



## Pulsed electric field combined with microwave-assisted extraction of pectin polysaccharide from jackfruit waste

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### ABSTRACT

The present methodology uses the combination of pulsed electric fields (PEF) and microwave-assisted extraction (MAE) of pectin from the jackfruit wastes. Two optimization tools such as Box-Behnken design (BBD) and artificial neural network (ANN) were used to study the extraction yields. The optimized operating conditions obtained after desirability analysis were PEF strength (11.99 kV/cm), PEF treatment time (5.47 min), followed by MAE at power density (647.30 W/g) and time of exposure (5.00 min). From the optimization results, the  $R^2$  values of BBD ranged from 0.89 to 0.98 as well as the SSE (sum of squared error) values varied across 0.076 to 0.781 whereas  $R^2$  values of ANN fluctuated around 0.95 to 0.99 and MSE (mean squared error) values varied from 0.008 to 0.1. It was observed the ANN was found to be more superior in execution than the BBD model. The extracted pectin was analyzed for structural and functional properties with the comparison to conventionally extracted pectin. The degree of esterification and methoxyl percentage of PEF and MAE pectin was less than the conventionally extracted pectin. The experimental and predicted values were similar for the pectin yield (%), but a higher prediction rate was observed in ANN modelling than BBD. Scanning Electron Micrographs showed an increased rupture and severing of parenchymal cells attributing to enhance extraction yields. Therefore, it can be concluded that the pulsed electric field treatment followed by microwave-assisted extraction resulted in higher pectin extraction with enhanced functional properties than the conventional method.

### 1. Introduction

Jackfruit (*Artocarpus heterophyllus*), associated with the mulberry family (*Moraceae*), is believed to have originated from southwestern tropical Apennines of India (Boning, 2006). Jackfruit is found to have a good amount of minerals such as potassium, magnesium, calcium, and iron, along with vitamins C, A, and B<sub>6</sub> (Ranasinghe, Maduwanthi, & Marapana, 2019). These inedible portions comprise interior perigones, central core, and outer rinds are unutilized and generally discarded as waste. Jackfruit processing industry and vendors normally dispose of these into fields, thereby sparking environmental nuisances. Pectin is a polysaccharide having hydro colloidal, stabilizing, and gelling characteristics. It is widely utilized to processing of food products, cosmetics,

pharmaceuticals, and other goods (Guerrero, Suárez, & Orozco, 2017). At present, pectin stays as an essential raw material, especially for the food processing sector and medicinal industries (Quoc et al., 2015). Pectin was extensively utilized in the form constituent in edible and biodegradable films due to its availability, low cost, and easy processing (Bykov, Makarova, Demidova, & Eremeeva, 2017). Pectin is such a naturally occurring biopolymer that it has been extensively recognized in the food industries and biotechnology (Rehman et al., 2019).

Pectin and pectin substances are present in plant cells' primary cell walls, especially across the middle lamella of plant tissues. The primary monomeric element of the complicated pectin molecule framework exists in D-galacturonic acid (GalA), its oxidation with sugar to form a D-galactose. This GalA element being connected with 1/4 galacturonosyl

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branches gets disrupted through side-chains bearing L-rhamnose elements, disrupting the linear conformation of poly-(Gal A) chain. Specific carboxyl groups among these poly-(GalA) chains get interesterified through methyl clusters which cause deviation within the degree of methyl esterification (DE or DM). Depending on the percentage of DE (greater or less than 50%), pectin is classified as high methoxylpectins (HMP) or low methoxylpectins (LMP). Homogalacturonans (HG), rhamnogalacturonan-I (RG-I), and substituted galacturonans (SG or RG-II) are the three general primary classifications of pectin (Ridley, O'Neill, & Mohnen, 2001). Pectin has a key food constituent utilized for thickening, emulsifying, and gumming factors in processed foods. Pectin is a lineage complicated polysaccharide having 1, 4-linked  $\alpha$ -D galactosyluronic remnants. The physicochemical properties and structural bodies of pectin rely upon the source and method of extraction. Pectin isolation depends on several factors (Pinelo, Sineiro, & Núñez, 2006) such as hydrolysis, proto-pectin, and solubilization. Conventional extraction method includes direct boiling using acidified water, which is time-consuming and degrades quality pectin. The application of combined novel technologies might help in conquering the inadequacies of conventional methods.

In recent years, several authors used different advanced techniques for the extraction of pectin polysaccharide from fruits and vegetables such as microwave-assisted extraction (Li, Han, Zou, & Zhang, 2013; Zarei et al., 2017; Misra & Yadav, 2020), ultrasound-assisted extraction (Minjares-Fuentes et al., 2014; Misra & Yadav, 2020), vacuum stem pulsed blanching (VSPB) (Wang et al., 2021), ohmic heating (Saberian, Hamidi-Esfahani, Gavlighi, & Barzegar, 2017), pulsed electric field and mild thermal processing (Moens, De Laet, Van Wambeke, Van Loey, & Hendrickx, 2020), radio frequency assisted extraction (Zheng et al., 2021), and the impact of ozone (Lee, Ting, & Thoo, 2021) and high hydrostatic pressure (Arachchige, Mu, & Ma, 2020) on pectin extraction. Advance methods result defined that maximum production for pectin with minimum time as against to conventional extraction. The application of non-thermal technologies generated internal heat, which heats moisture leading to evaporation causing tremendous pressure on the cell wall. This leads to swelling and rupture of plant cells resulting in leaching out phytoconstituents in the cell, aiding in extraction and reducing the period for extraction (Misra & Yadav, 2020; Srinivas, Mathew, Kothakota, Sagarika, & Pandiselvam, 2020). Microwave extraction reduces extraction time and energy consumption due to its more rapid and homogeneous action. Considerable pressure builds up in the material, which modifies tissue material properties, breaking down the cell structure and increasing tissue porosity, thus allowing better penetration of extracting solvents and increasing pectin yield (Dao, Webb, & Malherbe, 2021; Karbuz & Tugrul, 2021). Similarly, the pulsed electric field generated heat due to internal friction, and the accumulation of charged particles across the cell wall resulted in cell lysis due to electroporation. This could increase the pectin yield (Kumar, Patel, & Kumar, 2015; Moens, Huang, Van Loey, & Hendrickx, 2021). The efficiency of the PEF processing relies on treatment conditions (electric field strength, processing time, specific energy, pulse width and shape, frequency and temperature) in addition to product (cell size and shape, orientation in the electric field, conductivity) as well as medium properties (conductivity, composition, pH) (Moens et al., 2020; Puértolas, Luengo, Álvarez, & Raso, 2012). The pulse electric field process can transfer of subcellular contents, namely ions to the cell wall, the site of Pectin methyl esterase performance. Consequently, in a decrease of degree of methylation, an strengthen of ionic pectin crosslinking can assist PME activity. These all factors favor enhancing the pectin extraction yield (Jin et al., 2020; Ma & Wang, 2013; Novickij et al., 2020). In combined extraction methods the obtained pectin has similar chemical structures and a high degree of methoxylation (Karbuz & Tugrul, 2021). It could be hypothesized from the above statement that PEF pre-treatment followed by microwave-assisted extraction could enhance the pectin yield. For higher extraction yields, Box-Bhenkan (BBD) and ANN (artificial neural network) models were used to

optimize the operation parameters like applied PEF field strength, PEF treatment time, microwave power density, and applied time. These techniques can adapt numerous variable non-linear interactions over-determined interpretations, possess improve abstraction abilities, comes to indulgence to interference as well as lack of data, and can gain with observational data (Kothakota, Thimmaiah, Yadav, & Panday, 2013; Srikanth et al., 2020; Srinivas et al., 2020). There are no investigations on the extraction of pectin from jackfruit wastes by PEF treatment followed by microwave-assisted extraction. Therefore, the present study aims to apply PEF treatment for the enhanced pectin extraction from jackfruit wastes such as rind and core.

## 2. Materials and methods

### 2.1. Preparation of jackfruit waste for pectin extraction

Jackfruits (*Varikka* variety) were procured from the farm of Kerala Agricultural University, Thrissur, Kerala (India). The core (axis) and inner perigones of Jackfruit were detached and engrossed off one by one, followed by boiling in water at 90 °C for 5 min for enzyme inactivation and conventional hot air drying at 60 °C. The dried jackfruit waste was powder using a pulverizer and sieved over an 80-mesh screen. The sieved powder was vacuum packaged and stored in desiccators until further use.

### 2.2. Experimental setup

The description of the PEF reactor used for the experiments is depicted in Fig. 1. The reactor consists of an outer protective chamber, inlet system, pulsed generating system, treatment chamber, display unit, cooling system, and treated sample outlet. Aluminum (10 gauge) and stainless steel (1-in. food grade) have been utilized for a high insulation outer casing, consisting of a display and control unit for adjusting electric field strength, pulsed frequency, and power parameters. The 5 L capacity stainless steel inlet system is connected to the treatment section via a stainless-steel pipe (2 cm) and a flow control valve, which regulates the feed rate towards the chamber. It has a pulse generating system to produce voltage ranging from 5 to 20 kV with an input requirement of 230 V, 12 W power, and a 5 A current. The main PEF processing chamber consists of one Teflon cylindrical tube beneath and topmost electrodes at a distance of 0.05 m and additionally having two flow control valves. Valve 1 is opened, and valve 2 is closed indicates a feed towards the chamber, whereas valve two alone is opened directed the flow outward the section. The display unit on top of the PEF cell shows generated pulses' frequency and electric field strength. Two cooling units (cooling fan and bath) are provided in the PEF cell to prevent pectin degradation from sudden exposure to high temperature, activated by a microcontroller. Finally, PEF treated plant material was collected at the outlet system.

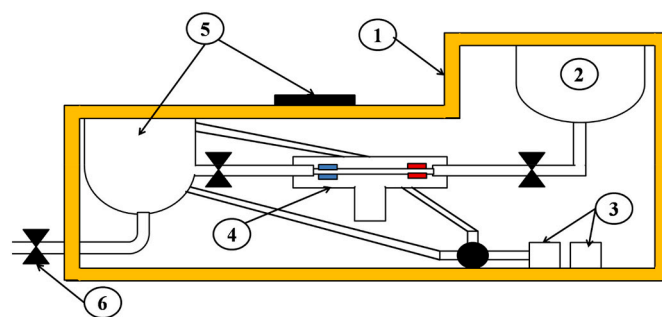


Fig. 1. Schematic view of PEF pre-treatment chamber: 1. Outer protective chamber 2. Inlet system 3. Pulse generating system 4. Treatment chamber 5. Cooling system 6. Treated sample outlet.

2.3. Extraction of pectin from jackfruit rind and core

The final product has extracted from jackfruit waste using PEF, microwave (MW), and conventional methods (CH) were indicated in diagrammatic flow chart analysis (Fig. 2). An extraction circumstance has been maximized as a function of initial studies as well as available literatures (Jafari, Khodaiyan, Kiani, & Hosseini, 2017; Misra & Yadav, 2020) have led to pH 2, 1:20 W/V solid-to-solvent ratio (pomace: citric acid water) in favor of eviction, 1:4 V/V liquid-to-liquid ratio (pectin extract: ethanol) as acceleration towards produce utmost pectin and low energy consumption during extraction of jackfruit waste powder.

2.3.1. Conventional heating (CH)

Jackfruit powder was dissolved in 200 mL of 1% citric acid solution of which has pH 2. The solution is then kept on a magnetic stirrer with a heating mechanism (VELP Scientific, Italy) at a temperature of 80 °C for 45 min by following a procedure given by Misra and Yadav (2020). The extract was centrifuged at 6000 rpm at 15 °C for 30 min. The accelerated pectin has been cleansed again over 80% ethanol twice until all impurities are removed. To separate the pectin from the ethanol, it was centrifuged at 4000 rpm for 15 min at 15 °C. The precipitated pectin was then dissolved and dried in a tray dryer at 60 °C for six hours and stored for further characterization.

2.3.2. PEF pre-treatment followed by Microwave heating (MW)

The PEF treatment illustrated in Fig. 1 was accomplished using a pulsed electric field generator. The sample was treated with electric pulses of predetermined electric field strength and pulse frequency for the required treatment time. The samples were treated at different pulse-field strengths (5 to 15 kV/cm) and treatment times (2 to 6 min) were then subjected to microwave-assisted extraction. The PEF pre-treated samples obtained after each set of combinations of process variables were kept in the microwave oven (Model: MH2016-E2; Energy Microwave System Pvt. Ltd. Industrial Microwave Furnace) operating at 915 MHz for different power densities (450 to 650 W/g) and exposure time (5 to 15 min) as per the experimental design (Table 1). The separation procedure followed is given in section 2.3.1.

2.4. Pectin yield (%)

The pectin yield percentage obtained by PEF followed by MW and conventional heating (CH) was calculated from alcohol insoluble solid value by a formula given by Muñoz-Almagro, Valadez-Carmona,

Table 1

Experimental design and experimental (RSM & ANN) results of parameters.

Run	Independent Variables				Pectin yield (%)		Energy consumption (kW-h)	
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	RSM	ANN	RSM	ANN
1	10	6	450	10	16.3	16.245	0.194	0.185
2	10	2	650	10	15.9	15.847	0.192	0.187
3	15	4	650	10	17.4	17.385	0.193	0.187
4	10	4	450	05	14.5	14.306	0.098	0.097
5	10	4	550	10	18.3	16.571	0.192	0.186
6	05	4	450	10	14.6	14.641	0.192	0.185
7	10	4	650	15	17.6	17.445	0.289	0.285
8	05	4	550	15	15.3	14.405	0.289	0.283
9	10	4	550	10	18.0	16.734	0.193	0.189
10	05	2	550	10	14.2	13.346	0.192	0.185
11	10	4	450	15	16.1	15.885	0.289	0.277
12	10	4	550	10	17.8	16.922	0.193	0.195
13	10	2	450	10	14.3	14.014	0.192	0.190
14	05	4	550	05	13.9	14.207	0.098	0.095
15	10	2	550	05	15.7	15.367	0.096	0.091
16	15	2	550	10	15.2	15.249	0.192	0.186
17	10	4	550	10	16.9	16.594	0.193	0.184
18	15	4	550	05	16.2	15.876	0.098	0.096
19	10	6	550	05	17.4	18.342	0.099	0.096
20	05	4	650	10	16.4	16.138	0.193	0.187
21	10	6	650	10	17.3	17.332	0.194	0.187
22	10	4	550	10	17.7	16.922	0.193	0.186
23	10	6	550	15	16.8	16.578	0.289	0.287
24	05	6	550	10	15.8	15.642	0.194	0.189
25	10	4	650	05	18.1	17.738	0.098	0.093
26	15	6	550	10	17.6	16.522	0.194	0.189
27	15	4	550	15	16.9	15.557	0.289	0.277
28	10	2	550	15	17.0	15.930	0.287	0.273
29	15	4	450	10	15.6	15.392	0.193	0.194

\*X<sub>1</sub> = Pulse Field strength (kV/cm); X<sub>2</sub> = PEF treatment time (min); X<sub>3</sub> = Microwave power density (W/g); X<sub>4</sub> = Time of exposure (min); RSM = Response Surface Methodology; ANN = Artificial Neural Network.

Mendiola, Ibáñez, and Villamiel (2019)

$$\text{Pectin yield (\%)} = \frac{\text{Mass of pectin obtained}}{\text{Mass of alcoholic insoluble solid}} \times 100 \quad (1)$$

2.5. Chemical and functional properties of extracted pectin

Following properties were analyzed for the PEF extracted pectin and compared with conventionally extracted pectin.

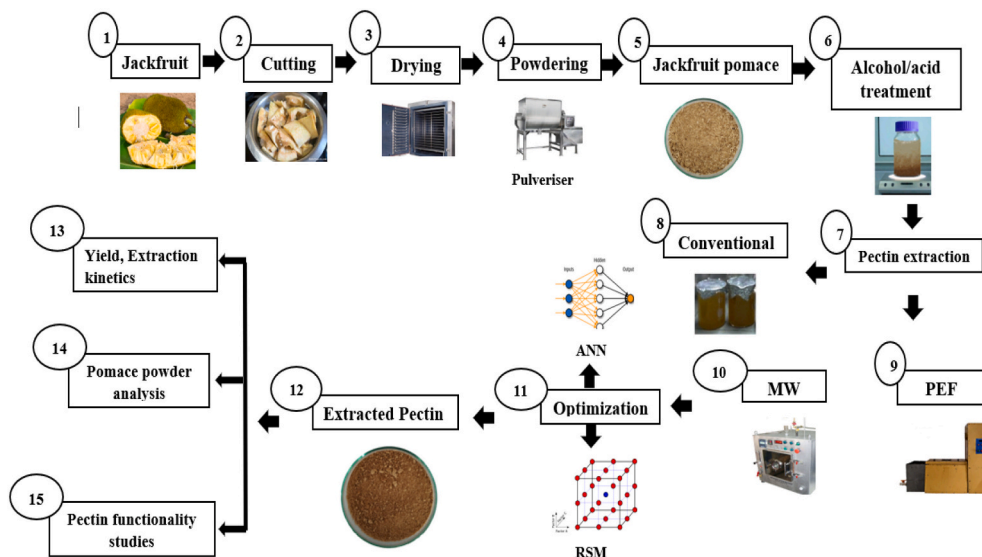


Fig. 2. Process flowchart for pulsed electric field and microwave assisted extraction of pectin from jackfruit rind and core.

### 2.5.1. Moisture, ash and protein content

The extracted pectin's moisture, ash, and protein content were calculated by the standard procedures given by AOAC (2016).

### 2.5.2. Equivalent mass

The equivalent mass of extracted pectin was determined by titrating with 0.1 N NaOH with a known weight of pectin and then the phenol red indicator drops have been added to the mixture immediately and standardized against with 0.1 N NaOH solution till it turns into a pink color

$$\text{Equivalent mass} \left( \frac{\text{g}}{\text{mol}} \right) = \frac{\text{Mass of pectin}}{\text{Number of NaOH moles consumed}} \quad (2)$$

### 2.5.3. Methoxyl percentage

The methoxyl percentage of the pectin samples were analyzed using the neutralized solution (Titration A) obtained from the equivalent weight calculation

$$\text{Methoxyl content (\%)} = \frac{\text{weight of pectin ml of alkali} \times \text{Normality of alkali} \times 3.1}{\text{Weight of sample}} \quad (3)$$

### 2.5.4. Anhydrouronic Acid (AUA) content

The anhydrouronic acid content of final product specimens have been measured by applying the standards of previously determined equivalent weight and methoxyl percentage to the following equation.

$$\text{AUA (\%)} = \frac{176}{Z} \times 100 \quad (4)$$

where,

176 = molar mass of anhydrouronic acid

$$Z = \frac{\text{mass of sample (mg)}}{\text{meq of alkali for free acid} + \text{m eq of alkali for methoxyl}} \quad (5)$$

### 2.5.5. Degree of esterification

The degree of esterification of extracted pectin samples was analyzed through volumetric approaches as Sucheta Chaturvedi, Sharma, and Yadav (2019) stated. The value was determined using the data obtained from methoxyl and anhydrouronic acid content with the following formula.

$$\text{DE (\%)} = \frac{176 \times \text{MeO (\%)}}{31 \times \text{AUA (\%)}} \times 10 \quad (6)$$

### 2.5.6. Solubility

The solubility of the extracted sample in water (cool and mess water) and solutions (Cold and Hot Alkali) used to be investigated according to the procedure explained as per Lokhande, Wani, and Siddiqui (2016). The visual appearance of the pectin in water (cold and hot water) and solutions (Cold and Hot Alkali) were noted.

### 2.5.7. Viscosity

The viscosity of pectin powder would have resolute using a shear rheometer (RS 12001 per min, Anton Paar, Germany) over concentric cylinder measuring systems (DIN 53019, particle size 0.1 mm, and Angular resolution 2  $\mu$ rad). Flow and viscosity curves by raising shear rate with 0.1 to 500  $\text{s}^{-1}$  have been determined at 30 °C. The values were noted and expressed in centipoise.

### 2.5.8. Color

The hunter color values L, a\*, and b\* have been analyzed utilized Aeros Spectrophotometer (Hunter Lab, Germany). The equipment has been initially fine-tuned by the one standard pursued through optimized samples. Extracted powder pectin specimens have been located in a Petridis or glass jar with open top by rotating platter prior noted reading results by way of dual-beam non-contact reflectance spectrometer.

## 2.6. Inherent morphological assessment of pectin

Scanning Electron Microscope (SEM) pictures of specimens have been attained carry out unprocessed samples and processed through a PEF, MW, and traditional heating. The models have been firmed over aluminum object supporter further flatter via gold in a modular coater (JEOL-3000F, Japan), at an expediting voltage of 12.0 kV.

## 2.7. Energy utilization

Using a three-phase AC static watt-hour energy meter, energy utilization was measured to understand the energy expended during PEF treatment and the microwave extraction process. The electrical energy consumed to conduct PEF pretreatment and microwave extraction for all the treatments as per the experimental design were calculated to find out the total energy requirement for each set of experiments.

## 2.8. Experimental procedure, modelling, and multi prolonged optimization

The pectin extraction parameters optimization was determined through BBD and ANN. The extraction parameters of Pulsed-field strength (5, 10, and 15 kV/cm), PEF treatment time (2, 4, and 6 min), microwave power density (450, 550, and 650 W/g), and time of exposure (5, 10, and 15 min) influences on pectin yield and energy consumption have been assessed using ANN and BBD experimental approach. The observational design network of predatory and response variables about twenty-nine runs, akin to indicated through Table 1. Carry out scientific method exploited multiple order rectilinear regression analysis for adequate the experimental date as well as classify a significant model particular were depicted at equation given below.

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 \quad (7)$$

where, Y is the response value;  $b_0, b_1, b_2, b_3, b_4$  represent correlation factor for linear terms;  $b_{11}, b_{22}, b_{33}, b_{44}$  existent correlation factors about squared expression;  $b_{12}, b_{13}, b_{14}, b_{23}, b_{24}, b_{34}$  exist correlation factors of cross-product words and  $X_1, X_2, X_3, X_4$  has to be summarized code for explanatory parameters. The statistical evaluation and ANOVA was determined through utilizing Design Expert Version 12.0a (Stat-Ease, Inc. USA). In favor of determine the confidence level by 0.01%, 1%, and 5% over single, mixed as well as square terms. The optimization challenges may have specified concisely in the form to identify the design parameters, establish better mathematical modelling as well as high pectin yield.

## 2.9. Artificial neural network (ANN)

The ANN becomes a proficient modelling implement, including various benefits across different well-known modelling methods, namely response surface methodology. ANN control various variables to model multi-component non-linear and linear regression issues (Chau, Le, Dao, Dang, & Dang, 2019; Srikanth et al., 2020; Srinivas et al., 2020). During the current progress, the ANN model consisted of four neurons (pulsed-field strength, PEF treatment time, microwave power density, and time of exposure) as input level, five neurons for the hidden level, and two neurons (pectin yield and energy consumption) for the output level. The number of hidden neurons were varied between 2 and 10 and the optimum number of neurons was selected by the hit and miss technique based on the statistical parameters determination- $R^2$  and mean square error-MSE achieved throughout training, validation, and testing (Mahanti, Chakraborty, & Babu, 2019). The stimulating operation to the hidden layer and exit layer of the network have been interpreted by way of elaborate function and linear function severally. The

training computation to improve the intake layer to concealed layer and a hidden layer to exit layer network was the Levenberg-Marquardt back propagation method. The total data points ( $29 \times 3 = 81$ ) were segregated on 60% training, 20% validation, and 20% testing utilizing the toolbox wizard. Since the problem is finite mapping, a positive response (feed-forward) network with one hidden layer was assigned. Whole statistics (both date entering and exit) have been standardized from  $-1$  to  $1$ . The posted information has been nourished to instruct the ANN model in feed-forward multilayer perceptron (MLP) category design utilizing the backpropagation algorithm (BP). Formerly the mean square error attained to  $10^{-2}$ , the training used to be completed. Design of neural network was done using "start" toolbox in MATLAB online R2020b.

## 2.10. Statistical analysis

The results obtained from conventional extraction technique and PEF-MW assisted extraction technique at optimized conditions were subjected to ANOVA and mean separation was carried out using Duncan's multiple-range test in SAS software (SAS Institute, Cary, NC, USA). All the experiments were replicated thrice. The data were depicted as mean  $\pm$  SD.

## 3. Results and discussion

### 3.1. Optimization of experimental design

The polynomial regression models were produced and exploited to transform different method parameters over-dependent parameters. The model's adequacy has been quantified by considering the coefficient of determination ( $R^2$ ), F-test, and lack of fit (Table 2). This additionally depicts those practical variables impact every paradigm anticipating different comparable responses.

#### 3.1.1. Pectin yield (%)

The incurred data obtained for each combination are represented in Table 1. The extracted pectin yields of different combinations varied between 13.9 and 18.3%. The maximum pectin yield (18.3%) was obtained at 10 kV/cm pulsed electric field strength treated for 4 min at 550

**Table 2**

F-value, P-value and significance of each variable on performance parameters of pectin extraction ANOVA table for the effect of pulsed field strength, PEF treatment time, microwave power density, exposure time and their interaction on the pectin yield and energy consumption.

Source	Pectin Yield (%)		Energy Consumption (kW-h)	
	F value	p-value	F value	p-value
Model	8.41	0.0001	34,960.38	< 0.0001
A-Pulse Field strength	17.95	0.0008	0.9589	0.3441
B-PEF treatment time	18.78	0.0007	77.6712	< 0.0001
C-Microwave power density	30.28	< 0.0001	0.9589	0.3441
D-Time of exposure	3.61	0.0784	489,359.1	< 0.0001
AB	0.46	0.5108	0	1.0000
AC	0	1	2.8767	0.1120
AD	0.35	0.5643	0	1.0000
BC	0.26	0.6207	0	1.0000
BD	2.57	0.1314	0	1.0000
CD	3.14	0.0983	0	1.0000
A <sup>2</sup>	32.16	< 0.0001	0.0207	0.8876
B <sup>2</sup>	10.94	0.0052	0.8760	0.3652
C <sup>2</sup>	8.91	0.0098	0.0207	0.8876
D <sup>2</sup>	4.99	0.0423	2.2317	0.1574
R <sup>2</sup>	0.8937		0.9899	
Adj R <sup>2</sup>	0.7874		0.9799	
Pred R <sup>2</sup>	0.4866		0.9799	
Adequate Precision	8.795		568.648	
Lack of Fit	1.4	0.3984	0.2083	0.9798

W/g microwave power density exposure for 10 min. The mathematic models formulated by the variation of the pectin yield with dependent parameters ( $X_1, X_2, X_3, X_4$ ) has more suitable in the multinomial mathematical equation observed in Table 3a (Eq. 1). The statistical results obtained from the regression analysis were tabulated in Table 2. The maximum  $R^2 = 0.893$  (Table 2) value is demonstrating that more closeness among independent and dependent values, therefore confirming that model is more suitable. From the results, it is clearly evident that the individual effect of pulsed-field strength, PEF treatment time, microwave power density, and microwave process time had a significant effect ( $p < 0.001$ ) on pectin yield (Table 2). On the other hand, the interaction effect of these parameters was found to be non-significant but the quadratic terms are significant. The impact on explanatory parameters ( $X_1, X_2, X_3$ , and  $X_4$ ) overproduction of pectin yield is demonstrated in the 3D response plot (Fig. 3 (a-d)). An increase in field strength with time during PFE extraction initially increased the pectin yield (up to 13 kV/cm field strength and 5.5 min processing time) subsequently diminished eventually the figure displaying a concave manner. Similarly, by increasing the power density and time of exposure, the pectin yield initially increased after that, showing a curvature manner. The increase in yields could be due to the electroporation of cells by strong electric fields. PEF pre-treatment fractured the sclerenchyma cells resulting in the formation of intercellular spaces; electro permeabilization efficiently released pectin from the tissues. At higher exposure times, the cell became contracted due to deplasmolysis, which finally led to a decreased pectin yield. In contrast to microwaves slackens the basement membrane, generates prompt gravimetric heating inside tissues because of the oscillations of ionized molecules, which leads to a quick increase in temperature, pressure accumulation in addition cytolysis, therefore discharging the pectin with the plasma membrane. In traditional processing, contrasting to microwaves, the heat dissipation is sluggish. It involves protracted moments to achieve temperatures, which might result in plasma membrane splitting. The chemical processing for 30 to 90 min comes to renewed highest extract pectin with usual of plant origin (Wang et al., 2015). The traditional thermal extraction has now been established outcome for observably maximum extraction yield contrast for alternative methods due to prolonged warming times. Guolin, Jeffrey, Kai, and Xiaolan (2012) reported the same information, such as the straight interconnection of microwaves over solution results in the liberation of intracellular materials to the solvent, which reduces the time of pectin extraction and increment in extraction efficiency. The other reason might be the inactivation of the methyl pectin esterase, which in turn decreased pectin's solubility, thereby increasing the extraction efficiency of pectin. The higher extraction yield of combined treatment might be attributed to the fact that microwave radiation as well as pulse filed strength are known to loosen the cell wall matrix and cause the severing of the parenchymal cells leading to increased interaction between the plant material and the extracting solvent, this observation was confirmed by Dranca, Vargas, and Oroian (2020). Karbuz and Tugrul (2021) also reported a similar finding in 2021, indicating the combined effect of microwave and ultrasound heating enhanced pectin yields in various fruit peels. Overall, PEF electric field pre-treatment followed by microwave extraction can be seen as an efficient method to increase the output of pectin.

**Table 3a**

Quadratic models developed using independent variables from experimental design.

Properties	Model
Pectin yield (%)	$Y_1 = 17.74 + 0.72A + 0.74B + 0.94C - 1.32A^2 - 0.77B^2 - 0.70C^2 - 0.52D^2$
Total energy consumption (kW-h)	$Y_2 = 0.19 + 1.200 \times 10^{-3} \times B + 0.095D$

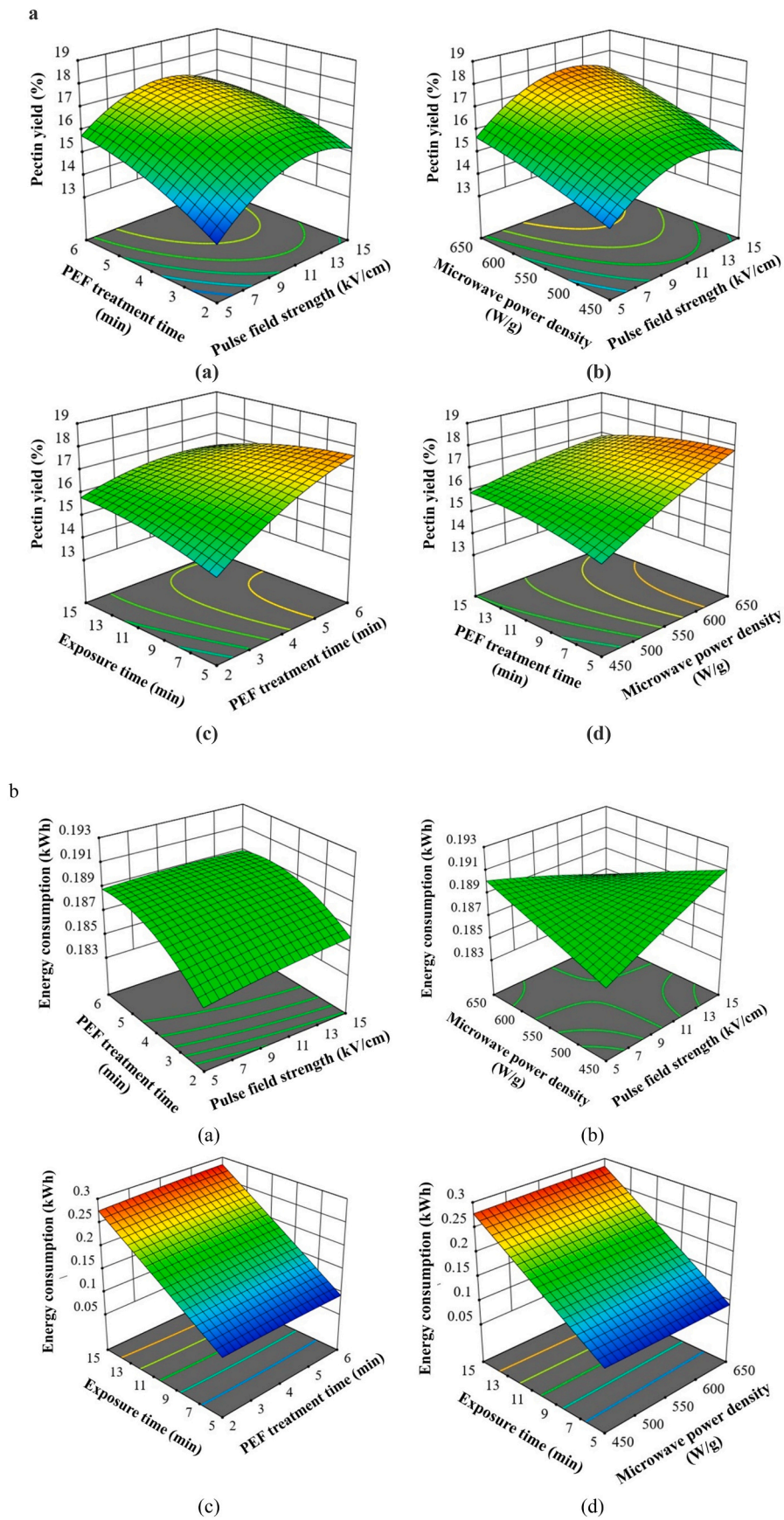


Fig. 3. a. Response 3D exemplifying the consequences of combined PEF and microwave assisted on pectin yield values.

Fig. 3b. Response 3D exemplifying the consequences of combined PEF and microwave assisted on energy consumption values.

### 3.1.2. Total energy consumption

The total energy consumption of pulsed electric field with microwave-assisted extraction ranging from 0.098 and 0.2885 kWh throughout actual experimental conditions, with minimum energy input obtained at 5 kV/cm PEF strength for 2 min of treatment followed by a microwave treatment at a power density of 450 W/g exposed for 5 min. The energy consumption of the optimized conditions, which resulted in the highest yield, is 0.1916 kW-h. A mathematical polynomial equation used to be internalizing within pectin extraction energy consumption of experimental data that produces a retrogradation model ascertained in Table 3a (Eq. 2). The maximum  $R^2$  (0.989), adjusted  $R^2$  (0.979), and non-significant lack of fit of the developed model indicated closeness among the experimental and predicted values. The ocular 3D contour plot (Fig. 3 (a-d)) depicts individual and interaction effect of PEF treatment time, pulsed field strength, microwave power density, and exposure time on energy consumption during pectin production. Increasing in PEF treatment time and field strength during the PEF-MW process constantly increase energy consumption (Fig. 3 (a)). Similarly, the energy consumption increased linearly with increasing microwave power density and exposure time (Fig. 3 (d)). The reason for microwave treatment the assimilation of electromagnetic radiation energy within the extraction method encouraged the extractant's thermal aggregation, bringing on the disintegration of pectin in the direction of the solution, which leads to more energy consumption. Conventional heating was minimal energy competent pursued by the pulsed electric field, whereas microwave system was valued the better energy proficient. While relatively increased yield occurs attainable over prolonged convective heating, the power spent becomes considerably greater. Therefore, considering work and energy consumed, PEF-MW was concluded as the more cost-efficient consequent to minimal specific energy (kJ/kg) within the selected techniques. Yang, Jin, Tian, Jin, and Xu (2016) drafted the practical method to extract pectin from orange peel waste through an electric field-assisted process concluded the same information that energy increases linearly by increasing the pulsed strength due to usage of specific electrical motive power as accessory energy results in significant enhancement on mass transfer as well as extraction efficiency. Dao et al. (2021) reported similar results for the fruit peel pectin extraction by combined methods indicated in microwave and pulse electric process heating the energy is directly conveyed into the material whereas in contrast to conventional heating, whereby heat is transferred to the target, firstly, by conduction via the walls of the reaction vessel, then by both conduction and convection through the reaction medium which leads more total energy consumption in conventional method than combined non thermal method.

### 3.1.3. Model optimization

The quadratic models developed using experimental data to predict the pectin yield and energy consumption were further optimized removing the non-significant terms to generate the multivariate analysis equation as predicting analog response. The equations were generated by using the significant variables were reproduced via MATLAB's "fitlm" action used demonstrated in Table 3a. It represents that the developed multivariate linear equations contained only the essential parameters, which promptly affected output parameters and improved the overall performance. Accordingly, the present statistical regression is given in Table 3a was endorsed as anticipating pulsed electric field microwave-assisted pectin extraction.

### 3.1.4. Process optimization by BBD

Multiple parameter optimization in favor of the restrained responses has now been carried out by implementing economic software (Design expert 11.0a) through the Box-Bhenkan method. The goal was to maximize pectin yield and the lowest energy consumption to optimize process parameters (PFS, PTT, MWP, and MWT). The preferred optimized pulsed electric field combined with microwave characteristics resulted in the highest output of 18.24%, the pulsed-field strength of

11.99 kV/cm, treatment time (4 min), Microwave power density 647.30 W/g, and time of exposure 5 min. The highest pectin yield (18.24%) has resulted from the lowest energy consumption i.e. 0.0964 kWh. As a result, the chosen models exist precisely to establish alterations in entire variables.

### 3.2. Interpretation of artificial neural network

ANN has now been additionally approved to establish the accurate model to ascertain the association among pectin yield as well as the process variables, the correspondence within the focus (practical value), and the exit values (ANN output) of training, validation, and testing have to be demonstrated are shown in Fig. 4 and b. The training information, substitution, and examination stated a virtuous fit ( $R^2 = 0.99$ ) among the ANN-artificial production data and the real observational data, characterizing that the anticipated model possessed an improved anticipating ability. The ANN about numerous concealed neurons can indicate little practice error, although maximizing conclusion error as a result of over-fitting. Applying the network analysis streamline the method, the ANN network at this investigation was restricted to preference the appropriate numeral of neurons at the concealed layer since the numeral of neurons in favor of entry, and exit plies have earlier been determined with the observational model. The quantity of five neurons at the concealed layer was resolute using practice various feed-forward structure of diverse network analysis additionally attenuative the lowest assess at the level means square error (MSE) of  $1.85 \times 10^{-10}$  and the highest consideration in the  $R^2$  of 0.999 (Table 3b). The deal among practical values and forecast data provided through the ANN has been displayed in Fig. 4b(a-b) for pectin yield and energy consumption; whole aspects are placed near straight as an arrow, which shows such ANN network anticipates the observational values carry out treated authenticate area completely. The assessment of multiple correlation coefficients ( $R^2 = 0.953$  pectin yield and  $R^2 = 0.999$  energy consumption) indicate a virtuous correspondence among observational and forecast data. Such an outcome displays this predictive precision of the model amounts to massive. Consequently, the acquired optimal network of ANN model for the current issues intricates a feed-forward neural network about four intake neurons, one concealed stratum over six neurons, and one exit stratum comprising two neurons. The optimal network of ANN structure by this instance is revealed in Fig. 4. Whole neurons with the concealed layer possess a log-sigmoid transfer function (log-sig), and the exit stratum neurons have a linear transfer function (purelin). This feed-forward network must be stated as ANN (4:5:2), attributing to the number of neurons within the entry, the concealed and exit layers, accordingly. This indicates that pectin yield and energy consumption can be properly predicted using the ANN model. Compared to RSM, ANN could predict all the responses with a single model rather than a separate one. This may be because ANN models can handle nonlinear responses better than RSM (Chau, Dao, & Nguyen, 2018). However, interaction and personal effects can't be interpreted using the ANN model compared to RSM.

### 3.3. Comparison of optimized (PEF-MW) and conventional heating (CH) pectin characterization

#### 3.3.1. Moisture, ash, and protein content

Food chemical codex suggests lower moisture content (<12%) for better acceptability of pectin powder. The moisture content of pectin extracted from both techniques are non-significant. Still the lowest moisture content was observed in pectin extracted from the PEF MW technique (8.95% w.b.) as compared to pectin extracted from the CH method (10.04%) (Table 4a), this implies its higher storage stability. The decrease in moisture content may be due to the splitting of water molecules entrapped in the pectin network by the charged particles and electrons generated during PEF applications. The water molecules' absorption of microwave radiant energy resulted in dissociation (Chehade,

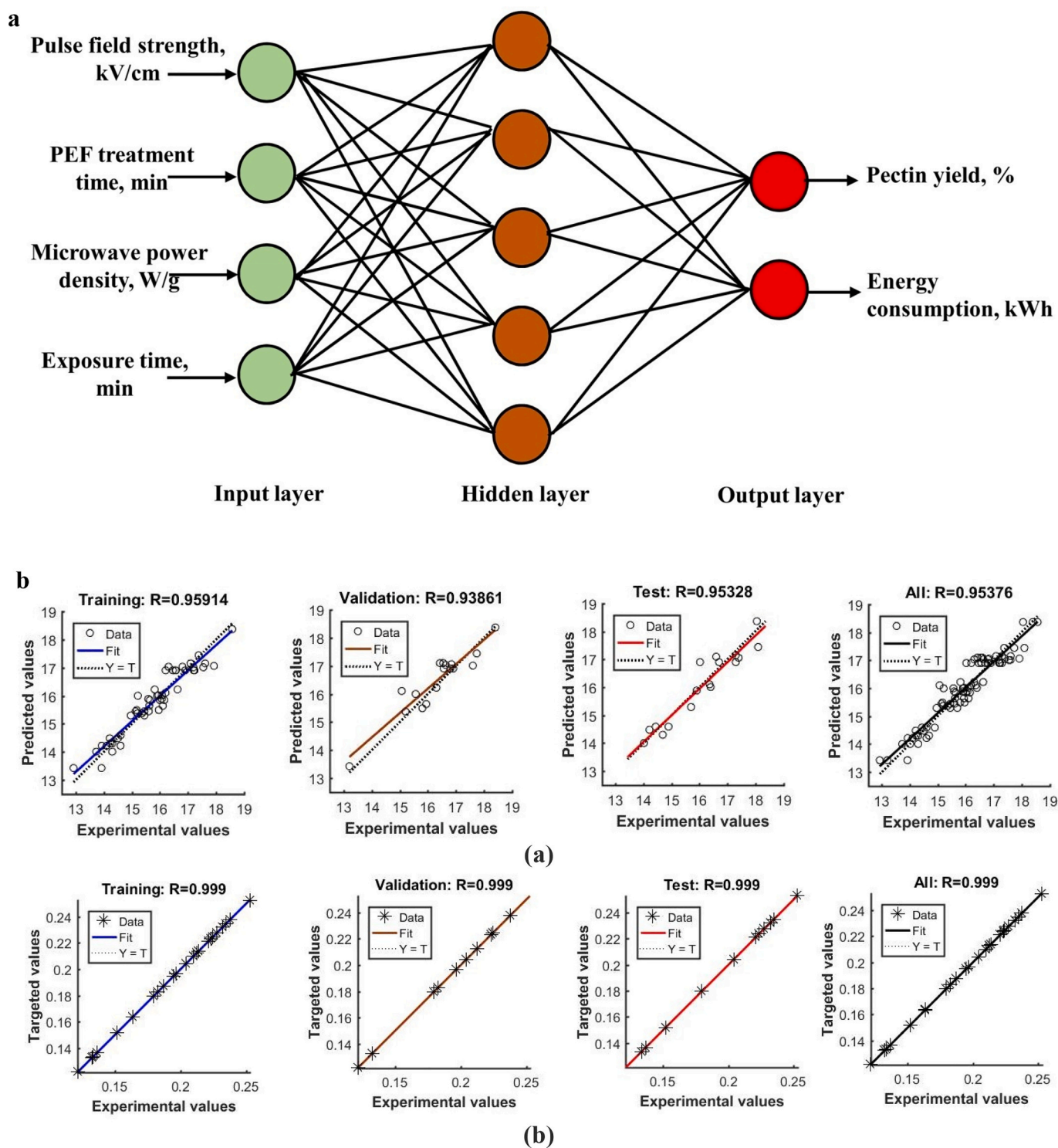


Fig. 4. a. Configuration of multilayer ANN model with four input neurons, six hidden neurons and two output neurons.

Fig. 4b. The relationship between experimental values and predicted values for the models developed using artificial neural network attributes (a) pectin yield and (b) energy consumption of PEFMW.

Lytle, Ishaq, & Dincer, 2020) could be another reason. Higher moisture content could initiate microbial growth and results in quality deterioration of pectin. Ash content indicates the number of inorganic impurities present in extracted pectin (Gull, Prasad, & Kumar, 2015). Yapo (2009) reported the reduction in ash content in pectin as an indication of the purity of pectin contributing to good gelling strength. The ash content of pectin extracted by the PEF-MW technique (6.78%) was non-significantly lower than the CH method (7.27%) indicating a superior

pectin quality (Table 4a). A similar decrease in ash content of pectin was also obtained in microwave-assisted extraction from jackfruit rind (Koh, Leong, & Noranizan, 2014). The above authors have also stated that the lower ash and protein content attributed superior gelling properties of pectin. Similarly, in the present study, the crude protein content of pectin extracted by PEF-MW technique (3.283%) was significantly ( $p < 0.05$ ) lower than the pectin obtained through the CH method (9.98%).



**Table 3b**  
Comparison of performance of Box behnken design and artificial neural network.

S.No	Box Behnken design				ANN			
	Variable	Experimental range	R <sup>2</sup>	SSE	Predicted range		R <sup>2</sup>	MSE
1	Pectin yield (%)	13.9–18.3	0.89	0.076	13.34–18.34	Training	0.959	0.116
						Validation	0.938	0.180
						Test	0.953	0.154
2	Energy consumption (kW-h)	0.096–0.289	0.99	0.781	0.093–0.287	Training	0.999	$6.98 \times 10^{-10}$
						Validation	0.999	$1.86 \times 10^{-10}$
						Test	0.999	$1.85 \times 10^{-10}$

**Table 4a**  
Functional properties of extracted pectin.  
Comparison of quality parameters of pectin extracted from conventional and PEF-MW assisted extraction technique.

Attribute	Unit	Conventional extraction	PEF-MW assisted extraction
Moisture content	%	10.04 ± 0.6a	8.95 ± 0.4a
Solubility in cold water	–	Insoluble	Insoluble
Solubility in hot water	–	Soluble	Soluble
Solubility in cold alkali	–	Soluble with precipitate	Soluble with precipitate
Solubility in hot alkali	–	Soluble	Soluble
Viscosity	cP	38.14 ± 0.2a	39.78 ± 0.1b
L*	–	56.61 ± 1.4a	73.63 ± 1.8b
a*	–	8.44 ± 0.9a	5.88 ± 1.1b
b*	–	17.06 ± 0.3a	16.59 ± 0.4a
Ash	%	7.27 ± 0.2a	6.78 ± 0.3a
Protein	%	9.98 ± .8a	3.283 ± 0.6b
Equivalent mass	(g/mol)	466.905 ± 1.9a	557.473 ± 1.7b
Methoxyl percentage	(%)	9.376 ± 0.2a	8.37 ± 0.5a
Anhydrouronic acid	(%)	67.85 ± 0.3a	69.44 ± 0.6b
Degree of esterification	(%)	78.45 ± 0.6a	68.43 ± 0.4b

### 3.3.2. Equivalent mass, methoxyl percentage, DE and GalA

The equivalent weight of pectin adds up to the whole number of unesterified free galacturonic acid across molecular chains of pectin (Kazemi, Khodaiyan, Labbafi, Hosseini, & Hojjati, 2019). Pectin extracted by PEF-MW technique showed a significantly ( $p < 0.05$ ) higher equivalent mass (557 g/mol) than that obtained through the CH method (467 g/mol) (Table 4a). Putra (2010) reported that the declination of equivalent mass is due to increased demethylated pectin methoxyl groups and partial degradation of pectin. Another reason may be caused by some breaking in the linear pectin molecule leading to a weaker network formation (Rodsamran & Sothornvit, 2019). A higher equal mass of pectin is a good indicator of gel formation. At the same time, methoxyl percentage indicates the quantity of methyl ester clusters in pectin. The methoxyl content of pectin becomes an essential component that identifies the gel-forming ability and setting time. The methoxyl percentage of pectin extracted from the PEF-MW technique (8.37%) was non-significantly lower than the CH method (9.376%) (Table 4a). However, the methoxyl percentage of pectin obtained through both extraction methods was more significant than 7, indicating high methoxyl content (Wignyanto & Rahmah, 2014; Dranca et al., 2020). In the presence of acid and high sugar, the high methoxyl pectin is desirable for good gel formation. Fruits-based products like jams and jellies require high methoxyl pectin, which is acidic and high sucrose content. The application of non-thermal technology increased equivalent weight but decreased the methoxyl percentage. The Pectin with low Methoxyl forms a thermo-irreversible gel, which implies that it remains gelled even once heated to temperatures that usually melt it (Fakayode & Abobi, 2018). Anhydrouronic acid (AUA) determination is a significant criterion for the characterization of pectin (Canteri et al., 2012:). More excellent anhydrouronic acid content indicates high impurities in pectin (Liang et al., 2012; Yang, Wang, Hu, Xiao, & Wu, 2018). The AUA content of PEF-MW extracted pectin (69.44%) was significantly ( $p < 0.05$ ) higher than the CH method (67.85%) (Table 4a). FAO has suggested a requirement of a minimum of 65% of galacturonic acid content for pectin to be considered pure. In the present investigation, the AUA

content of extracted pectin was more than 65% in both cases. The high ash content of extracted pectin directly signifies the presence of high impurity. The GalA and AUA increased PEF-MW due to forming micelles that establish chemical and physical interactions with hydrophilic or lipophilic substances (Su et al., 2019). Pectin with a degree of esterification (DE) value over 50% is categorized as higher ester pectin, whereas pectin with DE less than 50% is classified as lower ester pectin. Gelling ability of pectin increases with an increase in the percentage of DE. Both extraction methods yielded a DE value higher than 50% and are graded as higher ester pectin, resulting in high gelling power. However, the DE of pectin obtained from PEF-MW technique (68.43%) was significantly lower when compared with pectin obtained from CH method (78.45%) (Table 4a). The decrease in DE is due to the de-esterification of pectin caused by the interaction of charged particles and high-energy microwave radiations. Sucheta Chaturvedi et al. (2019) reported a similar decrease in DE value that the harsh conditions of microwave treatment have triggered the de-esterification of poly-galacturonic chains of pectin.

### 3.3.3. Solubility, viscosity, and color

The results of solubility, viscosity, and color are given in Table 4a; it was observed that both the pectin samples were insoluble in cold water but soluble in hot water, cold and hot alkali. The Solubility of the extracted compounds depends upon the dielectric properties of the sample and its extraction rate (Picot-Allain, Ramasawmy, & Emmambux, 2020). The viscosity of the pectin solution obtained from the PEF-MW treatment was (39.78 cP) significantly higher than the conventionally extracted pectin (38.14 cP). The higher viscosity is may be due to the change in conformation of the macromolecules and depolymerization of pectin chains caused by PEF treatment. The increase in the number of the depolymerized chains could restrict the flow, increasing viscosity. It is also associated with the decrease of intermolecular distance among the pectin molecules, enhancing intermolecular interactions, particularly hydrogen bonding and molecular entanglements (Picot-Allain et al., 2020). The color attributes (L\*, a\*, and b\*) indicate

lightness, red/green, and blue/yellow. The color of pectin acts as an influential factor since it affects the final product, mainly depending upon the raw material and extraction method. It is commonly colorless, milky, or yellowish. Kutlu, Isci, Sakiyan, and Yilmaz (2021) reported that phenolic compounds or other color pigments associated with pectin are responsible for the color of pectin and are influenced by temperature and extraction time interaction. Analogously, in the present work, the higher  $L^*$  value is observed in combined PEF-MW extracted pectin compared to conventional extraction. Lighter color indicates the minimal effect of heat during the extraction of pectin, and it is generally preferred (Guandalini, Rodrigues, & Marczak, 2019; Koh et al., 2014). The final combined PEF and microwave-assisted extraction yielded pectin of brownish-orange color. Usually, Millard browning or caramelization is accountable as the obscure color of conventionally extracted pectin (Sucheta Chaturvedi et al., 2019).

### 3.3.4. Scanning electron microscopy (SEM)

The surface topography of the extracted pectin samples obtained by both methods was studied using scanning electron microscopy. From the micrographs shown in Fig. 5, it can be inferred that the jackfruit samples

contain a relatively smooth surface before extraction. The micrographs obtained after conventional extraction show pores indicating structural decomposition and rupture of cells in a larger size, which might be due to exposure to heat for a longer time. Intensive vapor formation in the capillary-porous structure of the plant material, significant pressure is built up, modifying its physical properties. The capillary-porous characteristics of the fruit tissue improved due to the increase in the pressure and temperature inside the tissue. However, the surface ablation and severing of the parenchymal cells are more in PEF-MW samples compared to the conventional heating method. The increased surface area and intracellular spaces due to the PEF treatment enhanced the pectin yield compared to the traditional process. The electroporation caused by PEF-MW treatment resulted in a higher number of pore formation and cell disruption, indicating rapid structural rupture than another extraction method. Extensive destruction of plant tissue was observed in the combined PEF and microwave-assisted extraction process. The results suggest that extraction using microwave radiation has a strong destruction effect on plant tissues of Jackfruit than thermal process, causing swelling effect leading to the rupture of cells and better extraction. The pulsed electric field and microwave treated samples

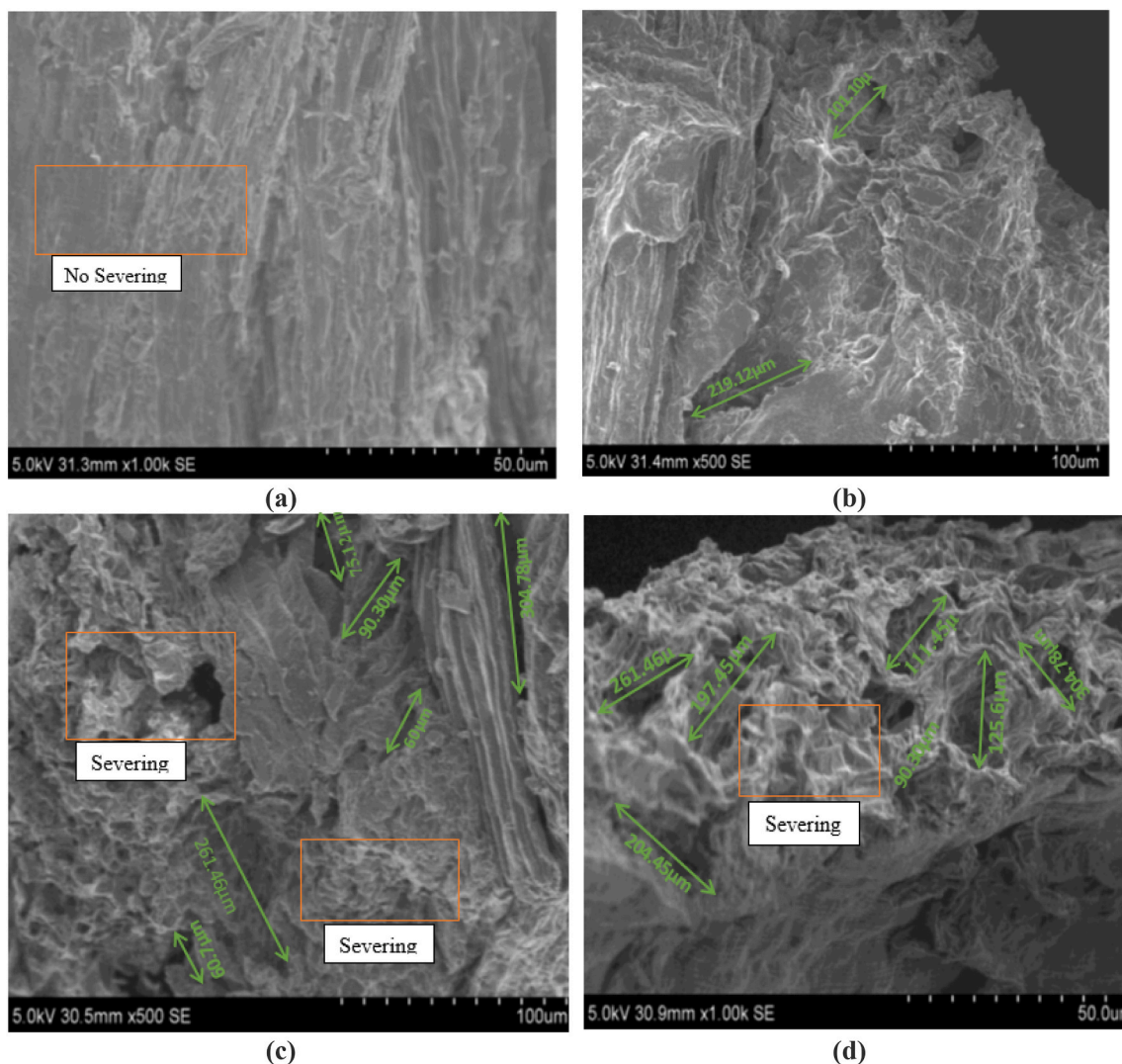


Fig. 5. SEM micrograph of Jackfruit powder at 500 x and 1000 $\times$  magnification (a) raw powder (b)conventional extraction (c-d) combined PEF and microwave assisted extraction.

Schematic diagram of pectin extraction pectin from jackfruit rind and core by microwave, Pulse electric field and conventional process.

**Table 4b**  
Energy consumption and cost of energy expended.

Parameters	Conventional extraction	PEF-MW assisted extraction
Yield (%)	17.1 ± 0.3a	18.24 ± 0.6b
Time (min)	45 ± 0.4a	10.46 ± 0.4b
Power consumed (W)	359.999 ± 1.6a	355.987 ± 1.9b
Energy consumed (kJ)	1333.33 ± 12.4a	567.522 ± 8.45b
Specific Energy (kJ/kg)	210,525.789 ± 10.7a	19,559.740 ± 13.7b
Price per unit of electricity (INR/kWh)	6 ± 1	6 ± 1
Cost per unit production of pectin (INR/Kg)	26.1 ± 1a	18.5 ± 1a
Cost per unit production of pectin (US\$/kW-h)	0.0857 ± 0.02a	0.0085 ± 0.003a

\*Same letters within the row indicates significant difference ( $p < 0.05$ ).

were found to have more cell rupture compared to models that have undergone conventional extraction methods. This indicates the combined impact of pulsed electric field and microwave processing causes increased cell rupture, represent the permeation (due to electric field strength) of intercellular matter which increases extraction efficiency of pectin from jackfruit waste powder, proving it an efficient method for pectin extraction with minimal application of heat (Dranca et al., 2020; Moens et al., 2021). Therefore, it can be stated that PEF-MW pectin exhibited higher functionality in terms of WHC or solubility as needed in food applications (Sengar, Rawson, Muthiah, & Kalakandan, 2020; Sucheta Chaturvedi et al., 2019).

### 3.3.5. Cost economics and energy consumption

The cost economics for a developed PEF-MW technique was calibrated and compared with the CH method is showed in Table 4b. Conventional heating used to be the lowest energy effective than the PEF-MW. Although microwave extraction has been considered the utmost energy component, since it used to be finalized, it remains the best cost-effective outcome in minimum specific energy (kJ/kg) within the three methods and valuable research despite industrial larger scale for extract pectin with jackfruit rind and core. The energy cost used would have been estimated at US\$ 0.0085 per kilogram of pectin developed. It becomes significant to observe electrical heating utilized to carry out analysis as well as predictions.

## 4. Conclusions

In the present study, multiple optimizations and modelling of pectin extraction from Jack fruit rind and core using pulsed electric field combined with microwave treatment and conventional heating (CH) have been identified. The pulsed-field strength (11.99 kV/cm), PEF treatment time (4 min), Microwave power density (647.30 W/g), and time of exposure (5 min) to anticipating the exploratory network that could improve the yield (18.24%) and lower energy (0.0986 kW-h) consumption. Moreover, the optimized sample has lower moisture (8.95%), ash (6.75%), methoxyl (9.376%), and degree of esterification than the conventional heating method. Similarly, good color, solubility (water and alkali), and viscosity (39.86 cp) with enhanced gelling properties were observed for optimized samples. The optimized model possesses a higher rate of extraction and higher yield due to immediate proportional heating ability, instantly interrelated through the degree of esterification, Anhydrous acid, particle size and water holding. PEF-MW extraction led to polymer disintegration, depolymerization, and subsequently a lesser fragment size as well as lower gelling capacity being apparent. BBD and ANN coefficient of determination were identified as 0.994 and 0.999, indicating excellent conformity to the observational values. The designs become evaluated according to this  $R^2$  (coefficient of determination), the sum of squared error (SSE) and mean squared error (MSE). The observational outcome of multiple linear regressions was furnished with the quadratic polynomial equations. The higher values about  $R^2$  (coefficient of determination) and lower values about MSE (mean square error) as ANN constitute superior

prognostication on observational data above the Box-Behnken design. Besides, further study is required to elucidate the structural characterization of pectin and also the produced pectin be utilized in edible film creation for food packaging.

## Declaration of Competing Interest

Authors declare that they have no conflicts of interest.

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