ASSESSMENT OF MICROWAVE POWER INTENSITY ON THE YIELD AND PHYSICOCHEMICAL PROPERTIES OF PECTIN EXTRACTED FROM ORANGE PEEL (*Citrus sinensis*)

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ABSTRACT

In this study, a microwave-assisted extraction method was evaluated for the extraction of pectin from orange peel, conducted at different microwave power intensities (100, 150, 200, 250, and 300 W) regarding yield and its effect on pectin characteristics (galacturonic acid content, degree of esterification, and methoxyl content). The highest pectin yield (28.93a ± 0.02%) was observed when using a microwave power of 150 W, although this value is statistically equal to the yield obtained at 200 W (27.68 ± 3.49). Regarding pectin characterization, the pectin extracted at 200 W showed the highest galacturonic acid content (79.03 ± 1.94), indicating that this power intensity presented the best condition for pectin extraction from orange peel.

Keywords: Microwave-assisted extraction. Orange peel. Pectin.

1 INTRODUCTION

The processing of orange juice generates approximately 50-60% organic waste composed of peels, seeds, and pulp, amounting to approximately 12,000,000 tons annually, which is of great environmental concern[1,2]. One solution for the low-cost utilization of orange peel waste is for pectin extraction[3]. Pectin is a complex polysaccharide consisting mainly of a linear chain of D-galacturonic acid branched with smaller sugar side chains. In the food industry, pectins are widely used for their thickening, emulsifying, and gelling properties in sauce and jam formulations[4].

Conventional pectin extraction typically requires high temperatures (80–90°C), low pH values (2–3), long extraction times (1–5 hours), and high solid-liquid ratios (1:30–1:50)[5]. However, these extraction conditions can decrease the quality of the extracted pectin[6]. An alternative extraction method needed to enhance pectin quality is microwave-assisted extraction (MAE). This extraction type can reduce energy and solvent consumption and decrease extraction time[7]. Rahmati et al. (2019)[7] conducted microwave-assisted extraction by varying the equipment power intensity at different levels (300, 450, 600, and 800W) to extract pectin from dragon fruit peels. The authors concluded that the microwave-assisted extraction process was effective in obtaining high-quality fruit peel pectin, with a galacturonic acid content ranging from 59.73 to 69.68%.

In this study, the main objective is to evaluate the microwave-assisted extraction method implemented in the extraction of orange peel pectin, conducted at different microwave power intensities (100, 150, 200, 250, and 300 W) regarding yield and its effect on pectin characteristics (galacturonic acid content, degree of esterification, and methoxyl content).

2 MATERIAL & METHODS

Oranges (*Citrus sinensis*) were purchased from a market (Montreal, Canada) and initially peeled. The peels were then cut into small pieces and dried in a circulating air oven (Model 40AF, Quincy Lab Inc) at 50°C until a constant weight was achieved. The dried peels were ground and sieved (40 mesh) to obtain the powdered sample. The orange peel powder was stored in a dry and light-protected environment until use.

The orange peel powder was mixed with distilled water acidified with HCl to a pH of 2 and a solid-liquid ratio of 1:30 (w/v)[8]. The solutions were placed in a sealed microwave reactor (Anton Paar 400 Monowave, USA), and the influence of different microwave powers on pectin yield and quality was investigated. Microwave powers used were 100, 150, 200, 250, and 300 W, with an extraction time of 6 minutes.

The precipitation and purification steps were conducted following Dranca et al. (2020)[9]. After each extraction, the solid material was separated from the pectin by centrifugation (4000 rpm) for 40 minutes (Sigma, MBI Lab Equipment), the supernatant was collected, filtered, precipitated by adding ethanol (96%, v/v), and kept at 4–6°C for 12 hours for complete precipitation. The precipitated pectin was separated by centrifugation (4000 rpm, 40 minutes), washed with ethanol, and dried in a circulating air oven at 50°C until a constant weight was achieved. The pectin yield was calculated using Equation 1:

$$Pectin\ yield\ (\%) = \frac{m}{m_c}\ x\ 100 \quad (1)$$

where m is the weight of dry pectin (g) and m_c is the mass of orange peel powder.

Pectin samples were analyzed for galacturonic acid (GA) content, degree of esterification (DE), and methoxyl content (MC).

Galacturonic acid content (GA) was determined by the m-hydroxydiphenyl spectrophotometric method suggested by Melton and Smith (2001)[10]. The degree of esterification (DE) of pectin samples was determined according to the method described in the USP NF 21 (United States Pharmacopeia – The National Formulary, 2003)[11]. The methoxyl content (MC) of pectin samples was measured according to Ranggana (1986)[12].

All pectin characterization analyses were performed in triplicate and statistically analyzed by the Tukey test at a significance level of 5%

3 RESULTS & DISCUSSION

The effects of microwave power on the yield and characterizations of pectin are presented in Table 1.

Table 1: Yield and characterization of pectin extracted from orange peel at different microwave powers.

Microwave power (W)	Pectin Yield (%)	GA (%)	DE (%)	MC (%)
100	$27.27^{a} \pm 0.93$	$71.41^{b} \pm 0.52$	$56.83^{a} \pm 0.44$	$8.09^{b} \pm 0.11$
150	$28.93^{a} \pm 0.02$	$74.93^{b} \pm 2.44$	$43.75^{c} \pm 2.94$	$10.15^{a} \pm 0.57$
200	$27.68^{a} \pm 3.49$	$79.03^{a} \pm 1.94$	$43.15^{c} \pm 0.98$	$10.09^{a} \pm 0.20$
250	$20.67^{b} \pm 1.21$	$57.02^{c} \pm 2.90$	$44.01^{c} \pm 1.41$	$10.91^{a} \pm 1.08$
300	$19.99^{b} \pm 0.11$	$54.26^{c} \pm 2.96$	$43.77^{c} \pm 3.38$	$10.09^{a} \pm 0.47$

The highest pectin yield value (28.93a ± 0.02%) was observed when using a microwave power of 150 W. However, there was no statistically significant difference between the powers of 100, 150, and 200 W. There was a tendency for yield to decrease as microwave power increased, with the lowest extraction yield (30.17 ± 1.49%) observed at 300 W. Nevertheless, there was no statistically significant difference ($p \leq 0.05$) between 250 W and 300 W. The extraction yield of pectin from orange peel was significantly decreased by microwave power, likely due to increased temperature inside the microwave reactor, leading to pectin degradation when higher powers were used[13].

The values for GA ranged from 54.26 to 79.03%. However, to be considered pectin, the GA value should not contain less than 65% galacturonic acid[14]. Hence, for powers starting from 250 W, the GA value is no longer acceptable for pectin. The DE values ranged from 43.15 to 56.83%, and the methoxyl content values for all samples were above 7%, indicating that the pectin is classified as high-methoxyl pectin. It was observed that the galacturonic acid content (GalA) and degree of esterification (DE) increased with the increase in microwave power up to 250 W. This trend was also observed in the work of Bagherian et al. (2011)[15], who observed the same behavior in grapefruit pectin extraction.

4 CONCLUSION

The effects of microwave power variation were investigated in the extraction of pectin from orange peel. It can be concluded that extraction carried out at 200 W resulted in high-quality pectin with a galacturonic acid content of 79.03% and a higher extraction yield of 27.68%. Therefore, it can be concluded that this power level provided the best condition for pectin extraction from orange peel.

REFERENCES

1 PFALTZGRAFF, L.A., DE BRUYN, M., COOPER, E.C., BUDARIN, V.,CLARK, J.H. Food waste biomass: A resource for high-value chemicals, Green Chem. 15 (2013) 307–314.

2 SATARI, B., KARIMI, K. Citrus processing wastes: Environmental impacts, recent advances, and future perspectives in total valorization, Resour. Conserv. Recycl. 129 (2018) 153–167.

3 WICKER, L., KIM, Y., KIM, M.J., THIRKIELD, B., LIN, Z., JUNG, J. Pectin as a bioactive polysaccharide - Extracting tailored function from less, Food Hydrocoll. 42 (2014) 251–259.

4 MARIĆ, M., GRASSINO, A.N., ZHU, Z., BARBA, F.J., BRNČIĆ, M., RIMAC BRNČIĆ, S. An overview of the traditional and innovative approaches for pectin extraction from plant food wastes and by-products: Ultrasound-, microwaves-, and enzyme-assisted extraction, Trends Food Sci. Technol. 76 (2018) 28–37.

5 KANG, J., HUA, X., YANG, R., CHEN, Y., YANG, H. Characterization of natural low-methoxyl pectin from sunflower head extracted by sodium citrate and purified by ultrafiltration, Food Chem. 180 (2015) 98–105.

[6] PAGÁN, J., IBARZ, A., LLORCA, M., PAGÁN, A., BARBOSA-CÁNOVAS, G. V. Extraction and characterization of pectin from stored peach pomace, Food Res. Int. 34 (2001) 605–612.

[7] RAHMATI, S., ABDULLAH, A., KANG, O.L. Effects of different microwave intensity on the extraction yield and physicochemical properties of pectin from dragon fruit (Hylocereus polyrhizus) peels, Bioact. Carbohydrates Diet. Fibre. 18 (2019) 100186.

[8] ALTAF, U., IMMANUEL, G., IFTIKHAR, F. Extraction and Characterization of Pectin From Turkish, 1 (2015) 91–94.

[9] DRANCA, F., VARGAS, M., OROIAN, M. Physicochemical properties of pectin from Malus domestica ' F ă lticeni ' apple pomace as a ff ected by non-conventional extraction techniques, Food Hydrocoll. 100 (2020) 105383.

[10] MELTON, L.D., SMITH, B.G. Determination of the Uronic Acid Content of Plant Cell Walls Using a Colorimetric Assay, Curr. Protoc. Food Anal. Chem. 00 (2001) E3.3.1-E3.3.4.

[11] USP, USP NF 21, The United States pharmacopeia – The national formulary, in: United States Pharmacopeial Conv., Rockville, 2003: pp. 1401–1402.

[12] RANGGANA, S. Handbook of analysis and quality control for fruit and vegetable products, 1986.

[13] GUO, X., HAN, D. , XI, H., RAO, L., LIAO, X., HU, X., WU, J. Extraction of pectin from navel orange peel assisted by ultra-high pressure, microwave or traditional heating: A comparison, Carbohydr. Polym. 88 (2012) 441–448.

[14] MÜLLER-MAATSCH, J., BENCIVENNI, M., CALIGIANI, A., TEDESCHI, T., BRUGGEMAN, G., BOSCH, M., PETRUSAN, J., VAN DROOGENBROECK, B., ELST, K., SFORZA, S. Pectin content and composition from different food waste streams in memory of Anna Surribas, scientist and friend., Food Chem. 201 (2016) 37–45.

[15] BAGHERIAN, H., ZOKAEE ASHTIANI, F., FOULADITAJAR, A., MOHTASHAMY, M. Comparisons between conventional, microwave- and ultrasound-assisted methods for extraction of pectin from grapefruit, Chem. Eng. Process. Process Intensif. 50 (2011) 1237–1243.

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