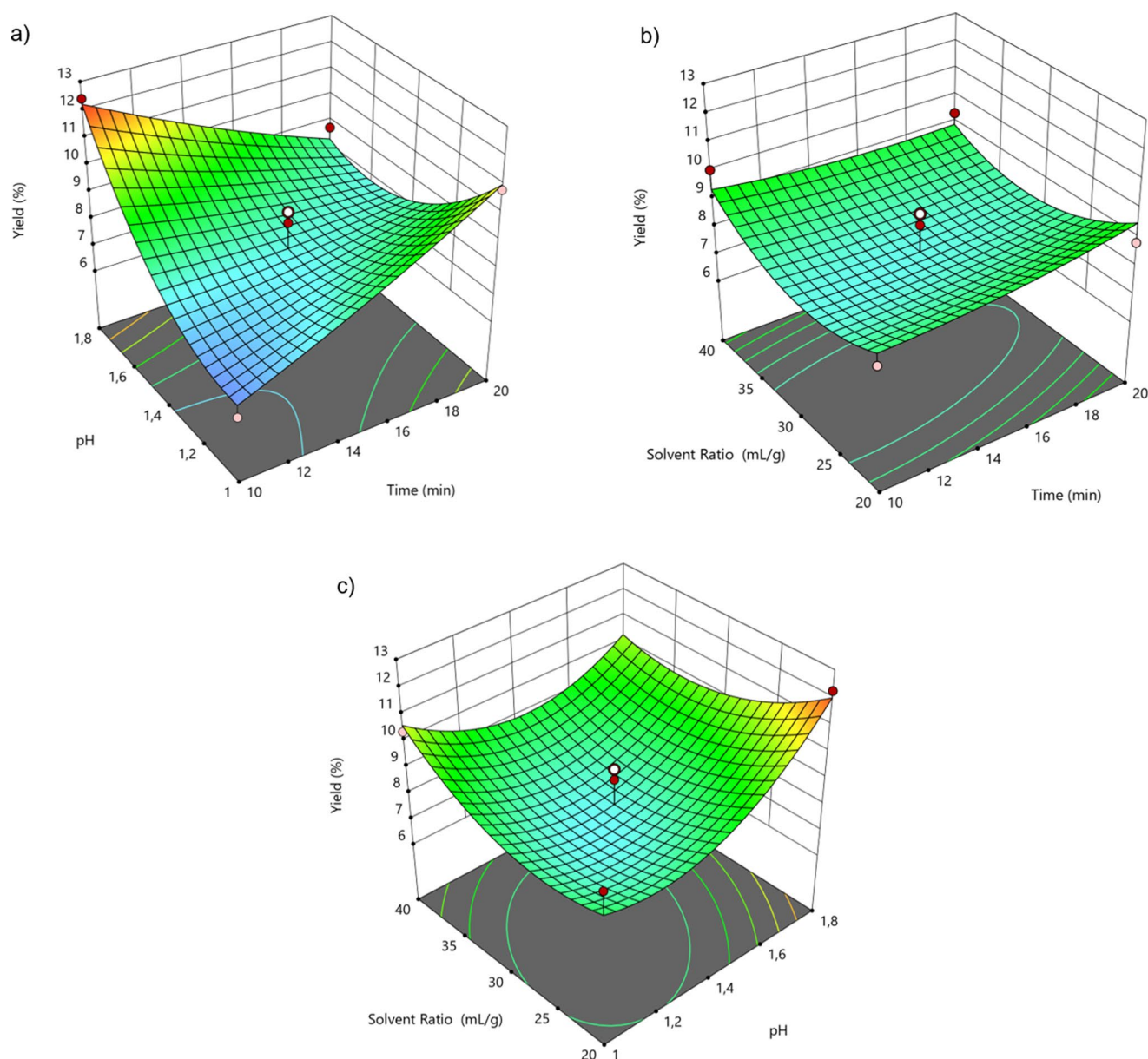


Fig. 2 Response surface plot for the effect on pectin extraction yield (a) Response surface of pH and Time (s) (b) Response surface of time and solvent ratio (mL/g) (c) Response surface for solvent ratio (mL/g) and pH

acidity in the system to improve extraction yield, since at very low pH insoluble pectin is hydrolyzed and becomes soluble [34–38]. Additionally, prolonged extraction times can lead to pectin degradation, which may negatively affect the yield [35, 36].

Figure 2b displays a relatively flat surface, with slight increases in pectin yield as both factors increase, but without any drastic changes. The plot suggests that within the studied range, neither extraction time nor solvent ratio has a strong individual effect on yield. However, their combined influence may contribute modestly to yield improvement, particularly at a solvent ratio of approximately 20 mL/g.

Figure 2c confirms that pH is the most influential variable, as the highest yields are obtained at values close to pH 1.8. In contrast, the solvent ratio exhibits a less pronounced effect on the response surface, reinforcing the role of pH as the primary determining factor. Under these optimal conditions, a maximum predicted pectin yield of 10.60% was obtained.

Based on this analysis, the optimal conditions identified as pH 1.8, 20 min of extraction time, and a solvent to solid ratio of 20 mL per gram were selected for experimental validation for pectin yield of 10.60%, considering that the selected solution had a desirability value of 0.714. Under these parameters, the extraction was performed in triplicate.

An experimental yield of $10.82 \pm 0.54\%$ was achieved, corresponding to a relative error of 2.07%. The experimental value at optimal conditions is very close to the value predicted by the model (10.60%), thus highlighting the efficiency of the methodology used for the optimization of the microwave pectin extraction from cupuassu pod husk. When comparing the present results with those reported for MAE applied to raw materials of the same botanical family, such as *Theobroma cacao* pod husk, our yield is higher. In that case, a yield of 9.64% was obtained using oxalic acid at a pH of 1.16, with 15 min of microwave irradiation at 400 W [24]. Addition, pectin extraction from cacao pod husk by conventional method acid hydrolysis has shown lower yields. For example, a yield of 7.62% was reported when citric acid was employed under conventional heating [39], while ascorbic acid yielded only 4.20% [39, 40].

Physicochemical characterization

Table 3 shows data for physicochemical characterization for cupuassu pod husk pectin extracted under microwave irradiation at optimized conditions.

Cupuassu pod husk pectin exhibited the typical characteristics of a high methoxyl pectin, with a methoxyl content of $22.79 \pm 0.58\%$, and a degree of esterification of $79.08 \pm 2.13\%$ (titration) and 79.97% (FTIR), indicating a high proportion of esterified carboxyl groups. This elevated methoxylation promotes gel formation through hydrogen bonding and hydrophobic interactions between pectin chains. Therefore, this pectin shows a gelling capacity, making it suitable for food applications requiring consistent textures without the need for additional gelling agents [14, 36].

It has been found that microwave-assisted pectin extracted from cocoa husk using citric acid concentration (2.5%, 5%, and 10%) yielded a pectin with a methoxyl content of 6.07% where the acid concentration was not significant in terms of pectin quality and was attributed to variables such as extraction time and power [18]. Alternatively, it was reported that in pectin extracted from cocoa shells assisted by microwaves and evaluating the effect of time and extraction time using citric acid and HCl where the pectin quality

was of a methoxyl content of 4.8% using citric acid the best to obtain better pectin quality. Finally, we can find that the pectin contained in cupuassu pulp has been characterized using the aqueous extraction technique at 25 °C, where it was found to have a 53.0% degree of esterification [41].

Another parameter indicator that supports the gelling behavior of pectin is the equivalent weight, which corresponds to the total content of free (non-esterified) galacturonic acid in the pectin. A higher EW reflects a greater gelling capacity. The presence of free galacturonic acid in the molecular chains contributes to the water-binding properties of pectin [42–44]. In this study, the EW value was 741.54 mg/mol for cupuassu pod husk pectin, which is relatively high and exceeds the values reported for other microwave-assisted extracted pectins, such as cocoa pod husk pectin extracted by microwave assisted, with 602.40 mg/mol [18] and 714.29 mg/mol [45].

Galacturonic acid is considered the backbone of the pectin molecule, and its content can be used as an indicator of pectin purity. The galacturonic acid content (GAC) obtained was $68.87 \pm 0.20\%$, a value close to that reported for pectin from cocoa pod husk ($74.5 \pm 0.3\%$) by sugar acid treatment with ascorbic acid [40]. According to the requirements established by the Food and Agriculture Organization of the United Nations and the European Union, industrial pectin must contain at least 65.0% galacturonic acid [46]. Therefore, the pectin extracted by MAE is suitable for the formation of strong gels in the food industry. It is worth noting that, according to previous reports, both MAE and UAE exhibit superior ability to fully release pectic compounds with higher galacturonic acid content from deeper parts of the plant structure [38].

Moisture content significantly affects the shelf life of pectin; water content increases susceptibility to microbial activity and reduces its functional quality and commercial value [47]. The moisture content of the cupuassu pod husk pectin was $9.8 \pm 0.19\%$, representing a low water level that favors safe storage conditions and extends shelf life [40]. This value is close to that reported for pectin extracted from cocoa pod husks using citric acid-assisted hydrothermal pretreatment (8.46%) [48] and higher than the values obtained with nitric acid and boiling water treatments ($2.7 \pm 0.3\%$ and $0.9 \pm 0.10\%$, respectively) [49, 50]. Overall, the low moisture content observed in this study suggests good storage stability and extended shelf life for the extracted pectin.

The ash content for cupuassu pod husk pectin was $10.19 \pm 0.25\%$, which is comparable to values reported for pectin from *Theobroma cacao* pod husk extracted using MAE. In that work, ash contents of 8.0% (citric acid medium) [18], 1.0–5.7% (itric acid), and 5.0–10.0% (Cl) were reported [51]. The ash content indicates the amount of minerals and organic matter present in the pectin, such

Table 3 Physicochemical characterization for Cupuassu pod husk pectin extracted under microwave irradiation at optimized conditions

Parameter	Value*
Methoxyl content (%)	22.79 ± 0.58
Equivalent weight (mg/mol)	741.55 ± 1.73
Galacturonic anhydrous acid content (%)	68.87 ± 0.20
Moisture (%)	9.8 ± 0.19
Ash (%)	10.19 ± 0.25
Degree of esterification by titration (%)	79.08 ± 2.13
Degree of esterification by FTIR (%)	79.97

*Values are presented as mean $\pm$ standard deviation of 3 replicates

as potassium, magnesium, iron and sodium, and a low ash content indicates a high purity of the pectin [43].

The variations observed when comparing the physico-chemical properties of pectin extracted from cupuassu pod husk with those of cacao species can be attributed to factors such as botanical differences, geographical origin, and extraction conditions, including the method employed [48]. The comparison is primarily made with *Theobroma cacao* due to the lack of information regarding pectin extraction from *Theobroma grandiflorum*, either from the pulp or the pod husk. To date, only one study has reported pectin extraction from cupuassu pulp, using water as a solvent and a multi-step methodology; that pectin showed a degree of methyl esterification of 53.0% and a degree of acetylation of 1.7% [37]. However, this report does not provide sufficient data for further comparison. Despite these variations, the pectin extracted in the present study appears to be a promising candidate for applications in the food industry. Despite these variations, the pectin extracted in the present study appears to be a promising candidate for applications in the food industry [40].

Structural characterization

Infrared spectrum

Figure 3 shows the infrared spectrum of the extracted pectin at optimal conditions. A broad absorption around 3311 cm^{-1} caused by the stretching of the hydroxyl groups is observed, as well as two peaks at approximately 2933 cm^{-1} which represent the asymmetric and symmetric stretching of the C-H bond and which include CH, CH_2 and CH_3 present in the galacturonic ring of the aliphatic carbons. The absorption at

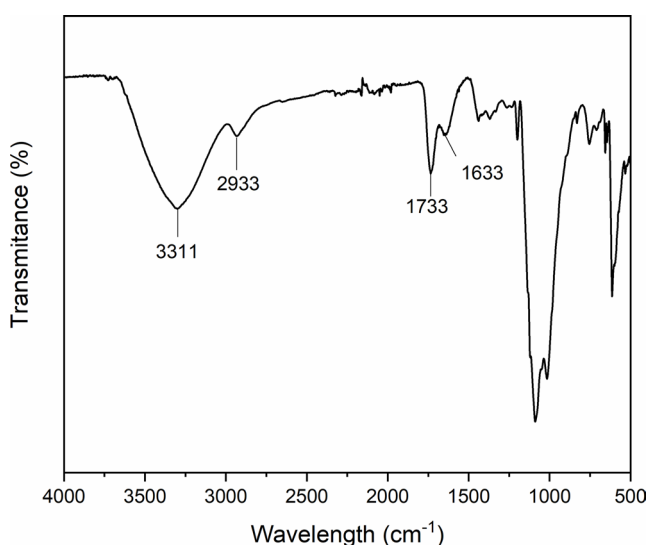


Fig. 3 FTIR spectrum for pectin extracted from cupuassu pod husk under optimized conditions

1733 cm^{-1} was given by the stretching vibration of the C=O bond of the esterified carboxyl group, while the absorption at about 1633 cm^{-1} was attributed to the stretching vibration of the carboxylate ionic bond [21].

^1H NMR analysis

Pectin as a complex native polysaccharide consists of a chain of galacturonic acid units linked by α -1,4 glycosidic bonds forming an α -D-galacturonan. Some of these carboxyl groups are presented as methyl ester, while the remaining ones may be partially or totally neutralized with sodium, potassium or ammonium [52].

Depending on the source of the pectin, galacturonan units exhibit acetylation at 2-O or 3-O position. Pectins contain neutral sugars, mainly L-rhamnose, whose distribution in the chain varies according to the source [53]. In citrus, apple and sunflower pectins, the L-rhamnopyranosyl units can be uniformly integrated into the galacturonan chain via (1 $\rightarrow$ 2) and (1 $\rightarrow$ 4) linkages, forming a rhamnogalacturonan [54].

^1H NMR spectrum (Fig. 4) of cupuassu husk pectin shows signal at δ 5.119 ppm was related to the anomeric proton (H-1) of the galacturonate and methyl galacturonate units [55]. The peak for the H-5 proton was also detected near the area at δ 5.012 ppm [55]. This behavior was reported for pectins isolated from the fruit of *Dillenia indica* and pectin from tomato residues [56, 57]. The spectrum reveals a sharp and broad peak at δ 3.835 ppm, attributed to the protons of methyl groups attached to the carboxyl group ($-\text{OCH}_3$) of the galacturonic acid (GaIA). Furthermore, a sharp peak at δ 2.206 ppm was assigned to protons of acetyl groups ($-\text{COCH}_3$) attached at the 2-O position, while a low intensity peak at δ 2.116 ppm corresponded to the protons of acetyl groups attached at the 3-O position of GaIA in the chain structure of pectin [55, 58]. The chemical shifts observed at δ 3.685, δ 3.988, and δ 4.493 ppm correspond to the protons H-2, H-3 and H-4, respectively [55, 59, 60]. The peaks around 1.288 and 1.292 ppm were related to protons (H-6) of the methyl groups of rhamnose in 2-O and 4-O positions [58]. The spectrum of the extracted pectin agrees well with the peaks typical of pectin structures reported by conventional and ultrasound methods.

Morphology

The morphology of cupuassu pod husk pectin extracted by MAE was studied by SEM. Figure 5a displays that pectin exhibits a morphology similar to irregularly shaped flakes, with varying sizes. Its surface is mostly flat but presents a slight roughness. However, as seen in Fig. 5b, the sample is heterogeneous, as some flakes have a smooth surface. Comparable results have been reported for the extraction

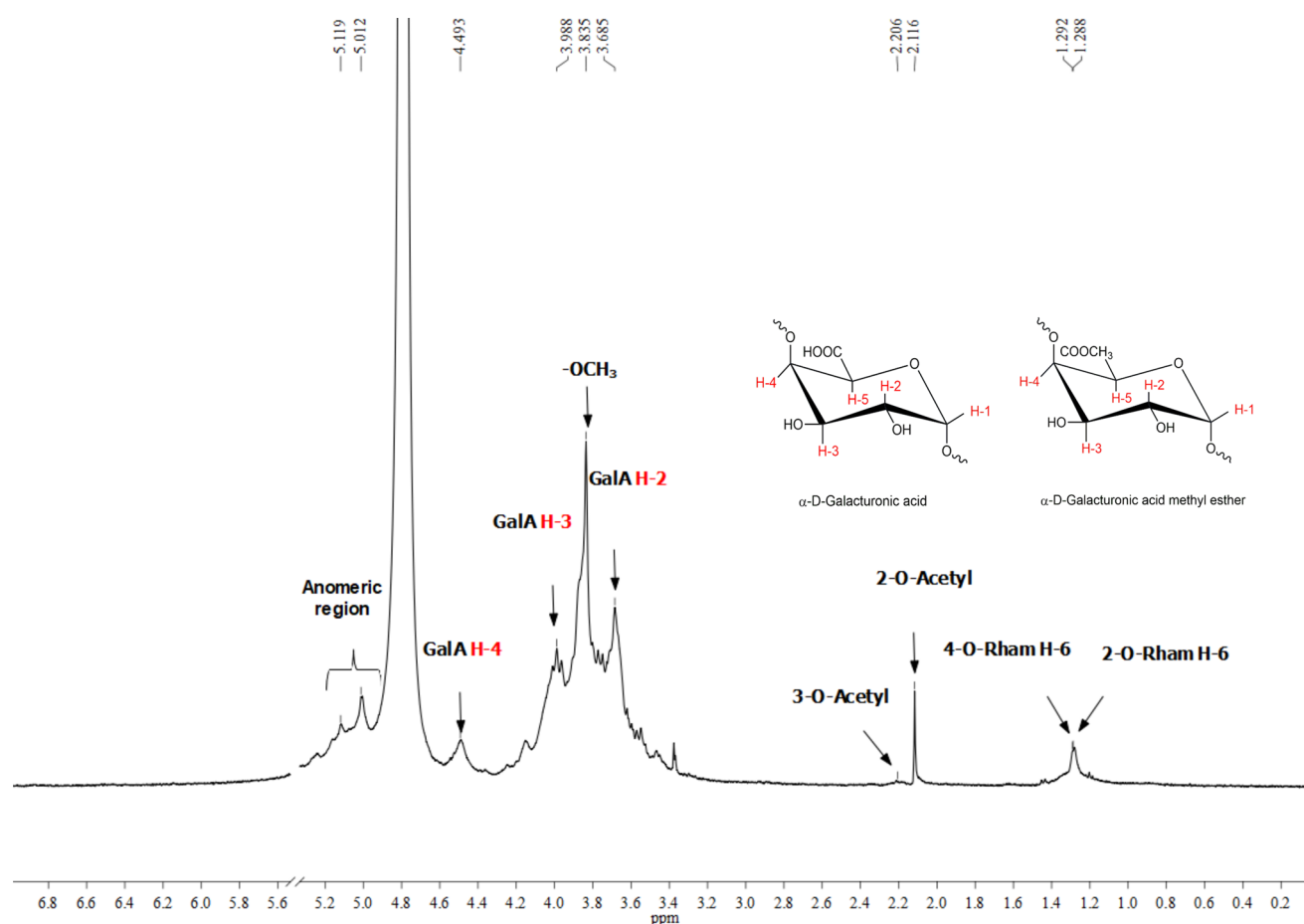


Fig. 4 ^1H NMR spectrum of cupuassu pod husk pectin obtained by microwave-assisted extraction

of grape pomace pectin and *Citrus maxima* Albedo and Flavedo pectin by MAE [55, 61, 62], suggesting that the roughness observed in the pectin morphology could be a consequence of the use of this method, possibly due to the rapid increase in temperature during the process [32, 42].

Figure 5c and d also show microstructures or granules adhering to the surface of the flakes, which could be associated with the presence of sugars, proteins and other macromolecules present in the sample [60].

Thermal analysis

Figure 6 shows TG and DTG thermograms of pectin extracted from cupuassu pod husk. The thermal decomposition proceeded through four distinct stages of mass loss, occurring within the temperature intervals of 50–142 °C (step I), 142–271 °C (step II), 271–388 °C (step III), and 388–763 °C (step IV). The DTG curve presents the maximum degradation temperatures (T_{max}) for each step at 99.83 °C, 249.22 °C, 302.12 °C, and 349.61 °C, respectively.

The first step resulted in an 8.0% mass loss (Fig. 6), which is attributed to the evaporation of both free and

absorbed water present in the sample, reflecting the hygroscopic nature of pectin (as discussed in the previous section) [28, 63]. The second mass loss stage, accounting for 18.0%, reflects the initial pyrolytic degradation of the polysaccharide chain, primarily through the release of weakly bound volatile compounds such as hydroxyl, methoxyl, and carboxyl groups incorporated into the galacturonic acid backbone [28, 63–65]. The third stage represents the most significant mass loss (20.3%), mainly attributed to the breakdown of the main galacturonic acid chain along with the decarboxylation of the acid side chains and carbon in the ring [29, 64, 66]. This temperature range (steps II and III), various gaseous products are generated, including CO, CO₂, and organic volatiles [66, 67]. The thermal resistance observed in these stages may be influenced by factors such as the degree of esterification, molecular weight of the pectin, and the presence of impurities or residual components from the extraction process [68].

The final weight loss of 23.0% (Fig. 6) is likely associated with the degradation of thermally more stable fractions and the oxidative decomposition of carbonaceous residues (char) [28, 29, 68]. Residual mass of 30.7% was obtained,

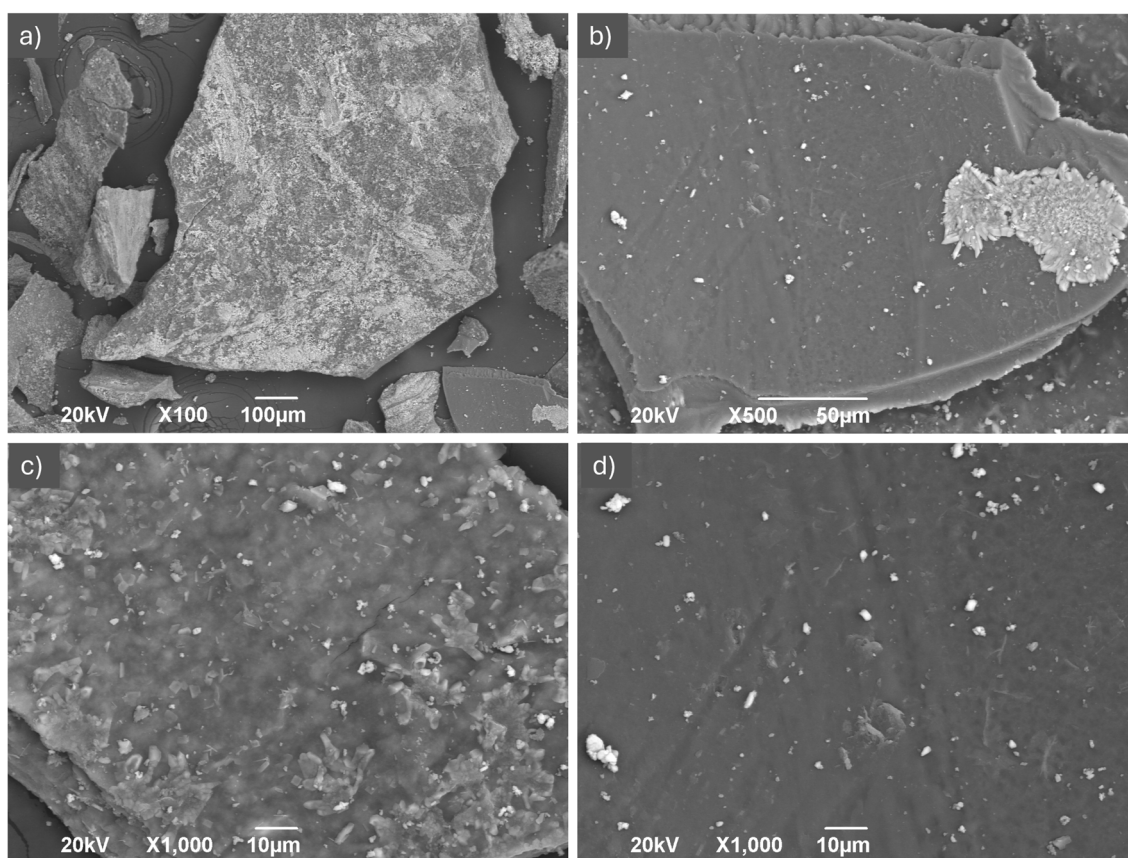


Fig. 5 SEM micrographs of cupuassu pod husk pectin at magnifications of (a) 100x, (b) 500x and (c), (d) 1000x

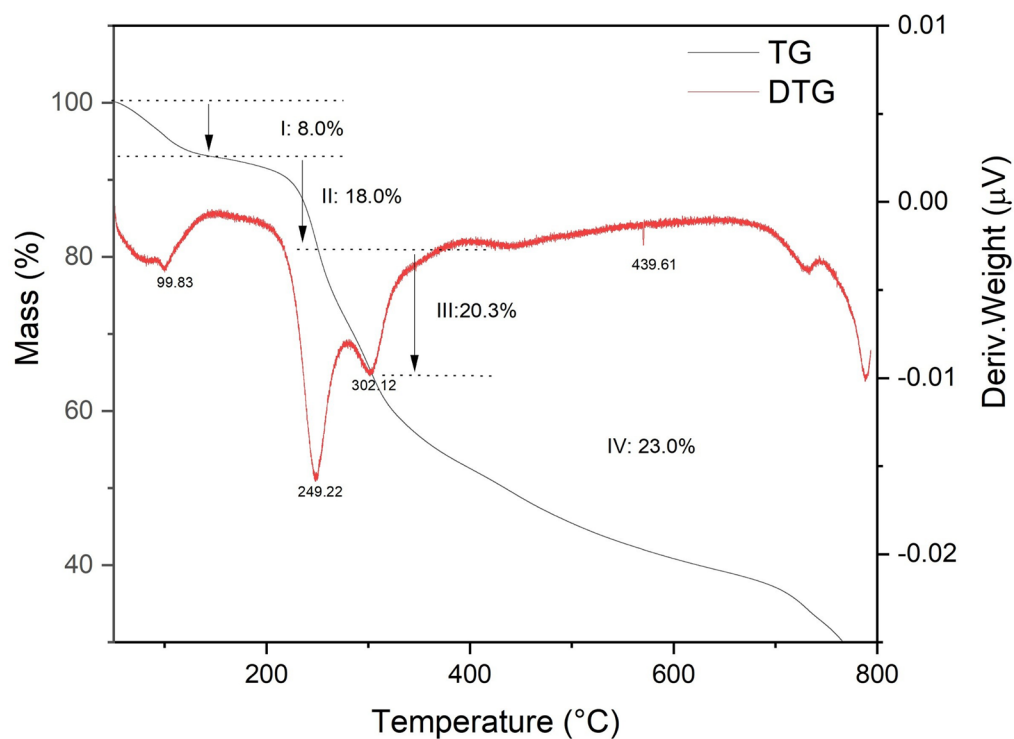


Fig. 6 Thermogravimetric (black) and Derivative Thermogravimetric (red) curves (TG/DTG) for microwave assisted extracted pectin from cupuassu pod husk

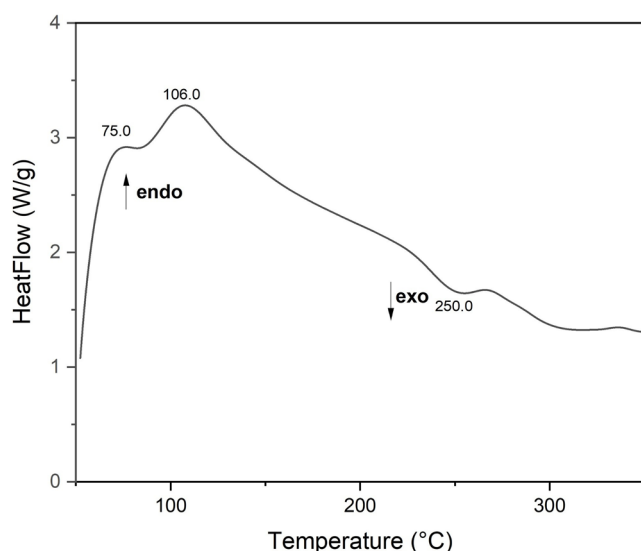


Fig. 7 Differential scanning calorimetry curve of pectin extracted from cupuassu pod husk using microwave-assisted extraction

associated with the formation of non-volatile carbonaceous material. Taken together, these results indicate that the pectin exhibits moderate thermal stability.

The DSC curves (Fig. 7) revealed two endothermic peaks at 75 °C and 106 °C, attributed to the water-holding capacity of polysaccharides and related to the presence of hydrophilic groups in pectin [28, 63, 66, 69]–[72]. The presence of an additional endothermic peak at 106 °C in the pectin extracted from cupuassu pod husk may be attributed to its high degree of methoxylation (22.79%), in contrast to the lower values observed in citrus-derived pectins, such as the commercial sample (7.06%) and that obtained from *Citrus limetta* peels (7.19%). These differences in the degree of methoxylation may promote stronger internal interactions and reduce molecular mobility, which could contribute to the increased thermal stability observed in cupuassu pod husk pectin [63]. Moreover, a weak exothermic peak was observed at 250 °C, which is related to the thermal degradation of cupuassu pod husk pectin structure. This behavior aligns with the chemical characteristics of pectin and corresponds to the major mass loss steps identified in the TG/DTG analysis [28, 63, 66, 69]–[72]. It is worth noting that the thermal behavior of the pectin extracted in this study displays typical characteristics of this polysaccharide and is comparable to that of commercial citrus pectins. In fact, its thermal stability appears to be higher than that of pectins obtained from *Citrus limetta* peels, as indicated by the higher temperatures at which key thermal transitions occur [63, 71, 72].

Conclusions

Microwave-assisted extraction was successfully optimized to extract pectin from cupuassu pod husk, obtaining a product with favorable physicochemical characteristics and high thermal stability. The optimal extraction conditions identified were pH 1.8, 20 min irradiation, and 20 mL/g solvent ratio, resulting in a yield of 10.82% and produced a high methoxyl pectin with an esterification degree of 79.08%. These features highlight its potential as a gelling agent in food products with high levels of soluble solids.

Structural analysis by FTIR corroborated the presence of key functional groups that confirm the purity and functionality of the pectin obtained, while thermal analysis evidenced a high thermal stability, with four well-defined stages of decomposition. The ^1H NMR analysis confirms that cupuassu husk pectin is a galacturonan polymer with α -1,4-linked D-galacturonic acid residues, methyl ester groups, rhamnose units, and acetylated galacturonic acid. This highlights its potential for industrial applications under high temperature conditions. Pectin morphology is predominantly composed of irregular flakes with a slightly rough surface, typical of pectin extracted by MAE. The results obtained position this pectin as a viable and sustainable alternative to traditional sources, promoting the valorization of agroindustrial wastes.

To add value to biowaste such as cupuassu pod husk through the production of pectin, future studies should consider several complementary approaches. First, biological tests such as toxicity assessments should be conducted to evaluate the safety and potential applications of the extracted pectin. Additionally, exploring alternative extraction methods, particularly ultrasound-assisted extraction, could offer further improvements in yield and process efficiency. In both this technique and MAE, it would be worthwhile to investigate the effect of solvent type on both the yield and quality of the pectin obtained. Moreover, it is essential to emphasize that pH control remains a critical parameter and should be carefully monitored in any future extraction strategy. Another relevant aspect to consider is the possible variation in pectin performance and quality depending on the geographical origin of the raw material. Finally, it is important to note that this study represents a first contribution toward reducing the existing knowledge gap on pectin extraction from cupuassu processing residues and provides a basis for future investigations into other influential variables.

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Author contributions Oscar D. Trujillo-Moncada: Original draft. Angie Lorena López Cuéllar: Methodology. Francis S. Sánchez-Garzón: Data Curation, Formal analysis. Oscar Forero-Doria: Formal analysis. Lina M Bolívar-Pineda: Resources, writing - review and editing. Luz Stella Nerio: Conceptualization, Writing - review and editing.

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Data availability All necessary data supporting the findings of this study are available on paper. Any other data required will be made available from the corresponding author upon reasonable request.

Declarations

Ethical approval No applicable.

Competing interests The authors declare no competing interest.

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