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RESEARCH

Microwave Assisted Green Extraction of Pectin from Citrus *maxima* Albedo and Flavedo, Process Optimization, Characterisation and Comparison with Commercial Pectin

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Abstract

The present study demonstrates that the albedo (A_P) and flavedo (F_P) portions of *Citrus maxima*, are often discarded as agricultural waste that has the potential to manufacture high-quality pectin. The efficacy of green microwave aided extraction (MAE) technology for pectin extraction yield from both the portions of citrus peels was examined using a response surface technique. However, further advancement has to be made in improving the functional parameters required for MAE. The optimal conditions for increased pectin yields for albedo (Y_{AP} %) and flavedo (Y_{FP} %) were found to be solid to solvent ratio (1:15), time (95 s) and power (450 watt). The predicted values of Y_{AP} (5.35%) and Y_{FP} (3.13) were in accordance with experimental values of Y_{AP} (5.57 %) and Y_{FP} (3.09 %) with small error % i.e., 3.90 % and 1.29 % for A_P and F_P respectively. The A_P and F_P extracted at optimum conditions were compared with commercial pectin (C_p) based on physicochemical properties, color, FT-IR, FE-SEM/EDX, XRD, and particle size analysis. Experimental result shows that A_P and F_P were classified as high methoxy pectin due to high DE values 72.76 % and 74.98 % respectively.

Keywords Microwave extraction · Pectin · Citrus *maxima* · Response surface methodology

Introduction

Citrus peels are the most prevalent commercial source of pectin owing to their high pectin concentration of 25-30 % in dry peel mass (Bagherian et al. 2011; Quoc et al. 2015). Pomelo, also known as "pummelo" or *Citrus maxima* (Burm.) Merr, is the largest citrus fruit of the *Rutaceae* (citrus) family, and its peels have been stated to be rich in numerous nutrients and functional compounds (Xiao et al. 2021). According to FAO (Food and Agriculture Organization of United Nations), over 9.3 million metric tonnes of pomelo are produced globally each year. The use of pomelo peels might result not only in value-added products/ingredients, but also in reduced environmental hazards (Xiao et al. 2021). Pectin, a complex polysaccharide predominantly consisting of α-1-4 d-galacturonic acid (GalA), has long been utilized as an important food additive, with a global demand of around 45,000 metric tonnes per year and a 4-5 % yearly growth rate (Jeong et al. 2013; Raji et al. 2017). This polysaccharide finds applications in the culinary, cosmetic, and pharmaceutical sectors for its thickening, stabilizing and gelling properties (Mahmud et al. 2020). According to FAO regulations, pectin should contain more than 65% GalA to be regarded "desirable" in terms of

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quality (Adetunji et al. 2017). The most frequent techniques of generating pectin are extraction, purification, and drying. The selection of an appropriate extraction technique is one of the most crucial variables impacting pectin quality and quantity.

The conventional hot-acidified water extraction method employed to extract pectin is tedious, laborious, and thermally hazardous. The process additionally necessitates high utilization of solvent while possesses low efficiency (Liew et al. 2016). Alternative non-conventional techniques, such as the MAE approach, also known as green extraction technology, has recently escalated to address the aforementioned constraints. MAE technique partitioned analytes from several sample matrices by combining non-ionizing microwave and heat solvent energy by ionic conduction and dipole rotation (Wang et al. 2007; Mahmud et al. 2020). During MAE, microwave radiation causes the moisture inside cells to heat and evaporate, resulting in a massive rise in pressure on the cell walls. As a result, it ruptures plant tissue and releases the targeted compounds into the surrounding solvent. Microwave interaction between pectinesterase and pectic compounds elevates pectin extraction (Kute et al. 2015).

MAE has recently been employed to extract pectin from a wide range of citrus resources, including sweet lemon peel, orange peel, and pomelo peel (Liew et al. 2016; Rahmani et al. 2019; Kute et al. 2020). MAE improves both the capillary-porous components and the water absorption capacity of the cell wall (Maran and Prakash 2015). These changes provide the possibility of increasing the extraction yield of various analytes from the plant cell wall, including pectin, cellulose, and hemicellulose (Maran and Prakash 2015). Furthermore, the energy promotes temperature rise by inducing polar molecules to vibrate, enhancing extraction yield (Rodsamran et al. 2019). When compared to conventional methods, it protects thermolabile components and efficiently extracts the target bioactive compounds from raw materials in a short period of time (Mandal et al. 2007; Karbuz and Tugrul 2021).

There is no comprehensive study on microwave aided pectin extraction from sole albedo and flavedo sections. Thus, the current study aims to accelerate the extraction processes and yield higher quality pectin from both portions of the pomelo peels while avoiding potential thermal degradation through optimization using a Box-Behnken design (BBD) with the assistance of MAE, to characterize the extracted pectin and compare its potentiality with commercial pectin.

Material and Methods

Materials

Fully grown *Citrus maxima* fruits were collected from the premises of the Graphic Era (Deemed to be University), Dehradun, Uttarakand. All the analysis of this study was investigated using analytical grade chemicals which were procured from Hi-Media, India. The chemicals NaOH, HCl, H_2SO_4, Gallic acid, Na_2CO_3, $CuSO_4$, K_2SO_4, Boric acid, Folin–Ciocâlteu reagent etc. were purchased from local suppliers.

Sample Preparation

To eliminate surface contamination, the fruits were washed thrice with tap water. The flavedo and albedo sections were manually separated and dried in a hot air oven at 55 ± 5 °C for 6-8 h. Dried flavedo and albedo were grounded in mixer (Sujata, 900 W) and stored at 4°C in an airtight container.

Microwave Extraction of Pectin

For pectin extraction, a household microwave oven (Samsung, MC28H5025VK/TL4) was employed. Flavedo and albedo powder were placed in 500 mL separate beakers and extraction process was performed according to the parameters listed in Table S1. The extraction was carried out with deionized water as the solvent, and the pH was lowered to 2.0 using HCl. After the completion of extraction process, the mixture was allowed to cool at 28 ± 2 °C. After cooling, the mixture was separated to biomass and supernatant by centrifugation (25 min at 5000 rpm), the supernatant was precipitated using 100 % (v/v) ethyl alcohol, and the purified pectin was dried in a hot air oven at 50 °C to a constant weight. After cooling the pectin was kept in an airtight container till further analysis. The flow diagram (Fig. 1) represents the methodology of microwave assisted extraction of pectin.

Experimental Methodology

The experiments were conducted using Box Behnken (BBD) model of response surface methodology (RSM) with three process parameters, each with three levels including solid to solvent ratio (1:10, 1:15 and 1:20), time (80, 95 and 110s), and microwave power (300, 450, 600 watt) (Table S1). Total 17 experiments were performed and response i. e., yield (%) was measured. The experimental data was put into the second order model equation (Eq. 1).

$$Y = \beta_0 + \sum_{a=1}^{n} \beta_a X_a + \sum_{a=1}^{n-1} \sum_{b=a+1}^{n} \beta_{ab} X_a X_b + \sum_{a=1}^{n} \beta_{aa} X^2{}_a \quad (1)$$

Where,

Y = Dependent variable

β_0, β_a, β_{aa} *and* β_{ab} are coefficients

X_a and X_b denotes independent variables (Where, a = 1, 2……n and b = 1, 2….n)

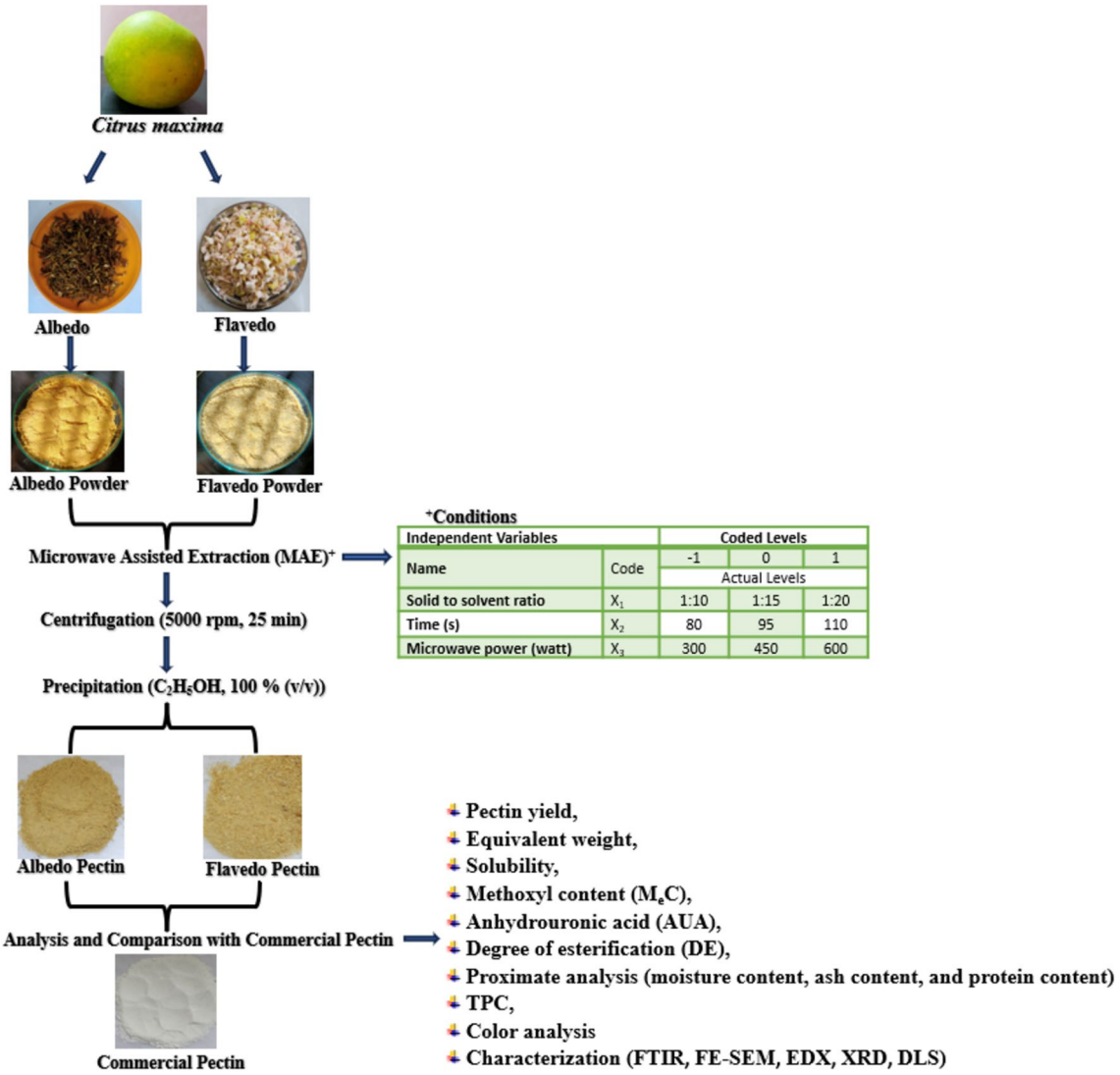


Fig. 1 Flow diagram of microwave assisted extraction of pectin

Quality Characteristics of Pectin

Pectin Yield

Rodsamran and Sothornvit (2019) method was adopted to estimate the pectin yield (%) using Eq (2):

$$\text{Pectin yield } (\%) = \frac{W_{dp}}{W_s} \times 100 \quad (2)$$

Where, W_{dp} = weight of dried pectin, W_s = Weight of sample

Equivalent weight (%) was calculated using Eq (3) given by Shubham et al. (2023)

$$\text{Equivalent weight } (\%) = \frac{W_s}{V_{a\times} N_a} \times 100 \quad (3)$$

Where, W_s = Weight of sample, V_a= mL of alkali solution, N_a = naoramilty of alkali solution

Solubility, Methoxyl Content (M_eC), Anhydrouronic acid (AUA) and Degree of Esterification (DE)

The modified technique reported by Wang et al. (2019) was adopted to measure pectin solubility (%). M_eC was determined by the method outlined by Zakaria et al. (2021). AUA was determined using the method provided by Mahmud et al. (2020), and DE was estimated using the method described by Zakaria et al. (2021), which included the use of M_eC and AUA. Eq 4, 5 and 6 were used to calculate M_eC, AUA and DE.

$$\text{Methoxyl content (\%MeC)} = \frac{V_{a\times}N_a \times M_w}{W_s} \times 100 \quad (4)$$

$$\text{AUA (\%)} = \frac{176 \times 0.1 \times 100\ (V_1 + V_2)}{W_s \times 1000} \quad (5)$$

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

Where, M_w = molecular weight of MeO, V_a= mL of alkali solution, N_a = naoramilty of alkali solution, V_1 & V_2 are the volume of alkali in equivalent weight and in methoxy content respectively and 176 is the molecular weight of AUA.

Proximate Analysis

Proximate analysis i.e., moisture content, ash content, and protein content of pectin was determined according to the standard procedure AACC (2002).

Total Phenolic Content (TPC)

TPC of pectin was determined using a modified technique developed by Kumar et al. (2022). In brief, 1 mL of ethanolic pectin extract was added to 0.5 mL Folin Ciocalteu (10%), 2.5 mL Na_2CO_3 (20 %), and vortexed for 1 min. The absorbance was measured at 725 nm using a UV Spectrophotometer (Shimadzu 1900) after 45 min of incubation in the dark. TPC (µg GAE/mg) was calculated using the Gallic Acid standard curve.

Color Analysis

The colour index for lightness (L), hue (a), chroma (b) was determined by the colorimeter (Colour Flex EZ, Hunter's colour lab, Virginia, USA). Total colour difference (ΔE), brown index (BI), yellow index (YI), white index (WI), Chroma (C) and hue angle (h) of pectin were determined according to Ataei and Hojjatoleslamy (2017).

Particle Size

Particle size analyzer (ZEN1690, Malvern Instruments Ltd) was used to determine the size of pectin particles.

FT-IR

PerkinElmer FT-IR spectrophotometer was used to analyze functional groups of pectin. The KBr pellet technique previously described by Jaiswal et al. (2021) was employed. Briefly, the powdered pectin sample mixed with potassium bromide powder (KBr) and pulverized to prepare pellets at high pressure. The FT-IR analysis of extracted pectin were measured in the range of range of 500-4000cm^{-1}.

FE-SEM Analysis

The morphology of extracted pectin was examined using a FE-SEM (Aperio 2 Thermofisher) at 10 kV operating voltage. Secondary electron (SE) imaging method was used to get the images.

X-ray Diffraction Spectra Analysis

The phase evaluations as well as structural properties of pectin like crystallinity were analyzed by the XRD (Rigaku X-ray diffractometer) pattern of pectin. In this study diffraction angle range was from 10° to 80°, and the scanning rate was 20/min.

Statistical Analysis and Model Validiation

The experimental data obtained in this study were analyzed for numerical optimization of the processing factors in terms of responses using Design Expert Software (Version 13.0.1). The analysis of variance was performed to determine the significance ($p < 0.05$) of model terms such as coefficient of determination (R^2), F-values, p-values, and lack of fit. To assess statistical significance ($p < 0.05$), the F value was utilized at numerous significance levels to estimate the statistical significance of all terms in the second order model Eq (1). R^2 should be more than 90% for a good fit model. To validate the results, the designed model was validated with optimized parameters. The (%) error was used to determine the fit of the design model using Eq. (7).

$$\text{Error (\%)} = \frac{(\textit{Experimental value} - \textit{Predicted value})}{\textit{Experimental value}} \times 100 \quad (7)$$

For physicochemical analysis, the experiments were carried out in triplets and results were indicated as mean ± SD.

Results and Discussion

Model Fitting and Experimental Data Analysis

The effect of process variable i.e., solid to solvent ratio (X_1), time (X_2), and microwave power (X_3) on Y_{AP} % and Y_{FP} % have been investigated and assessed in this study.

Table 1 shows the experimental findings of pectin yield (%) for various combinations of independent variables. Design Expert was used to compute analysis of variance (Table 2) for model fitting and validation of experimental data.

Impact of Independent Variables on Y_{AP} and Y_{FP}

Based on the results, the experimental data of Y_{AP} and Y_{FP} were ranged from 0.48 to 5.7 % and 0.69 to 3.3 %, respectively over the entire range of experimental condition (Table 1). Furthermore, according to the statistical analysis, the proposed model was appropriate and significantly fit ($p<0.05$) fitting all statistics ($R^2 = 0.95$; 0.96, $R^2_{adj} = 0.89$; 0.91) and met the intended model adequacy values in both Y_{AP} and Y_{FP}, respectively. There was also non-significant ($p>0.05$) lack of fit, which improved the model's reliability. To fit the empirical relationship shown by Equation (5) and (6), a second order polynomial model were attempted:

$$Y_{AP}(\%) = 5.34 - 0.2562\,X_1 - 0.1287X_2 - 0.0250\,X_3 + 0.02075\,X_1X_2 - 0.86\,X_1X_3 + 1.60\,X_2X_3 - 1.27\,X_1^{\,2} - 1.59\,X_2^{\,2} - 1.56\,X_3^{\,2} \quad (5)$$

$$Y_{FP}(\%) = 3.12 + 0.2487\,X_1 - 0.0237\,X_2 + 0.2250\,X_3 + 0.1325\,X_1X_2 - 0.3750\,X_1X_3 + 0.0750\,X_2X_3 - 0.8513\,X_1^{\,2} - 1.30\,X_2^{\,2} - 0.0938\,X_3^{\,2} \quad (6)$$

Table 2 ANOVA for the fitting and validation of experimental data to the model

Factors	Responses	
	Y_{AP} (%)	Y_{FP} (%)
Model F-Value	15.53[a]	19.55[a]
Model p- Value	0.0008	0.0004
Lack of Fit	2.77[NS]	6.17[NS]
Std. Dev.	0.5672	0.2661
Mean	3.26	2.06
C.V. (%)	17.41	12.90
R^2	0.95	0.96
Adjusted R^2	0.89	0.91

*NS: insignificant; C.V.: coefficient of variation

From Eq. (5) & (6), high value of coefficient has a greater effect on the Y_{AP} and Y_{FP}. In Eq. (5), interactive term (X_1X_2, X_2X_3) showed a positive effect whereas linear terms (X_1, X_2 and X_3), interactive term (X_1X_3) and quadratic terms (X_1^2, X_2^2 and X_3^2) showed a negative effect on Y_{AP} (Table S2). From Eq. (6), it was observed that linear terms (X_1 and X_3), interactive terms (X_1X_2 and X_2X_3) showed a positive effect whereas linear term (X_2), interactive term (X_1X_3) and quadratic terms (X_1^2, X_2^2 and X_3^2) had a negative impact on Y_{FP} (Table S2). According to the findings, the positive coefficient of independent factors implies that as its level increased, Y_{AP} and Y_{FP}

Table 1 Experimental data of Y_{AP} and Y_{FP}

Std	Independent Variables			Responses	
	Solid to solvent ratio	Time (s)	Microwave power (watt)	Albedo Pectin (A_P) Y_{AP} (%)	Flavedo Pectin (F_P) Y_{FP} (%)
1	10	80	450	2.68	0.8
2	20	80	450	2.32	0.98
3	10	110	450	2.21	0.69*
4	20	110	450	2.68	1.4
5	10	95	300	1.9	1.1
6	20	95	300	2.54	2.4
7	10	95	600	4.2	2.7
8	20	95	600	1.4	2.5
9	15	80	300	4.36	1.9
10	15	110	300	0.7	1.5
11	15	80	600	0.48*	1.8
12	15	110	600	3.22	1.7
13	15	95	450	5.7**	3.3**
14	15	95	450	5.5	3.1
15	15	95	450	5.5	2.9
16	15	95	450	5.4	3.1
17	15	95	450	4.6	3.2

X_1: solid to solvent ratio; X_2: time; X_3: microwave power; *,** represents minimum and maximum values, respectively.

also increased, but the negative coefficient shows that Y_{AP} and Y_{FP} declined dramatically as independent variables increased.

To visualize the impact of independent variables on Y_{AP} and Y_{FP} response surface (3D) plot (Fig. 1 a, b & c) and (Fig. 2 a, b & c) were developed. Fig 1 (a) – (c) and Fig. 2 (a) – (c) depicted the impact of solid to solvent ratio (1:15), time (95s) and power (450 watt) on YAP and Y_{FP} at central points. Attributing to Fig. 1 (a) & (b) and Fig. 2 (a) & (b), Y_{AP} and Y_{FP} increased significantly from low solid to solvent ratio (1:10) to maximum value at central point (1:15) and further decreased as solid to solvent ratio increased (1:20). The addition of solvent may effectively absorb microwave energy by albedo and flavedo matrices, leading to swelling of cells and breaking of cell walls, which is beneficial to increasing the contact surface area between both the matrix and the solvent, resulting in an increase in pectin extraction yield to solid to solvent ratio of 1:15 (Maran et al. 2015; Zakaria et al. 2023). Higher solvent concentrations, on the other hand, would reduce microwave adsorption of material since the solvent absorbed more energy. As a result, the break of the material cell wall and mass transmission may be adversely impacted, resulting in a drop in pectin yield (Li et al. 2010). Our findings were consistent with the results reported by Shams et al. (2020) and Su et al. (2019), who investigated the MAE of pectin from faba bean dried hull and orange peel and found a reduction in pectin production when the solid to solvent ratio was high.

From Fig. 2 (a) & (c) and Fig. 3 (a) & (c) it was observed that Y_{AP} and Y_{FP} increased significantly from shorter exposure time (80 s) to maximum value at central point (95 s) and further decreased as exposure time increased (110 s). The entire process explains the phenomenon of thermal accretion into the mixture as a result the absorption of microwave energy, which increased pectin dissolution into solid and solvent mixture till 95s (central point) and subsequently reduced the Y_{AP} and Y_{FP} to 110 s. This finding is in close agreement with Maran and Prakash (2015); Shams et al. (2020) and Zakaria et al. (2021) who reported that longer exposure time may cause breakdown of pectin molecules thus affecting pectin extraction rate.

Figures 2 (b) & (c) and Fig. 3 (b) & (c) depicts that Y_{AP} and Y_{FP} increased significantly from less microwave power (300 watt) to maximum value at central point (450 watt) and further decreased as the microwave power increased (600 watt). The entire process explicates the phenomenon of polar solvent penetration that can efficiently absorb microwave power causing efficient heating and thereby increasing the solvent penetration rate into solid matrix, resulting in effective transport of materials via molecular interaction with the electromagnetic field and therefore offering a rapid transfer of energy to the solid-solvent matrix, allowing the extraction of components (Yan et al. 2010; Latorre et al. 2013). Furthermore, microwave power also accelerates cell rupture by triggering a sudden temperature and internal pressure rise within the sample, promoting the destruction of the sample surface which in turn

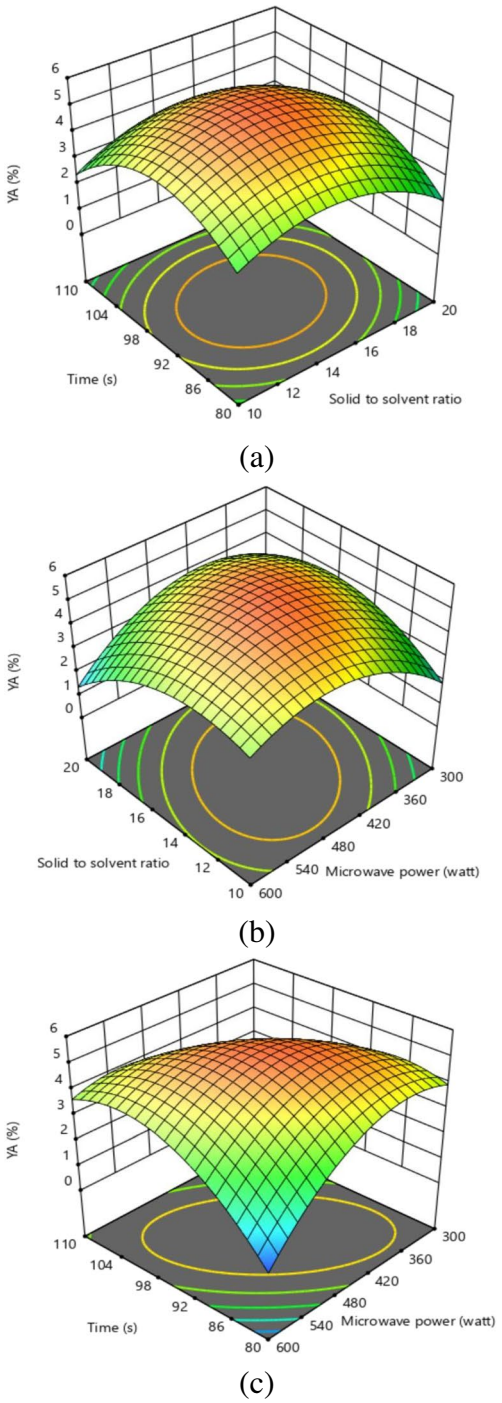


Fig. 2 3D response influence on Y_{AP} **(a)** time and solid to solvent ratio **(b)** solid-to-solvent ratio and microwave power; **(c)** time and microwave power

results in the exudation of pectin within the plant cells into the surrounding solvents (Zhang et al. 2008) and thus increasing the extraction yield. Intense microwave power, on the other hand, may reduce Y_{AP} and $_{YFP}$ due to thermal breakdown of the released pectin. The results of this study are consistent with those of Zakaria et al. (2021) and Zakaria et al. (2023),

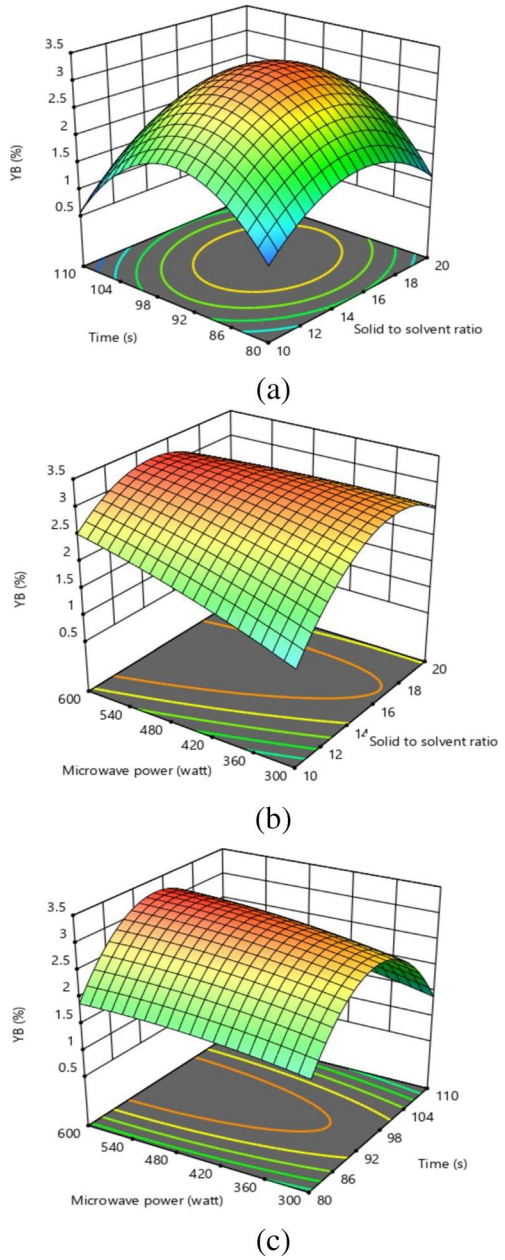


Fig. 3 3D response influence on Y_{FP} **(a)** time and solid to solvent ratio **(b)** solid to solvent ratio and microwave power **(c)** time and microwave power

who investigated microwave aided extraction of pectin from pineapple peel and ananas comosus peel and concluded that microwave power ranging from 450 watt to 550 watt is best for pectin extraction.

Numerical Optimization and Validation of Microwave Assisted Extraction Process Conditions

Numerical optimization was performed using design expert software (ver. 13.0.1) to achieve the highest Y_{AP} and Y_{FP}. The constraints selected to the independent variables and response for optimization has been provided in Table S3. Based on statistical analysis of experimental data, independent variables that provided the desired Y_{AP} and Y_{FP} were solid to solvent ratio (1:15), time (95s) and power (450 watt) respectively. Table S3 depicts that the predicted and experimental values are in reasonable agreement with non-significant difference and low error % between the means.

The graphical representation of predicted and actual values of Y_{AP} and Y_{FP} are shown in Fig. S1 (a) & (c), and it can be easily observed that actual values of Y_{AP} and Y_{FP} are adjacently merged in predicted values of Y_{AP} and Y_{FP}. Fig. S1 (b) & (d) represents that all the experimental data selected were placed within the permissible limits (-2 to +2), showing that the generated model is a good fit for the experiments (Saikumar et al. 2023).

Physico-Chemical Properties of Extracted Pectin from A_P and F_P at Optimum Condition

The moisture content (MC) of pectin recovered from MAE of the C_P (6.96 %) was greater than that of the A_P (4.8 %), and the F_P (3.53 %) (Table 3). The MC of C_P, A_P, and F_P were within the IPPA (2002) with a permitted limit of less than 12 %. The MC of A_P and F_P lies within the range (5.2 % - 7.6 %)

Table 3 Physico-chemical properties of C_P, A_P, and F_P

Parameters	C_P	A_P	F_P
M.C. (%)	6.96 ± 0.124	4.80 ± 0.244	3.53 ± 0.286
Ash (%)	1.38 ± 0.012	3.41 ± 0.026	2.22 ± 0.033
Protein (%)	10.6 ± 0.216	11.36 ± 0.032	5.76 ± 0.089
Equivalent weight	92.31 ± 0.316	294.08 ± 0.063	81.64 ± 0.549
Methoxyl Content	8.93 ± 0.055	8.50 ± 0.012	11.40 ± 0.081
AUA (%)	70.38 ± 0.439	66.32 ± 0.153	86.31 ± 0.070
DE	72.03 ± 0.026	72.76 ± 0.074	74.98 ± 0.082
TPC µg GAE/mg	12.04 ± 0.095	40.33 ± 0.065	30.69 ± 0.322
Hot water Solubility	Soluble	Soluble	Slightly soluble
Cold water Solubility	Soluble	Slightly soluble	Slightly soluble
Color Index			
L	67.11 ± 0.158	56.70 ± 0.481	49.20 ± 0.231
a	-4.30 ± 0.094	2.10 ± 0.124	1.4 ± 0.036
b	6.30 ± 0.081	22.40 ± 0.205	22.50 ± 0.126
ΔE	0	19.29 ± 0.042	24.30 ± 0.069
H	-44.18 ± 0.020	55.90 ± 0.011	56.76 ± 0.032
YI	13.41 ± 0.012	56.43 ± 0.013	65.04 ± 0.041
BI	4.82 ± 0.038	51.65 ± 0.124	60.81 ± 0.132
WI	66.22 ± 0.133	51.20 ± 0.201	44.47 ± 0.156
Chroma	7.62 ± 0.026	22.49 ± 0.017	22.42 ± 0.019

of the MC of citrus peel pectin (Devi et al. 2014). Dananjaya et al. (2020) and Raftani Amiri et al. 2023 reported 10.35 % and 9.21 % MC in purified pectin obtained from pomelo peel and pomegranate peel respectively which were higher than the moisture content of C_P, A_P and F_P. This research demonstrated that pectin from MAE had lower water holding capacity than that extracted by traditional extraction, which is preferable for safe storage and prevents deterioration of pectin quality owing to pectinase generation as a result of microorganism growth (Ismail et al. 2012; Koh et al. 2014).

Ash content in pectin varies with different maturity index of different samples. The ash content of the extracted pectin was found to be within the permissible limit (< 10 %) suggested by IPPA (2002) i.e., C_P (1.38 %) followed by F_P (2.22 %) and C_P (3.41 %), thereby more supportive for gel formation (Begum et al. 2014) and thus might be indicative of pre-mature samples. When the above-mentioned results were compared to other studies done on conventional extraction, the ash content of extracted pectin was much higher than that of MAE (Koh et al. 2014). The ash level of pomelo peel pectin extracted with ethanol and water was 3.56% and 4.14%, respectively (Dananjaya et al. 2020; Sotanaphun et al. 2012). Sönmez and Giray (2011) postulated that the low ash content was due to the short extraction duration of MAE for pectin extraction. In another study Raftani Amiri et al. 2023 reported 3.30 % ash content in pomegranate peel pectin which is higher than C_P and F_P while almost near to the ash content of A_P of the present study.

Citrus pectin's emulsifying powers are dependent on protein content. Since protein is amphipathic, it may improve the emulsifying properties of citrus pectin by binding to the water-oil interface and decreasing interfacial tension (Chen et al. 2018). According to Table 3, A_P has superior emulsifying properties due to its high protein content (11.36%) when compared to C_P (10.6 %) and FP (5.76 %). In this study, the protein concentration of A_P, C_P, and F_P was greater than the citrus pectin protein (3.67 %) reported by Chen et al. (2018) and eggplant peel pectin (2.53 %) reported by Kazemi et al. (2019).

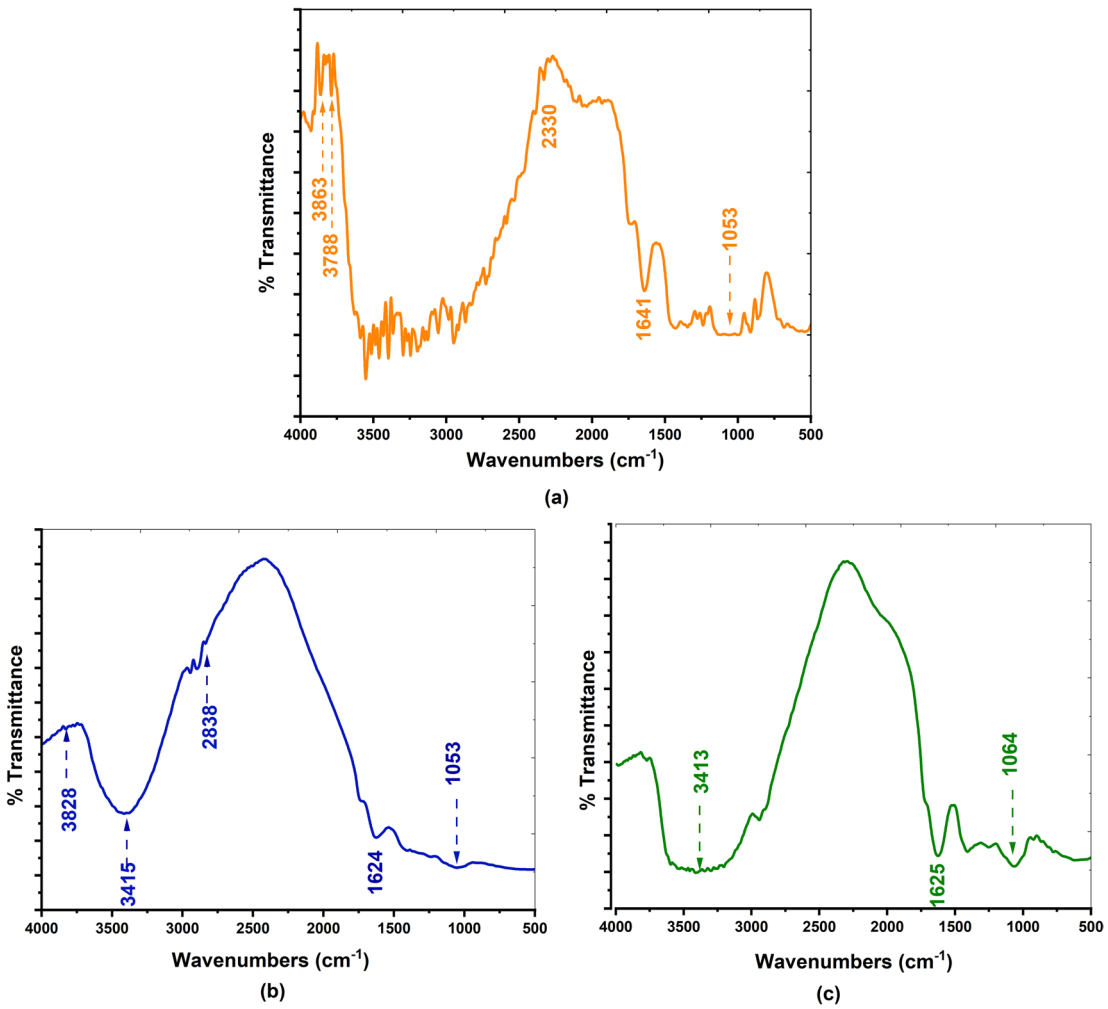
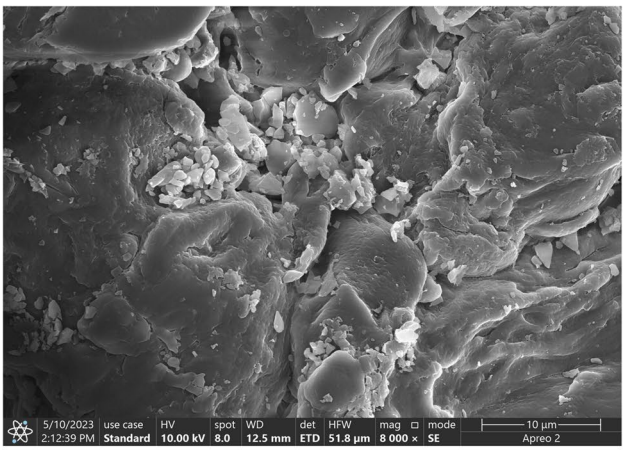
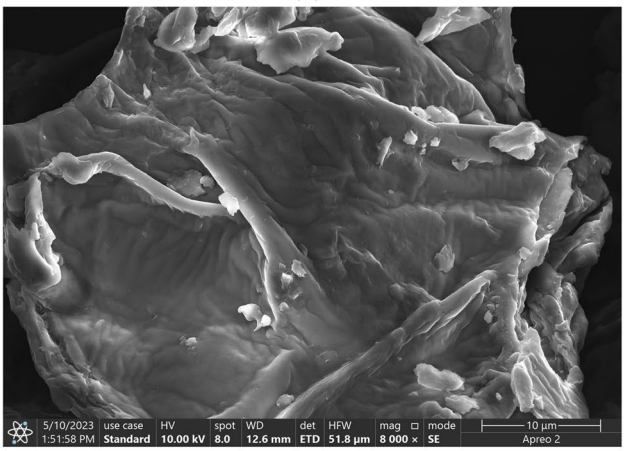


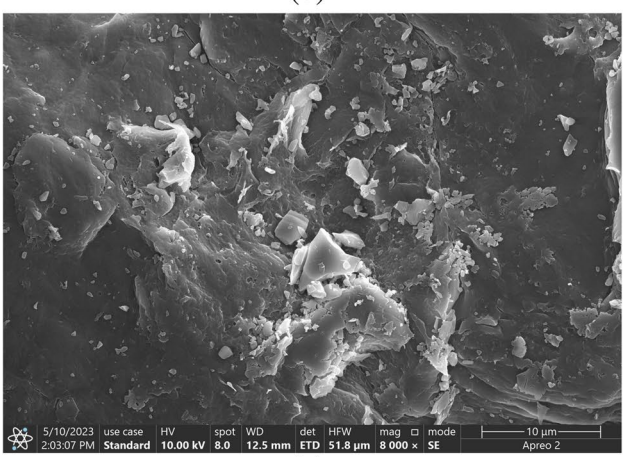
Fig. 4 FT-IR spectrum **(a)** C_P **(b)** A_P and **(c)** F_P



(a)



(b)



(c)

Fig. 5 FE-SEM micrograph of **(a)** C_P **(b)** A_P and **(c)** F_P

Equivalent weight of pectin depends on method of extraction and raw material used (Shams et al. 2020). The highest equivalent weight was noted for A_P (294.08) while lowest for F_P (81.64) (Table 3). High equivalent weight represents the higher gel forming capacity of pectin. In a previous study conducted by Dananjaya et al. (2020) reported higher equivalent weight (1245.26) of purified pomelo pectin.

Pectin is classified into two types based on DE: low methoxyl pectin (DE 50%) and high methoxyl pectin (DE > 50%). Methoxy content is a significant measure in defining gel capacity and influencing pectin setting time, and it varies between 0.2 % and 12 % depending on the technique of extraction and raw material utilized (Huang et al. 2014). Pectin influences the emulsifying, texturing, and gelling qualities of foods (Mellinas et al. 2020; Spinei and Oroian 2022). The esterification level in F_P is much greater than in A_P and C_P (Table 3). The methoxy content value in this investigation also depicted the similar results (Table 3), which might be attributable to the pre-mature samples carrying high methoxyl pectin class (> 7%). Because of the high DE value, the recovered pectin in this investigation represents under high methoxyl pectin (Table 3), which is closely linked to findings of Wang et al. (2015), Quoc et al. (2015), and Van Hung et al. (2021). These methoxy content findings were comparable to those found for mango peel (7.33%), banana peel (7.03%), pomelo peel (8.57%), lime (9.92%), passion (8.81%-9.61%), and dragon fruit pectin (2.98% to 4.34%) (Ismail et al. 2012). The methoxy content of pectin increases its spreading quality and sugar binding ability (Madhav and Pushpalatha, 2002). The value of AUA (%) determines the purity of pectin. According to AOAC (1995), the AUA% of pectin should be greater than 65 %. The AUA value for C_P, A_P, and F_P in this study is higher than the recommended value. Our findings are consistent with prior research by Sotanaphun et al. (2012), Kanmani et al. (2014), and Dananjaya et al. (2020), who reported 74.12 %, 85.57 %, and 68.27 % AUA in pomelo peel pectin, respectively.

TPC was found to be highest in A_P, followed by F_P and C_P (Table 3) than pectin extracted from similar peel waste using ultrasonic assisted extraction (Wang et al. 2015).

Color Analysis

Pectin colour has a significant impact on the presentation of gel formed (Kute et al. 2020). Color index of C_P, A_P, and F_P are tabulated in Table 3. The highest lightness value was observed in C_P (67.11) and lowest for F_P (49.20). It was observed that C_P has lighter than A_P, and F_P in color. The negative value of a in C_P and positive values in A_P, and F_P indicated that C_P, A_P, and F_P were characterized by greenness and redness respectively. The positive b value is highest for F_P and lowest for C_P, which represents that F_P and A_P shows more yellowness compared to C_P which is confirmed by highest YI for F_P followed by A_P and C_P. The highest ΔE was noted for F_P and lowest for A_P compared to C_P. Highest WI and BI were observed in C_P and F_P respectively. The brown color of F_P may be

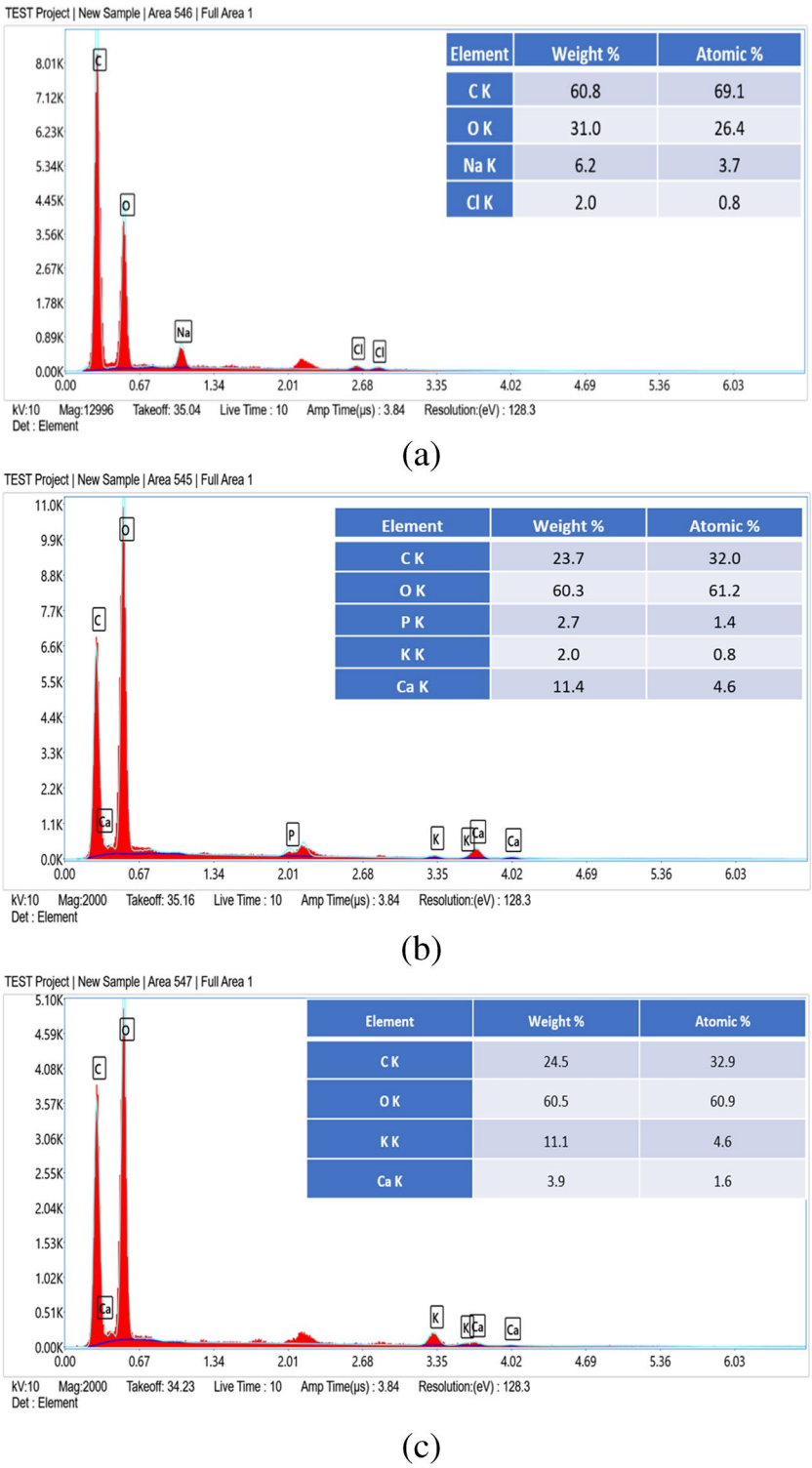


Fig. 6 EDX analysis of **(a)** C_P **(b)** A_P and **(c)** F_P

attributed to the presence of carotenoids (Kute et al. 2020). Among C_P, A_P, and F_P, the highest chroma and h value was predicted in A_P and F_P, respectively. The comparable color propensity was reported for apple peel pectin (Zheng et al. 2021) and lime peel pectin (Rodsamran and Sothornvit 2019).

Characterization of C_P, A_P, and F_P

FT-IR Analysis

The absorption band arises in the 3828 cm^{-1} and 3863 cm^{-1} regions for A_P and C_P, respectively correlates to O-H group

stretching (Fig. 4). The peak depicted at 3415 cm^{-1} is attributed to the C-H stretching of the CH_2 group (Pasandide et al. 2017). The peak emerging at 2838 cm^{-1} corresponds to bending vibration of O-H bond. Peak observed at 1624 cm-1, 1625 cm^{-1} and 1641 cm^{-1} for A_P, F_P, and C_P, respectively is most likely caused by C=O stretching vibrations of the free (non-methyl-esterified) carboxyl group (Hosseini et al. 2019; Mamiru and Gonfa 2023). AP has a peak at 1053 cm^{-1}, FP has a peak at 1064 cm^{-1}, and 1057 cm^{-1} for C_P confirms the presence of polysaccharide in all the pectin (Lasunon and Sengkhamparn 2022).

FE-SEM/EDX Analysis

The FE-SEM micrograph of the C_P, A_P and F_P is depicted in the Fig. 5. The morphological analysis of the isolated pectin was done using FE-SEM micrograph analysis. The FE- SEM images of both A_P and F_P show flat surfaces with some roughness and wrinkles on the flat surface whereas C_P has more roughness and wrinkles on the flat surface. Similar results were reported by Kazemi et al. 2020 and Raftani Amiri et al. 2023 who stated that microwave and ultrasound assisted pectin has smooth surface.

The elemental composition of C_P, A_P, and F_P is determined by EDX analysis (Fig. 6). EDX results revealed that carbon % in C_P is substantially greater than in A_P and F_P, but oxygen % in A_P and F_P is higher than in C_P. Furthermore, C_P has a trace of sodium, whereas A_P and F_P have a trace of calcium. Our findings are in reasonable agreements with Salima et al. (2022) who also reported high oxygen content in pumpkin pectin.

XRD Analysis

The XRD pattern employed to evaluate the type (crystalline or amorphous) of C_P and extracted A_P and F_P under ideal conditions (Fig. 7). The XRD pattern of C_P shows crystalline structure, whereas the A_P and F_P XRD spectrum suggests

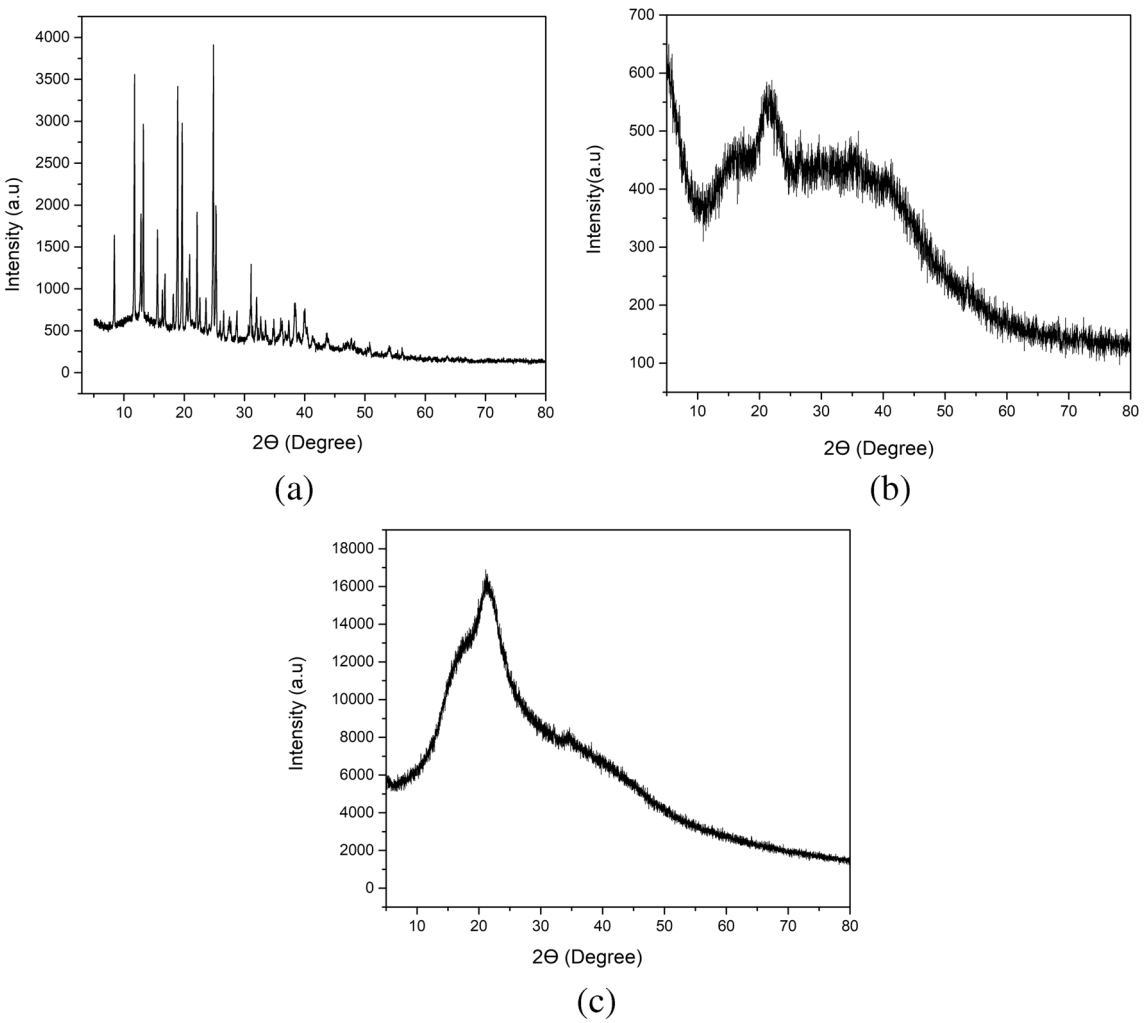


Fig. 7 XRD analysis **(a)** C_P **(b)** A_P **(c)** F_P

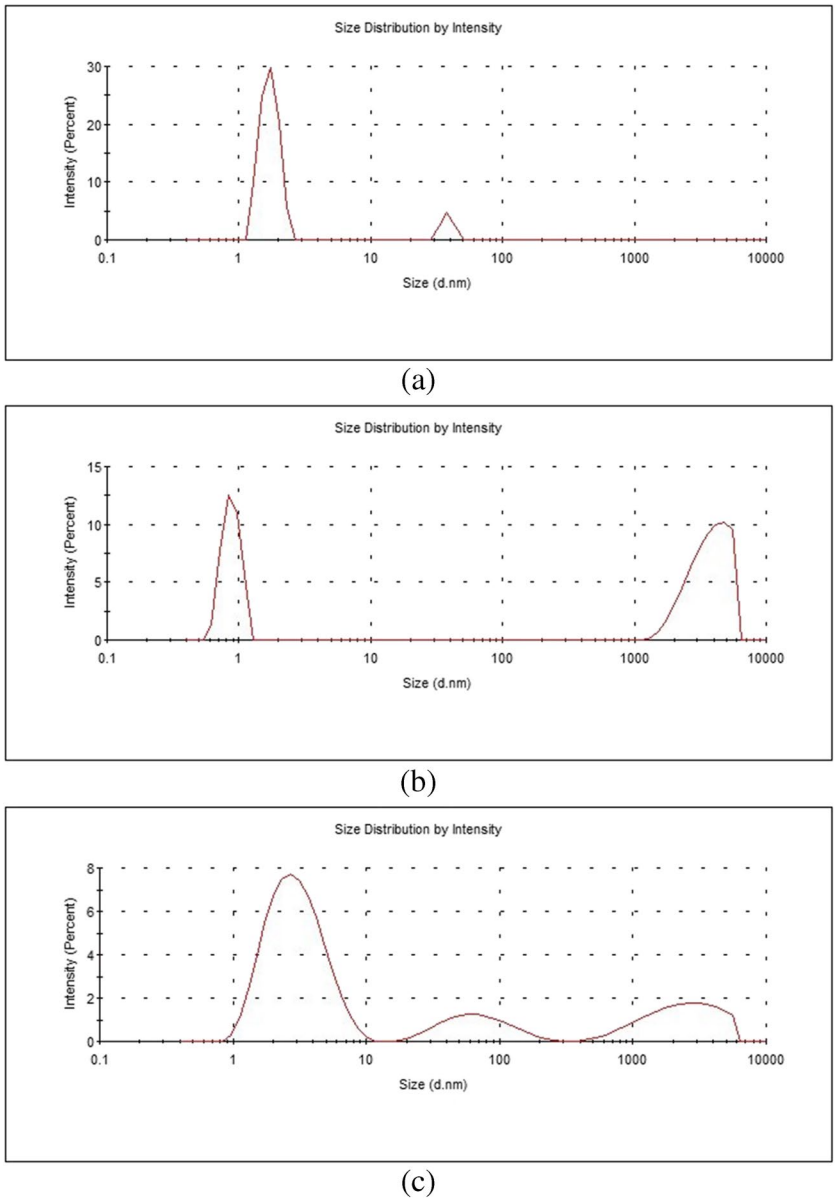


Fig. 8 DLS spectra of **(a)** C_P **(b)** A_P and **(c)** F_P

amorphous nature. The results of this investigation matched the XRD spectrum of sour orange peel pectin (Hosseini et al. 2019), pomelo peel pectin (Dananjaya et al. 2020), watermelon rind pectin (Mamiru and Gonfa 2023) and pomegranate peel pectin (Raftani Amiri et al. 2023).

Particle Size Analysis

Particle size is critical in food component compositions, material handling, and processing. The rate of diffusion is related to particle size (Rivadeneira et al. 2020). DLS was used to compute the average particle size of the C_P, A_P, and F_P. Figure 8 depicts the spectra of the DLS investigation. The average particle size of A_P is 0.95 nm, whereas some particles have an average particle size of 1200 nm. The particle size of the A_P was found to be substantially smaller than that of the C_P within the range of 8 nm, with some particles reaching a size of 85 nm. Furthermore, the size of F_P was found to be around the 14 nm. The average particle size of the F_P has been found to be variable.

Conclusions

Extraction of pectin from A_P and F_P portion of Citrus *maxima* using Box- Behnken design with three independent variables i.e., solid to solvent ratio, time and power were performed through green microwave extraction technology (MAE). The optimal conditions responsible for higher Y_{AP} % and Y_{FP} % were solid to solvent ratio (1:15), time (95 s) and

power (450 watt). Experimental values of Y_{AP} (5.57 %) and Y_{FP} (3.09 %) were observed at optimal values. The results of physicochemical properties, color, FT-IR, FE-SEM/EDX, XRD, and particle size analysis showed that A_P and F_P have similar properties of commercial grade pectin. From the results it could be concluded that high quality pectin from albedo (A_P) and flavedo (F_P) portions of Citrus *maxima* can be extracted using green microwave extraction technology in short processing time.

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Author contributions All authors contributed to the study conception and design. Investigation and project administration was carried out by S.A., P.K. and A.H. Conceptualization and Supervision was carried out by S.K. and V. K. The first draft of the manuscript was written by S. K. Methodology and analysis by W.A., and S.A. Review and editing were performed by A. D., R.M., B.B. and S. U. and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Data availability All the data is available in the manuscript.

Declarations

Ethical statement Not Applicable

Competing interests The authors declare no competing interests.

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