Effect of extraction conditions on obtaining pectin from agroindustrial coffee by-products

Efecto de las condiciones de extracción en la obtención de pectina a partir de subproductos agroindustriales del procesamiento del Café

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***Abstract*— Pectin is a product of industrial interest that uses agroindustrial residues to obtain it. To address the reduction of waste generated in coffee agroindustrial production and explore potential applications of by-products, this study investigates the impact of various physical factors (pH, temperature, and reflux time) on the extraction of pectin from discarded coffee husks (*Coffea arabica*) in the San Juan de Rioseco area, following the desiccation process. The extraction process involves hydrolysis in an acidic medium using hydrochloric acid, followed by coagulation with 96% ethanol, filtration, and subsequent drying at 45°C. The quality of the obtained pectin is assessed through infrared spectrophotometry to determine the degree of esterification and extraction yields under different conditions. Wet material yields high methoxyl pectin, with esterification levels ranging from 56% to 75%, while the yield remains below 1%. Analysis of the main components and surface graphs reveals an inverse relationship between temperature and esterification degree, as well as a direct relationship between time and yield. Based on these findings, the optimal extraction conditions are determined to be pH 2.0, a temperature of 90°C for 1 hour. Overall, the results suggest that byproducts from the coffee agroindustrial process hold promise as a source of pectin.**

***Index Terms*— acid hydrolysis, coffee byproducts, degree of esterification, pectin, response surface methodology, solid-liquid extraction, waste management.**

***Resumen*— La pectina es un producto de interés industrial que usa residuos agroindustriales para su obtención. Para abordar la reducción de residuos generados en la producción agroindustrial del café y explorar posibles aplicaciones de subproductos, este estudio investiga el impacto de varios factores físicos (pH, temperatura y tiempo de reflujo) en la extracción de pectina a partir de cáscaras de café desechadas (*Coffea arabica*) en el área de San Juan de Rioseco, después del proceso de desecación. El proceso de extracción implica la hidrólisis en un medio ácido utilizando ácido clorhídrico, seguido de la coagulación con etanol al 96%, filtración y posterior secado a 45°C. La calidad de la pectina obtenida se evalúa mediante espectrofotometría infrarroja para determinar el grado de esterificación y los rendimientos de extracción bajo diferentes condiciones. El material húmedo produce pectina de alto contenido de metoxilo, con niveles de esterificación que oscilan entre el 56% y el 75%, mientras que el rendimiento se mantiene por debajo del 1%. El análisis de los componentes principales y los gráficos de superficie revela una relación inversa entre la temperatura y el grado de esterificación, así como una relación directa entre el tiempo y el rendimiento. Basándose en estos hallazgos, las condiciones óptimas de extracción se determinan como un pH de 2.0, una temperatura de 90°C durante 1 hora. En general, los resultados sugieren que los subproductos del proceso agroindustrial del café tienen potencial como fuente de pectina.**

***Palabras claves*— Extracción sólido líquido, grado de esterificación, hidrólisis ácida, manejo de residuos, metodología de superficie de respuesta, pectina.**

I. Introduction

COFFEE is one of the most important agricultural products in the world and Colombia is internationally recognized for its coffee quality, aroma, and flavor. There are mainly two methods for the treatment of coffee fruits: wet processing and dry processing. Approximately half of the world coffee harvest is processed by the wet method in which the coffee berry is subjected to mechanical and biological operations in order to separate the seed from the exocarp (husk), the mesocarp (mucilaginous pulp), and the endocarp (seed). This is the preferred method for processing in Colombia [1], [2].

The coffee pulp is one of the main by-products in the coffee-making process and constitutes between 40 and 50% of the wet weight of the fruit (including the husk)[1], [2]. This residue is rich in carbohydrates, proteins, minerals, and appreciable amounts of tannins, caffeine, and potassium [1], [3], [4]. These wastes are generally underutilized. Its disposal in tropical coffee-producing countries creates a problem because its elimination causes environmental contamination, due to the putrefaction of organic matter. This is why the need arises to develop and implement adequate management mechanisms for this waste material [1].

In the framework of a circular economy, efforts have been

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made to develop mechanisms for the treatment and management of coffee waste, including its use as raw material for the production of food, beverages, vinegar, biogas, caffeine, pectic enzymes, proteins, compost, and pectin, etc. [1], [5]. This last product, pectin, is the central topic of this article.

Pectin is a natural biopolymer with various applications in the food, pharmaceutical, and biotechnology industries; and has been commonly used as a thickening, gelling, and colloidal stabilizer agent in food and beverages [6], [7]. It is a complex heteropolysaccharide composed of d-galacturonic acid residues linked by α-1-4 bonds that form homogalacturonan chains [6], [8]; and it is found commercially, almost exclusively, as a derivative of citrus peel or apple pomace [9], [10], both by-products in the manufacture of apple juice (or cider). However, taking into account the growing industrial demand for pectin in global markets (more than 5% per year) due to the wide variety of technological and biological applications of this polysaccharide [6], [11], it is important to find new sources that are unconventional and have competitive and economic yields, to obtain this product of industrial interest [10].

Obtaining pectin from various sources has been a widely studied and published topic, but it is difficult to characterize this as a model system due to the heterogeneous nature of the polymer and the particular characteristics of each plant. A useful tool that can be used for both statistical modeling and experimental design is the response surface methodology (RSM). From this methodology a model is obtained that allows the establishment of the best conditions in the extraction process by evaluating parameters such as pH, temperature and extraction time, the degree of esterification and the extraction performance. The response surface methodology has been previously reported for the adequacy of the pectin extraction process from other plant sources such as banana peel [12], pomegranate peel [13], passion fruit peel [14], pea hulls [15], citrus peel [16], cocoa peel [17], [18], mango peel [9], guava [19], jackfruit [20], and others. With the aim of generating a proposal for the utilization of residues in the agro-industrial sector within the framework of the circular economy, in this study, the effect of the extraction conditions on the efficacy of obtaining pectin and its degree of esterification from agroindustrial coffee residues is reported, using a response surface methodology.

II. Methods

A. Agroindustrial material

The agroindustrial coffee residues were obtained from coffee farms in the municipality of San Juan de Rioseco in the department of Cundinamarca, Colombia. These residues were crushed to reduce their particle size, and preserved by freezing at -80ºC. The sampling unit for the experiments was selected by waste quartering method, and consisted of 100 grams of residue for each experimental replicate.

B. Pectin extraction from coffee by-products

Pectin extraction was carried out by acid hydrolysis using hydrochloric acid in a closed reflux with a condensation system according to what was reported [9], [10], [20]. A ratio of 1 portion of coffee residue to 4 portions of water was used. In the extraction, the effect of three variables was measured:

1) the pH was adjusted by adding 1 mol L^{-1} hydrochloric acid with a potentiometer (between 1.5 and 3.5 pH units),

2) the hydrolysis time (between 15 minutes and 90 minutes), and

3) the hydrolysis temperature is controlled in a thermostated water bath (between 70ºC and 90ºC).

The extraction conditions evaluated were selected based on the literature review conducted in the previously referenced articles where the Response Surface Methodology (RSM) is used as a method for optimizing conditions.

The warm, acidified extract was cooled to room temperature and centrifuged at 6000 rpm for 15 minutes. The supernatant was precipitated with 96% ethanol in a 1:1 v/v ratio and then allowed to stand for one hour to allow the pectin to float. The floating pectin was filtered off and rinsed with 96% ethanol. The resulting pectin was dried in a forced convection oven at 45°C for 12 h. The extraction yield was calculated gravimetrically and reported as the percentage of pectin extracted compared to the total weight of the raw material used. The following formula (equation 1), was used to determine pectin yield:

$$\%\, Yield\ of\ pectin = \frac{Weight\ of\ pectin}{Weight\ of\ byproducts} * 100 \quad (1)$$

All procedures were performed in triplicate.

C. Determination of the degree of esterification

Fourier transform infrared spectra (FTIR-ATR) were obtained from pectin obtained in the frequency range of 4000-800 cm-1 in a Shimadzu IR PRESTIGE-2 spectrophotometer. The samples were placed in a reflectance attenuator (ATR). The empty glass was used as a reference. The degree of esterification was calculated by the equations (2) and (3) of Pappas et al. [21], according to that reported by Minjares-Fuentes et al. [22]:

$$DE = 124R + 2.2013 \quad (2)$$

$$R = \frac{A_{1740}}{A_{1740} + A_{1630}} \quad (3)$$

Where DE is the degree of esterification, A1740 y A1630 were defined as the absorbance intensities of the bands caused by vibrations of the esterified and unesterified carboxyl groups at 1740 cm^{-1} and 1630 cm^{-1} [22].

D. Experimental design and statistical analysis

A central compound design (CCD) with three independent variables (temperature, time and pH) was used for the experiment. The response surface methodology (RSM) was used to evaluate the effect of the study variables and determine the best conditions for the extraction of pectin from agroindustrial coffee residues, using as dependent variables the pectin extraction yield and the degree of esterification. With the results, a complete second-order model was tested according to equation 3.

$$y = k_0 + k_1 t + k_2 T + k_3 pH + \beta_1 t^2 + \beta_2 T^2 + \beta_3 pH^2 + \alpha_1 tT + \alpha_2 tpH + \alpha_3 TpH + e \quad (3)$$

Where y is the answer; ki, βi and αi are constant; t and T are the extraction time and temperature respectively; and e is the random error associated with the model. The RSM analysis, principal component analysis, and residues plots was performed in the trial version of Minitab 17.

III. Results and Discussion

Pectin is a natural biopolymer with various applications in food, pharmaceutical and biotechnology industries. In this work, the obtaining of this industrially valuable product from agro-industrial coffee by-products was proposed.

The degrees of esterification of the pectin obtained under working conditions ranged between 60% and 75% and makes it possible to classify them as high methoxyl pectin. Pectin can be characterized by different parameters, the most important being the degree of methoxylation or degree of esterification (GE), which refers to the percentage of methoxylated C atoms in the galacturonic acid skeleton, and is related to the gelation properties [12]. Pectin can be classified as high methoxy content if the degree of esterification is greater than 50%, and low methoxy pectin content for degrees of esterification less than 50% [12], [23]. This percentage is the one that determines the possible use of pectin in the industry. Pectin with a high degree of esterification is preferred.

Lower temperatures and times, and higher pH values produce pectin with greater methoxylation that agrees with that reported in pectin from other plant sources [9], [12], [13]. Another parameter of interest is the yield percentage that was relatively low compared to other plant sources reported in the articles that have been cited, finding results between 0.14% and 2.0% under the study conditions.

In order to optimize the extraction, the response surface methodology using Minitab 17 was used to define the conditions that produced a maximum percentage of yield while maintaining a high degree of methoxylation. Based on the results of the surface model of responses for the degree of esterification and the percentage of yield, equations 4 and 5 were formulated and three-dimensional graphs were built (Fig. 1 and 2 where two variables are represented in a 3D surface graph while that the other variable remained constant).

$$\%R = -1{,}90 - 0{,}75pH + 0{,}783t + 0{,}057T - 0{,}177\,pH^2 - 0{,}173t^2 - 0{,}00053T^2 + 0{,}0180pH * T \quad (4)$$

$$DE = 323 - 52{,}0\,pH + 1{,}1t - 4{,}69T + 1{,}28\,pH^2 - 3.52t^2 + 0{,}0211T^2 + 0{,}528\,pH * T \quad (5)$$

The three-dimensional graphs are the graphical representations of the regression models that provide a method to visualize the relationship between the responses and experimental levels of each variable and the type of interactions between pairs of variables for the responses percentage of yield (Fig. 1) and degree of esterification (Fig. 2).

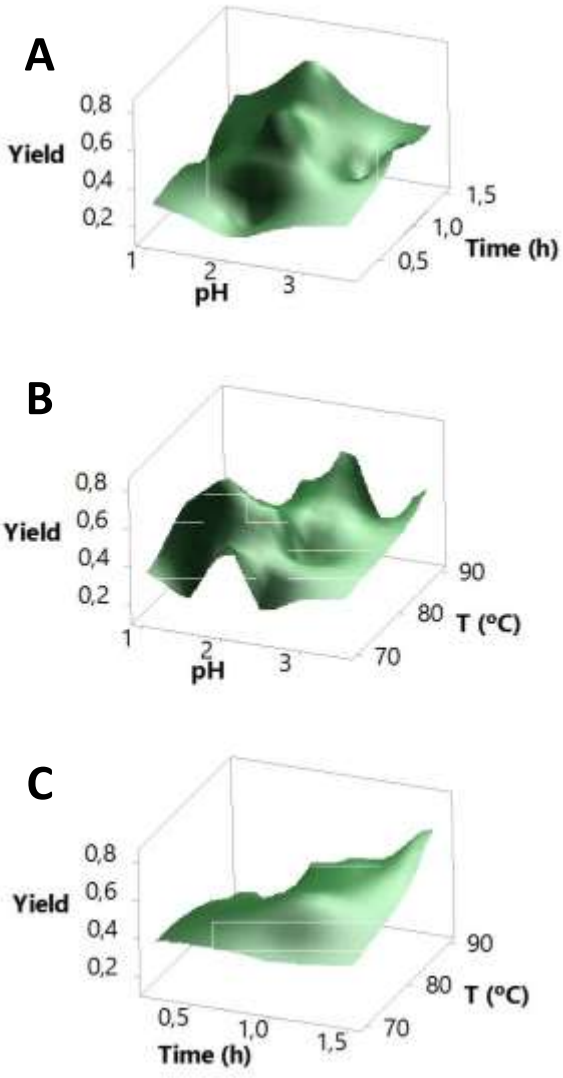

Fig. 1. Surface graphs for the variables studied. A Yield (%) as a function of pH and time. B: Yield (%) as a function of pH and temperature. C: Yield (%) as a function of time and temperature. The surface plots were made in Minitab 17.

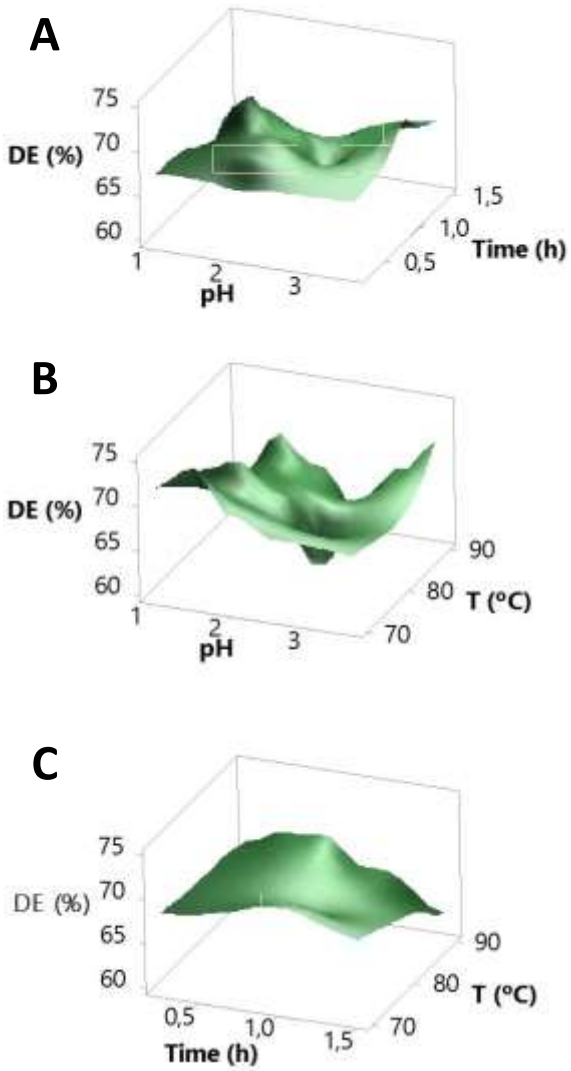

Fig. 2. Surface graphs for the variables studied. A: Degree of esterification (DE) as a function of pH and time. B: Degree of esterification (DE) as a function of pH and temperature. C: Degree of esterification (DE) as a function of time and temperature. The surface plots were made in Minitab 17.

Similar to that of pectin obtained from grape residues [22], pectin from banana peel [24], and pectin from walnut processing wastes [6], we found that the pH is the parameter that most affects the performance in the extraction of pectin in agroindustrial coffee residues.

The pectin yield decreases with increasing pH at different extraction temperatures. This indicates that extraction is favored by acidity. However, in an environment that is too acidic, pectin will over-hydrolyze, degrease and be subject to cleavage [16], obtaining lower yields, so an extraction pH of close to 2 is recommended for the process.

At constant pH, it was determined that the longer the time and the higher the temperature the yield for obtaining pectin was higher. However, the product obtained decreases in quality, since it has lower degrees of esterification. This is due to the

fact that high temperatures and long exposure times favour hydrolysis reactions of glycosidic bonds, demethylation reactions, and polymer breakdown [16].

In the residual plot (Fig. 3A and 3B), it can be observed that the residuals are randomly distributed around zero, without showing a discernible pattern. This suggests that the statistical model adequately captures the variability of the experimental data and that there is no systematic trend in the residuals. Therefore, we can infer that the model is a good representation of the data. The Principal Component Analysis (PCA) confirms the findings of the response surface model. Fig. 3C shows the PCA projection plot. Principal Component 1 explains 33.6% of the variance and confirms the positive relationship between extraction time and pectin yield, while the degree of esterification shows a negative relationship with extraction time and temperature.

According to the model obtained through response surface methodology, it was determined that the optimal conditions for coffee pectin extraction, in relation to the response variables, are pH 2, an extraction temperature of 90°C, and a hydrolysis time of one hour. The suitability of the models in predicting the responses was tested by conducting pectin extraction under the selected optimal conditions. The experimental values aligned with the predicted values (Table I). Overall, it was observed that the model adequately predicts the response variables, as evident from the correlations between the predicted and experimentally obtained results, as depicted in the residual plots. The adjusted model provides a better approximation for predicting the degree of esterification, exhibiting only a 1.5% difference compared to the values predicted by the model for the optimal extraction conditions.

TABLE I
EXPERIMENTAL AND PREDICTED RESULTS OF THE STUDY VARIABLES ACCORDING TO THE EQUATION OF THE RESPONSE SURFACE MODEL.

	Experimental (%)	Predicted (%)	Difference %
Yield	0.58 ± 0.04	0.62	6.4
Degree of esterification	65.6 ± 0.5	66.6	1.5

The results presented correspond to the best conditions for the extraction of coffee pectin with respect to the response variables (pH 2, an extraction temperature of 90°C and a hydrolysis time of one hour). The experimental results are presented as the mean ± standard deviation for an n=3

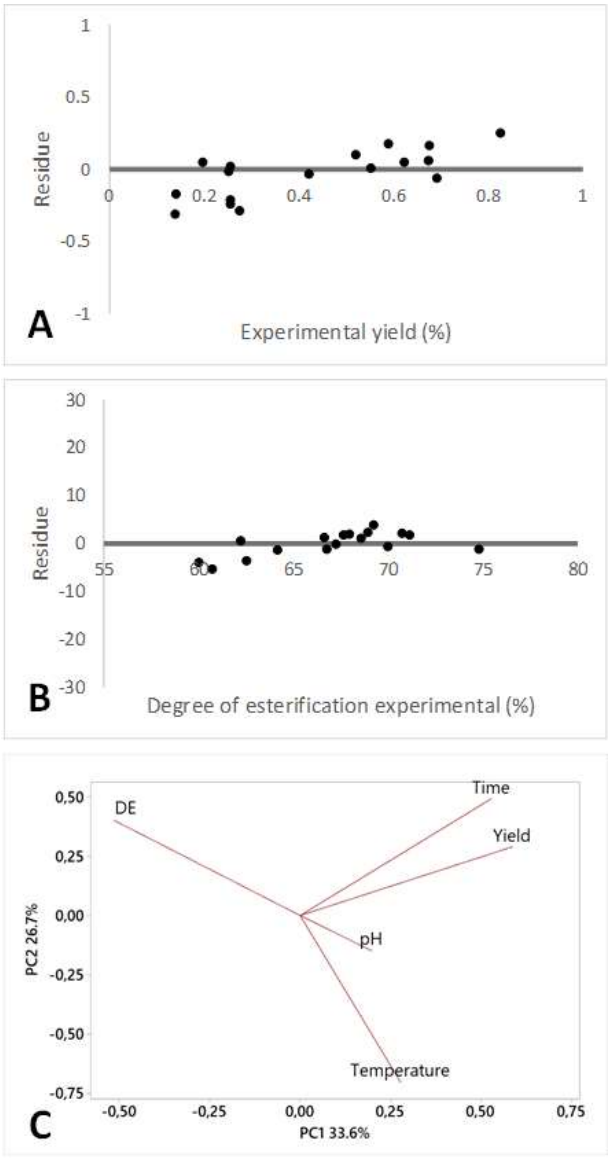

Fig. 3. Model verification: Residue between the experimental results and the results predicted by the response surface model for A. The yield, and B. The degree of esterification (DE). C. PCA projection plot

The pectin extracted under the response surface model conditions was characterized by infrared FTIR spectrophotometry. The infrared spectrum of the pectin extracted from the coffee husk under the extraction conditions resulting from the response surface model is shown in Fig. 4. A wide absorption around 3321 cm^{-1} of polysaccharides caused by stretching of hydroxyl groups is observed, as well as two peaks between 3000 cm^{-1} and 2850 cm^{-1} that corresponded to the asymmetric and symmetric stretching of the CH bond in the galacturonic ring of aliphatic carbons. The absorption at 1732 cm^{-1} corresponded to the stretching vibration of the C=O bond of the esterified carboxyl group, while the absorption at approximately 1647 cm^{-1} was attributed to the vibration stretching of the carboxylate ion bond (non-esterified carboxyl group). The relationships of these absorptions are used to

calculate the degree of esterification, according to those reported by Minjares-Fuentes et al. [22] and according to equation 2, showing a degree of esterification that classifies pectin as high methoxy by being greater than 50% [10], [25]. The absorption peaks between 1010 cm^{-1} and 1150 cm^{-1} indicate that the sample contains pyranose similar to that reported by Wang et al. [26]. The results found for coffee pectin both in the form of the infrared spectrum and its absorptions agree with those reported in pectin obtained from tomato residues [25], in banana peel [12], in pomegranate peel [13], and pectin extracted from grapefruit peel [26].

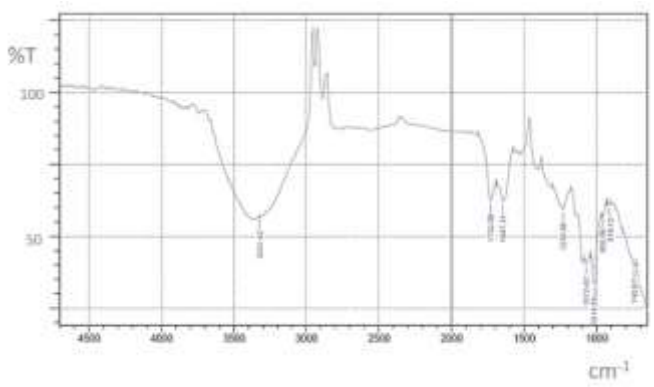

Fig. 4. FTIR spectrum for extracted pectin at pH 2 with closed reflux and heating at a constant temperature of (90 ° C x 60 Minutes).

Taking into account that pectin is used as an additive (thickening agent, gelling agent and colloidal stabilizer) [27], [28], raw material that in Colombia must be exported from producing countries such as Germany, Mexico, Brazil, China, India among other producing countries [27], the results found are an approximation to the possibility of generating a suitable waste management proposal for the coffee agroindustry.

IV. CONCLUSION

The pectin was successfully extracted from agroindustrial coffee by-products from the municipality of San Juan del Rio Seco, Cundinamarca, under different conditions of pH, temperature and extraction time. The high temperature and strongly acidic pH conditions resulted in a higher extraction yield, but at the cost of decreasing the degree of methoxylation for a maximum of 75% for a minimum of 60%. The optimal conditions for pectin extraction, defined as those that produced a maximum extraction yield, while maintaining a degree of methoxylation that classifies the pectin obtained as having a high degree of methoxylation, were: 90°C, 1 h, pH 2.0.

The results suggest the potential for conducting a pectin extraction process from this type of waste, aiming to achieve proper waste management for the agroindustry. While the yield results are lower compared to conventional pectin sources, it is recommended to implement drying processes before extraction to enhance this parameter. Additionally, future studies should consider scaling up to a pilot plant to assess the process efficiency and evaluate the characteristics of the product obtained.

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REFERENCES

[1] R. Padmapriya, J. A. Tharian, and T. Thirunalasundari, “Coffee waste management-An overview,” *Int J Curr Sci*, vol. 9, pp. 83–91, 2013.

[2] É. Mendes dos Santos *et al.*, “Coffee by-products in topical formulations: A review,” *Trends Food Sci. Technol.*, vol. 111, pp. 280–291, 2021, doi: 10.1016/j.tifs.2021.02.064.

[3] M. C. Echeverria and M. Nuti, “Valorisation of the Residues of Coffee Agro-industry: Perspectives and Limitations,” *Open Waste Manag. J.*, vol. 10, no. 1, pp. 13–22, 2017, doi: 10.2174/1876400201710010013.

[4] L. R. Palomino García, C. R. Biasetto, A. R. Araujo, and V. L. del Bianchi, “Enhanced extraction of phenolic compounds from coffee industry’s residues through solid state fermentation by Penicillium purpurogenum,” *Food Sci. Technol.*, vol. 35, no. 4, pp. 704–711, 2015, doi: 10.1590/1678-457X.6834.

[5] A. K. Singh and R. Sharma, “A review on sustainable management of coffee industry by-products,” *J. Crit. Rev.*, vol. 7, no. 10, pp. 686–691, 2020, doi: 10.31838/jcr.07.10.137.

[6] K. Asgari, M. Labbafi, F. Khodaiyan, M. Kazemi, and S. S. Hosseini, “High-methylated pectin from walnut processing wastes as a potential resource: Ultrasound assisted extraction and physicochemical, structural and functional analysis,” *Int. J. Biol. Macromol.*, vol. 152, pp. 1274–1282, 2020, doi: 10.1016/j.ijbiomac.2019.10.224.

[7] A. M. I. Encalada, C. D. Pérez, S. K. Flores, L. Rossetti, E. N. Fissore, and A. M. Rojas, “Antioxidant pectin enriched fractions obtained from discarded carrots (Daucus carota L.) by ultrasound-enzyme assisted extraction,” *Food Chem.*, vol. 289, pp. 453–460, 2019, doi: 10.1016/j.foodchem.2019.03.078.

[8] C. L. O. Petkowicz and P. A. Williams, “Pectins from food waste: Characterization and functional properties of a pectin extracted from broccoli stalk,” *Food Hydrocoll.*, vol. 107, p. 105930, 2020, doi: 10.1016/j.foodhyd.2020.105930.

[9] A. do N. Oliveira, D. de Almeida P, E. B. de Oliveira, S. Henriques Saraiva, P. C. Stringheta, and A. Mota Ramos, “Optimization of pectin extraction from Ubá mango peel through surface response methodology,” *Int. J. Biol. Macromol.*, vol. 113, pp. 395–402, 2018, doi: 10.1016/j.ijbiomac.2018.02.154.

[10] M. Yu, Y. Xia, M. Zhou, Y. Guo, J. Zheng, and Y. Zhang, “Effects of different extraction methods on structural and physicochemical properties of pectins from finger citron pomace,” *Carbohydr. Polym.*, vol. 258, p. 117662, 2021, doi: 10.1016/j.carbpol.2021.117662.

[11] N. Muñoz-Almagro, L. Valadez-Carmona, J. A. Mendiola, E. Ibáñez, and M. Villamiel, “Structural characterisation of pectin obtained from cacao pod husk. Comparison of conventional and subcritical water extraction,” *Carbohydr. Polym.*, vol. 217, pp. 69–78, 2019, doi: 10.1016/j.carbpol.2019.04.040.

[12] T. Í. S. Oliveira *et al.*, “Optimization of pectin extraction from banana peels with citric acid by using response surface methodology,” *Food*

Chem., vol. 198, pp. 113–118, 2016, doi: 10.1016/j.foodchem.2015.08.080.

[13] P. H. F. Pereira *et al.*, "Pectin extraction from pomegranate peels with citric acid," *Int. J. Biol. Macromol.*, vol. 88, pp. 373–379, 2016, doi: 10.1016/j.ijbiomac.2016.03.074.

[14] E. E. Santos, R. C. Amaro, C. C. C. Bustamante, M. H. A. Guerra, L. C. Soares, and R. E. S. Froes, "Extraction of pectin from agroindustrial residue with an ecofriendly solvent: use of FTIR and chemometrics to differentiate pectins according to degree of methyl esterification," *Food Hydrocoll.*, vol. 107, p. 105921, 2020, doi: 10.1016/j.foodhyd.2020.105921.

[15] F. Gutöhrlein, S. Drusch, and S. Schalow, "Extraction of low methoxylated pectin from pea hulls via RSM," *Food Hydrocoll.*, vol. 102, p. 105609, 2020, doi: 10.1016/j.foodhyd.2019.105609.

[16] Z. Xue *et al.*, "Optimization of Pectin Extraction from Citrus Peel by Response Surface Methodology," *Food Sci.*, vol. 32, no. 18, pp. 128–132, 2011.

[17] L. Hennessey-Ramos, W. Murillo-Arango, J. Vasco-Correa, and I. C. Pazastudillo, "Enzymatic Extraction and Characterization of Pectin from Cocoa Pod Husks (Theobroma cacao L.) Using Celluclast® 1.5 L," *Molecules*, vol. 26, p. 1473, 2021.

[18] L. C. Vriesmann, R. F. Teófilo, and C. Lúcia de Oliveira Petkowicz, "Extraction and characterization of pectin from cacao pod husks (Theobroma cacao L.) with citric acid," *LWT - Food Sci. Technol.*, vol. 49, pp. 108–116, 2012, doi: 10.1016/j.lwt.2012.04.018.

[19] M. M. Kamal, M. Akhtaruzzaman, T. Sharmin, M. Rahman, and S. C. Mondal, "Optimization of extraction parameters for pectin from guava pomace using response surface methodology," *J. Agric. Food Res.*, vol. 11, p. 100530, 2023, doi: 10.1016/j.jafr.2023.100530.

[20] N. T. Kim Tran, V. Bao Nguyen, T. Van Tran, and T. T. Thanh Nguyen, "Microwave-assisted extraction of pectin from jackfruit rags: Optimization, physicochemical properties and antibacterial activities," *Food Chem.*, vol. 418, p. 135807, 2023, doi: 10.1016/j.foodchem.2023.135807.

[21] C. S. Pappas, A. Malovikova, Z. Hromadkova, P. A. Tarantilis, A. Ebringerova, and M. G. Polissiou, "Determination of the degree of esterification of pectinates with decyl and benzyl ester groups by diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) and curve-fitting deconvolution method," *Carbohydr. Polym.*, vol. 56, pp. 465–469, 2004, doi: 10.1016/j.carbpol.2004.03.014.

[22] R. Minjares-Fuentes, A. Femenia, M. C. Garau, J. A. Meza-Velázquez, S. Simal, and C. Rosselló, "Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach," *Carbohydr. Polym.*, vol. 106, no. 1, pp. 179–189, 2014, doi: 10.1016/j.carbpol.2014.02.013.

[23] D. Zhang *et al.*, "Comparative proteomic analysis of cucumber roots infected by Fusarium oxysporum f. sp. cucumerium Owen," *Physiol. Mol. Plant Pathol.*, vol. 96, pp. 77–84, 2016, doi: 10.1016/j.pmpp.2016.09.002.

[24] T. Happi Emaga, S. N. Ronkart, C. Robert, B. Wathelet, and M. Paquot, "Characterisation of pectins extracted from banana peels (Musa AAA) under different conditions using an experimental design," *Food Chem.*, vol. 108, no. 2, pp. 463–471, 2008, doi: 10.1016/j.foodchem.2007.10.078.

[25] A. N. Grassino, M. Brnčić, D. Vikić-Topić, S. Roca, M. Dent, and S. R. Brnčić, "Ultrasound assisted extraction and characterization of pectin from tomato waste," *Food Chem.*, vol. 198, pp. 93–100, 2016, doi: 10.1016/j.foodchem.2015.11.095.

[26] W. Wang *et al.*, "Characterization of pectin from grapefruit peel: A comparison of ultrasound-assisted and conventional heating extractions," *Food Hydrocoll.*, vol. 61, pp. 730–739, 2016, doi: 10.1016/j.foodhyd.2016.06.019.

[27] R. Ciriminna, N. Chavarría-Hernández, A. I. Rodríguez Hernández, and M. Pagliaro, "Pectin: A new perspective from the biorefinery standpoint," *Biofuels, Bioprod. Biorefining*, vol. 9, no. 4, pp. 368–377, 2015, doi: 10.1002/bbb.1551.

[28] A. A. Sundar Raj, R. Jayabalan, and T. V. Ranganathan, "A Review on Pectin: Chemistry due to General Properties of Pectin and its Pharmaceutical Uses," *Sci. Rep.*, vol. 1, no. 12, p. 550, 2012, doi: 10.4172/scientificreports.550.

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