Highlights

- Banana peel can be utilized as a promising alternative for the commercial production of pectin.
- The process parameters were investigated and optimized using artificial neural network (ANN) and response surface methodology (RSM).
- The physiochemical characterization of pectin was carried out at optimized operating conditions.
- The extracted pectin was characterized in terms of moisture, anhydrouronic acid, and degree of esterification, ash, equivalent weight and methoxyl contents.
- Experiments carry out using Neural Network Toolbox of MAT LAB version 8.1 (R2013a) and Central composite design (CCD) through design expert 11.0.0 software to evaluate the effect of process variables on oil yield.

Artificial Neural Networks (ANNs) and Response Surface Methodology (RSM) Approach for Modeling and Optimization of pectin extraction from banana peel

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Abstract

The present study, the influence of three independent variables for extraction of pectin were investigated and optimized using artificial neural network and response surface methodology on the yield and degree of esterification of banana peel pectin obtained using acid extraction method. The results revealed that properly trained artificial neural network model is found to be more accurate in prediction as compared to response surface method model. The optimum conditions were found to be temperature of 82^{o}C, pH of 2 and extraction time of 102 min in the desirable range of the order of 0.977. The yield of pectin and degree of esterification under these optimum conditions was 15.64% and 65.94, respectively. Temperature, extraction time and pH revealed a significant ($p < 0.05$) effect on the pectin yield and degree of esterification. The extracted banana peel pectin was categorized as high methoxyl pectin, based on the high methoxyl content and degree of esterification. In general, the findings of the study show that banana peel can be explored as a promising alternative for the commercial production of pectin.

Keywords: Banana peel; Pectin; Artificial Neural Networks; Response surface methodology; Extraction

Introduction

The cultivated desert banana and plantain (Musa sp.) are considered as one of the most important food crop for tropical and subtropical region and play important role in food security and economy (Waghmare and Arya, 2016). In Ethiopia banana is considered as the most popular fruit crop that is most broadly grown and consumed. It covers about 59.64% of the overall fruit area, approximately 68.00% of the entire fruits produced, and about 38.30% of the total fruit producing farmers (Woldu et al. 2015). Bananas are most widely consumed as a raw, and processed into products such as banana flour, chips/crackers, and puree. Banana fruit peels constitute about 30% of the fruit, and represent an environmental problem due to their large nitrogen and phosphorus contents as well as their high water content, making them highly susceptible to microbial degradation(Oliveira et al. 2016). Utilization of banana peels as a source of high value compounds like pectin (Happi Emaga et al. 2008), cellulose nanofibers and phenolic compounds is interesting not only from an economic point of view, but also from an environmental perspective(González-Montelongo et al. 2010).

Pectin is a water-soluble, methylated ester of polygalacturonic acid which contains methoxylgalacturonic acid and galacturonic acid as their key components(Liu et al. 2015) and present within all dicotyledonous plant cell walls. Pectin is considered as one of the most valuable products which can be primarily extracted from apple pomace, citrus peel, guava extract, sugar beet and sunflower heads. Pectin's are widely used as a functional ingredient within the food industry, pharmacy and cosmetic manufacture thanks to its ability to form aqueous gels, dispersion stabilizer (Mohamed, 2016). Generally, two types of pectin are available in nature such as high methoxyl pectin (greater than 50% DE) and low methoxyl pectin (below 50% DE) forms gel after heating in sugar solutions at concentration above 55% and pH below 3.5. On the other hand, the formation of gel with a low methoxyl pectin (LMP; DE < 50%) requires the presence of calcium ions, extending the use of this gelling agent to a broader range of foods(Wai et al. 2010).

The effect of process parameters on pectin yield during extraction from banana peels have been studied by several researchers, however, to the best our knowledge there was no report on the detailed analysis of process parameters for extraction of pectin and degree of esterification(DE) from banana peel by coupling ANN and RSM methodology. The RSM is a statistical mathematical tool that widely employed to examine multiple regression analysis using quantitative data obtained from appropriate experiments to determine and simultaneously solve multivariant equations. In recent years, artificial neural network (ANN) has arisen as an efficient and attractive approach for nonlinear multifactor modeling due to its generic structure and ability to learn from historical data. ANN is a powerful mathematical method suitable for modeling and simulation of various processes in real engineering application.

Therefore in the present work, RSM and ANN linked genetic algorithm-based models have been developed to predict the relationship between the input variables and the output variables. Subsequently, the results predicted by the ANN and RSM techniques were compared statistically to the coefficient of determination (R^2), root mean square error (RMSE), mean absolute error (MAE), standard error of prediction (SEP%), and absolute average deviation (AAD%) based on the validation data set for their predictive and generalization capabilities. An effective RSM model and a feed-forward neural network on back-propagation were developed by utilizing the experimental data, and the efficiency of both models was compared. Therefore, it is important to identify appropriate extraction conditions to obtain maximum possible yield of pectin from banana peel. Therefore, this study was conducted to investigate the effect of extraction conditions namely pH, temperature and time on yield and the degree of esterification of banana peel pectin and to optimize these conditions to extract maximum possible pectin by employing ANN and RSM.

2. Materials and methods

2.1 Materials

Banana (varieties: Dwarf Cavendish and Giant Cavendish) were collected from some selected hotels, juice processing houses and restaurants in Jimma, Ethiopia. All chemicals used for the extraction process were of analytical reagent grade.

2.2 Raw material preparation

The fresh banana peels were segregated according to their type and chopped into approximately 1cm^2 pieces using a stainless steel knife for easy drying and washed with water three times. Sample drying was carried out in an oven at 60°C for 48 hours to obtain easily crushable material. The dried peel was milled using a mechanical grinder and then screened to pass through a sieve size of 60 meshes and packed in an airtight, moisture-proof bag at room temperature and ready for the extraction process.

2.3 Preparation alcohol insoluble solids

Banana peel powder of the samples were homogenized in boiling ethanol (solid–liquid ratio of 1:10, w/v) with a final ethanol concentration of 80% (v/v) at 70^{o}C for 20min in a shaking water-bath to inactivate possible endogenous enzymes and remove AIS. Then after, the resulting residue was washed with distilled water and air-dried at 50°C.

2.4 Pectin extraction

In this study, pectin was extracted according with the methodology proposed by (do Nascimento Oliveira et al., 2018), with a few modifications. An alcohol insoluble solid was mixed in a conical flask with the extracted solution (solid-liquid ratio of 1:40 (w/v). Pectin was extracted from alcohol insoluble solids by using three different extraction conditions to study the effect of each condition on the pectin yield. The extraction was done at different temperatures (52.5, 60, 71, 82, and 89.5^{o}C), pH (1.66, 2, 2.5, 3 and 3.34) and extraction time (44.7, 60, 82.5,105 and 120 min). The hot acid extracts were separated from the alcohol insoluble solids residue by filtering through nylon/muslin cloth and cooled immediately by chilled water, dispersed in an equal volume of 96% ethanol, stirred 5 min for proper mixing and allowed to stand for 3h. The precipitate was washed 2-3 times by 70% acidic ethanol (0.5% HCl), 70% ethanol and finally 95% ethanol. Finally, the precipitate was dried at 40°C in hot air oven overnight to remove the moisture until a constant weight was reached. The ground powder pectin was kept in airtight container. According to Ranganna [10], The percentage yield of the extracted pectin was determined using the following equation:

$$\text{Yield of Pectin}(\%) = \frac{\text{Amount of extracted pectin (g)}}{\text{Initial amount of mango peel (5g)}} \text{x}100 \quad (1)$$

2.5 Analytical methods

Sample of dried banana peel pectin was subjected to quantitative test in order determine its physicochemical characteristics. From the results obtained, the optimal conditions that gave the optimum yield were used for subsequent analysis.

Table 1Standards Methods used for chemical characterization of pectin

Parameters	Methods
Equivalent Weight	(Ranganna, 1995).
Methoxyl Content	(Ranganna, 1995)
Total Anhydrouronic Acid Content	(Mohamed et al. 1995)
Degree of Esterification	(Liew et al. 2014a)
Moisture Content Determination	AOAC Method 934.01(AOAC,2005)
Ash Content Determination	AOAC, (2005)
Viscosity	SV-10 Model Vibro Viscometer

Experimental design and statistical analysis

In the present work, extraction of pectin was studied to determine the optimized conditions for the pectin yield and degree of esterification. RSM is a collection of mathematical and statistical techniques to utilize quantitative information from an appropriate experimental design to identify optimum conditions. The influence of temperature, pH and extraction time were determined through a RSM, and central composite design (CCD), requiring a total of 20 experimental runs employed to determine the best combination of parameters for the extraction process. The responses and the process variables are modeled and optimized using analysis of variance (ANOVA) to predict the statistical parameters using RSM. The independent process variables range were selected based on (Fakayode and Abobi, 2018). Generally, CCD involves six factorial points, eight axial points and six points at the center were carried out with the alpha factor of 1.414. All factors have to be adjusted at five coded levels (-α, -1, 0, +1, + α) (Nahar et al. 2017). The relationship between the coded and the actual value of the variables is shown in Table 3.The variables were coded according to the equation:

$$N = 2^{m} + 2m + m_{c} = 8 + 2x3 + 6 = 20 \qquad (2)$$

where N is the total number of experiments required, m is the number of variables and m_c is number of replicates. The relationship of the variables and response was determined by second-order polynomial multiple the quadratic regression equation.

$$Y = b_o + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j$$

(3)

where Y is the predicted response (i.e. yield and DE), n is the number of independent variables, b_0 is the constant coefficient, b_i is the linear coefficients, b_{ij} is the second-order interaction coefficients, b_{ii} is the quadratic coefficients and x_i and x_j are the coded values of the independent variables.

The outcomes were summarized and statistically analyzed by using Design Expert version 11software (Stat-Ease Inc., Minneapolis, USA) and Neural Network Toolbox of MAT LAB version 8.1(R2013a). The ANOVA test was employed to estimate the statistical significance of the regression model. The coefficient of determination R^2, adjusted R^2, and predicted coefficient

R^2, lack of fit from ANOVA were used in the determination of the quality of the developed model.

Table 2 The coded and the actual form of the independent variables

S. No	Code variables	Actual level of variables
1	-α	$\mathbf{X}_{min}$
2	-1	$\left(\frac{\mathbf{X}_{max}+\mathbf{X}_{min}}{2}\right)-\left(\frac{\mathbf{X}_{max}-\mathbf{X}_{min}}{2}\right)*2^{\frac{n}{4}}$
3	0	$\left(\frac{\mathbf{X}_{max}+\mathbf{X}_{min}}{2}\right)$
4	+1	$\left(\frac{\mathbf{X}_{max}+\mathbf{X}_{min}}{2}\right)+\left(\frac{\mathbf{X}_{max}-\mathbf{X}_{min}}{2}\right)*2^{\frac{n}{4}}$
5	α	$\mathbf{X}_{max}$

Notes: n is the number of variables for any particular experiment, n = 3

2.6 Artificial Neural Network Modeling

In present study, the ANN was developed for describing the extraction condition of pectin to enhance the yield. The data generated from the experimental design planned through CCD (Table 3) were used to constitute the optimal architecture of ANN. ANNs were introduced recently into the field of engineering studies as a tool for optimization and modeling of systematic variable studies involved in a particular process. ANN has been applied for the purpose of simulation on the same experimental data used for RSM.

The neural network architectures were trained by Levenberg–Marquardt back-propagation algorithm. The network architecture consisted of an input layer of three neurons (Temperature, pH and, extraction time), an output layer of two neurons (pectin yield and DE), and a hidden layer. There are 60% of data points were selected for training to develop the neural network, 20% of the data set used for validation and 20% data sets for testing. The more data sets in training reduce processing time in ANN learning and improve the generalization capability of models. This step makes possible the assessment of the generality of the ANN model. The number of neurons in the hidden layer can be calculated from the expression $2(n + m)^{0.5}$ to 2n + 1 where n is the number of neurons in the input layer and m is the number of neurons in the output layer (Sundarraj et al. 2018a). A network is built each of them is trained separately, and therefore, the best network is selected based on the accuracy of the predictions within the testing phase. Levenberg–Marquardt back propagation is presented in Figure 1.

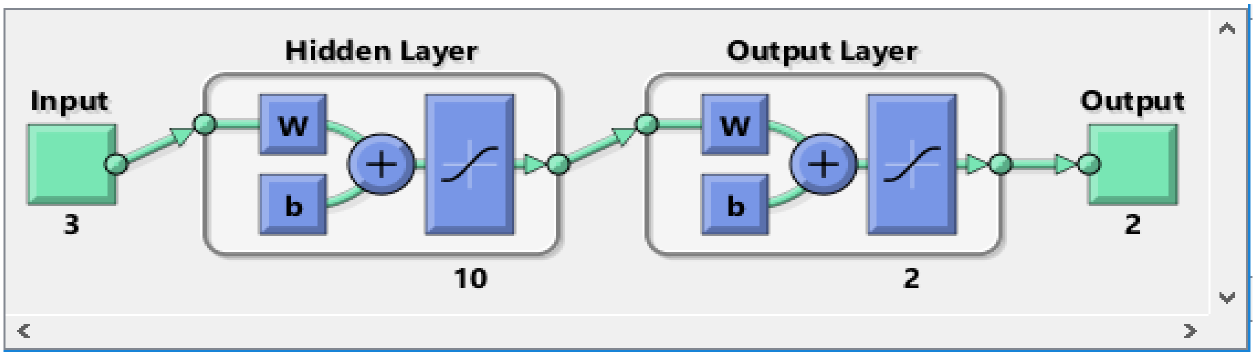

Figure 1. Feed-forward with the backward propagation neural network used in the current study

The network inputs and target have been normalized before training. The input and target data for the individual ANN nodes were normalized within a range of -1(new x_{min}) to 1(new x_{max}) in order to achieve fast convergence to obtain the minimal RMSE values. For normalization target data the following equation

$$x_{norm} = \frac{2(x_{ac} - x_{min})}{x_{max} - x_{min}} - 1 \quad (3)$$

$$x_{ac} = \frac{(x_{norm} + 1)(x_{max} - x_{min})}{2} + x_{min} \quad (4)$$

where X_{min}, X_{max} and X_{Ac} are the minimum, maximum and actual data, respectively. The normalization of inputs and target was performed to avoid overflows that may appear due to very large or very small weights. The training process was run until a minimum of the MSE was reached in the validation process. The performance of the trained network was estimated based on the accuracy of the network with the test data. Feed forward with backward propagation is one of the most common neural networks used in solving engineering problems. All calculations were done using the Neural Network Toolbox of MAT LAB version 8.1(R2013a) utilized throughout the study (Joel et al. 2018).

2.7 Comparative analysis of RSM and ANN models

In order to evaluate the goodness of fitting and prediction accuracy of the constructed models was performed by using the root mean square error (RMSE), mean absolute error (MAE), correlation coefficients (R^2), standard error of prediction (SEP), and absolute average deviation (AAD) were calculated between experimental and predicted data. The formula used for error analysis were calculated by equation (5) to (9) (Liew et al. 2014b). To study the modeling abilities of the RSM and ANN models, the values predicted by the RSM and ANN models are plotted against the corresponding experimental values.

$$SEP = \frac{RMSE}{Y_e} x100 \quad (5)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}\left(Y_{i,p} - Y_{i,e}\right)^2}{\sum_{i=1}^{n}\left(Y_{i,p} - Y_{e}\right)^2} \quad (6)$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n}\left(Y_{i,e} - Y_{i,p}\right)^2}{n}} \quad (7)$$

$$MAE = \sum_{i=1}^{n}\left(\frac{Y_{i,e} - Y_{i,p}}{n}\right) \quad (8)$$

$$AAD = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|Y_{i,p} - Y_{i,e}\right|}{\left|Y_{i,e}\right|} \quad (9)$$

where, $Y_{i,e}$ is the experimental data, $Y_{i,p}$ is the predicted data obtained from either RSM or ANN, Y_e is the mean value of experimental data and n is the number of the experimental data. The final network was selected based on the lowest error in the train and depending upon the test data.

3. Results and discussion

3.1 Pectin yield

The yields of pectin extracted and degree of esterification using 1M H_2SO_4, from banana peel powder ranges from 10.52 to 15.87% and 61.27 to 65.95, respectively of the dry weight of peel depending on the extraction conditions. RSM has been widely adopted to investigate the effects of several design factors influencing a response by varying them simultaneously in a limited set of experiments. Thus, temperature, pH and extraction time were examined as factors to investigate the correlation between the process variables to the pectin yield and DE by using CCD. The complete experiment variables design matrix together with the values of experimental responses is presented in Table 3. The analysis of variance was carried out to investigate the model terms, to select a suitable model, and to detect the significances of the model equation.

Table 3 Central Composite design matrix and experimental results

Run	Coded variable			Decoded Variable			Dependent Variable	
	A	B	C	Temp(^{o}C)	pH	Time (min)	Pectin yield (%)	DE
1	-α	0	0	52.5	2.5	82.5	12.08	62.98
2	0	0	0	71	2.5	82.5	14.32	64.97
3	1	-1	-1	82	2	60	14.65	65.13
4	1	1	1	82	3	105	12.84	63.25
5	0	0	0	71	2.5	82.5	14.26	64.88
6	1	1	-1	82	3	60	11.67	62.16
7	-1	1	-1	60	3	60	10.52	61.27
8	-1	-1	-1	60	2	60	12.74	63.11
9	0	0	+α	71	2.5	120	14.49	65.07
10	0	0	0	71	2.5	82.5	14.19	64.75
11	0	-α	0	71	1.7	82.5	14.86	65.52
12	0	0	0	71	2.5	82.5	14.23	64.79
13	0	0	0	71	2.5	82.5	13.98	64.43
14	0	0	-α	71	2.5	44.7	12.81	63.23
15	1	-1	1	82	2	105	15.87	65.95
16	+α	0	0	89.5	2.5	82.5	13.98	64.39
17	0	0	0	71	2.5	82.5	14.13	64.68
18	-1	-1	1	60	2	105	13.78	64.24
19	-1	1	1	60	3	105	12.53	63.08
20	0	+α	0	71	3.3	82.5	10.62	61.58

Table 4 Analysis of variance for response surface quadratic model of pectin yield and DE

	Pectin yield (%)		Degree of Esterification	
Source	F-value	p-value	F-value	p-value
Model	98.12	< 0.0001	76.86	< 0.0001
A-Temperature	129.51	< 0.0001	80.32	< 0.0001
B- pH	475.98	< 0.0001	365.53	< 0.0001
C - Extraction time	117.81	< 0.0001	98.37	< 0.0001
AB	19.04	0.0014	19.06	0.0014
AC	1.29	0.2833	2.84	0.1231
BC	2.50	0.1451	2.41	0.1514
A^2	53.96	< 0.0001	52.41	< 0.0001
B^2	88.71	< 0.0001	69.08	< 0.0001
C^2	10.65	0.0085	18.78	0.0015
Residual				
Lack of Fit	4.97	< 0.0516	1.68	< 0.2906

3.2 RSM modeling fitting

The statistical significance of the regression model (linear, interaction, and quadratic) effect of all the response variables was checked by the Fisher statistical test (F-test) in the ANOVA. The ANOVA is used to calculate the coefficient of determination, the significance of linear, lack of fit, and interaction effects. The statistical analyses show that quadratic models fit very well into the data for the response. The probability value (p-value) was employed to check the significance of the coefficient, indicating the interaction between each independent variable (Table 4). The smaller the p value and the higher the value of F, the more significant is the corresponding coefficient. The analysis of variance results showed a perfect fit of the quadratic regression model for banana peel pectin (F-value of 76.86) and ($p < 0.0001$). The significance of the models was obtained for the responses indicated by p values less than 0.05. In this study, the p value of "Lack of Fit" for pectin yield and DE were 4.97 ($p > 0.05$) and 1.68 ($p > 0.2906$), respectively, indicating that lack of fit was insignificant relative to the pure error. Therefore, the results obtained verified that the mentioned models (Eqs. 10 and 11) were accurate enough to predict the pectin yield and DE within the range of the variables studied. The predicted quadratic model for the two responses was highly significant ($p < 0.0001$). The analysis shows that for pectin yield, A, B, C, AB, A^2, B^2, C^2 were found having a significant effect on the pectin yield while AC and BC were not significant influence on the pectin yield. In the case of Degree of esterification, A, B, C, AB, A^2, B^2, C^2 were found having a significant effect on the DE, while AC and BC were not significant (Table 4).

Table 5 shows that the coefficient of variation (CV %) and standard deviation for the two responses in this study were reasonably low and acceptable, indicated a better precision and reliability of the experiment. Thus, the model is adequate for predictions in the range of the experimental variables. The goodness of fit of the models was further scrutinized using R^2 value. It had been suggested that R^2 value should be at least 0.80 for a good fit of a model. The regression model found to be highly significant with the R^2 value of pectin yield and DE was 0.9888, and 0.9857, respectively, indicating a close agreement between the observed and the theoretical values predicted by the model equation (Figure 2 and 3). Moreover, the value of the adjusted R^2 for pectin yield and DE was 0.9787, and 0.9729 respectively, which confirmed that the model was highly significant, indicating good agreement between the experimental and predicted values of the response variables.

Adjusted R^2 and predicted R^2 should be within 20% to be in good agreement as suggested by (Owolabi et al. 2018). This requirement is satisfied in this study with a predicted R^2 value of pectin yield and DE was 0.9200, and 0.9236 respectively. From the above analysis, it can be concluded that these models are suitable for predicting the pectin yield and DE from banana peel powder within the limits of the experiment.

Table 5 Regression coefficients of the predicted second-order model for the response variables

S. No	Response parameter	Pectin yield	Degree of esterification
1	Std. Dev.	0.2058	0.2162
2	Mean	13.43	63.97
3	C.V%	1.53	0.3380
4	R^2	0.9888	0.9857
5	Adjusted R^2	0.9787	0.9729
6	Predicted R^2	0.9200	0.9236
7	Adeq Precision	34.0709	29.3956
8	Model suggested	Quadratic	Quadratic

3.3Development of regression model equation

The experimental results obtained from the pectin yield and DE based on CCD is presented in Table 3. So as to create a simple model with a minimum of equation terms and also to prevent over-fitting, the insignificant coefficients (CE), which have values nearest to zero, are eliminated from the models. The coded equation is very important for identifying the relative impact of the factors by comparing the factor coefficients. The second-order polynomial function representing pectin yield (Y), degree of esterification (DE) can be expressed as a function of the three independent variables, namely temperature (A), pH (B) and extraction time (C) in terms of coded factors after excluding the insignificant terms were given in Equation (10) and (11), respectively. It should be noted that Eqs. (10) and (11) are only valid within the range of tested conditions: 52.5°C < temperature < 89.5°C, 44.7 min < extraction time < 120 min, and 1.7 < pH < 3.3.

$$\text{Pectin yield} = +14.18 + 0.6338A - 1.24B + 0.6058C - 0.3175AB - 0.3974A^2 - 0.5512B^2 - 0.1780C^2 \quad (10)$$

$$\text{Degree of Esterification} = +64.76 + 0.5244A - 1.14B + 0.5816C - 0.3337AB - 0.4115A^2 - 0.5110B^2 - 0.2483C^2 \quad (11)$$

Equation (10) and (11) shows the relationship between the percentage of pectin yield and DE and operating parameters. Single-factor shows the influence of a specific factor, while the combined quantities of two factors show the impact of the interaction between two variables. The positive signs in the models signify the synergetic effects of factor, while the negative sign indicates the antagonistic effect.

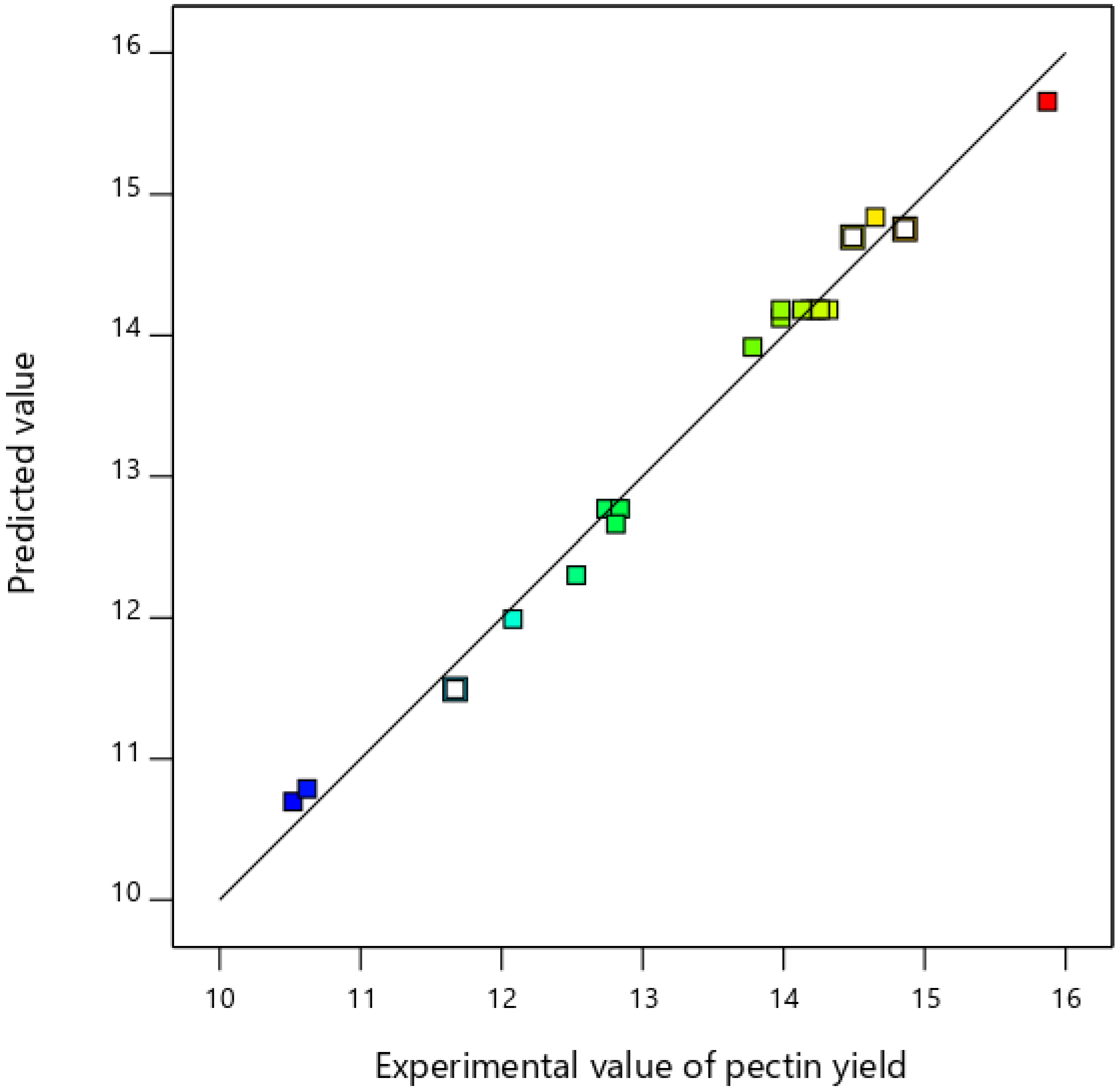

Figure 2 Correlation between experimental and predicted value of pectin yield

The model was further evaluated by the experimental values versus predicated plot of response parameters as shown in Figures 2 and 3. The Figures outcomes demonstrated that the predicted values were very good agreement with the experimental values, in which all the data points are concentrated near the diagonal line, and no scattered points were observed. The points of all predicted and actual responses fell in 45^{o} lines, indicating that the developed model is appropriate to predict the pectin yield and DE. From the graph it is clear that the values derived experimentally match closely with that developed by the model. Similar studies have been reported for pectin extraction from jackfruit waste(Sundarraj et al. 2018a) and pomelo peels (Nikolova and Georgieva, 2014).

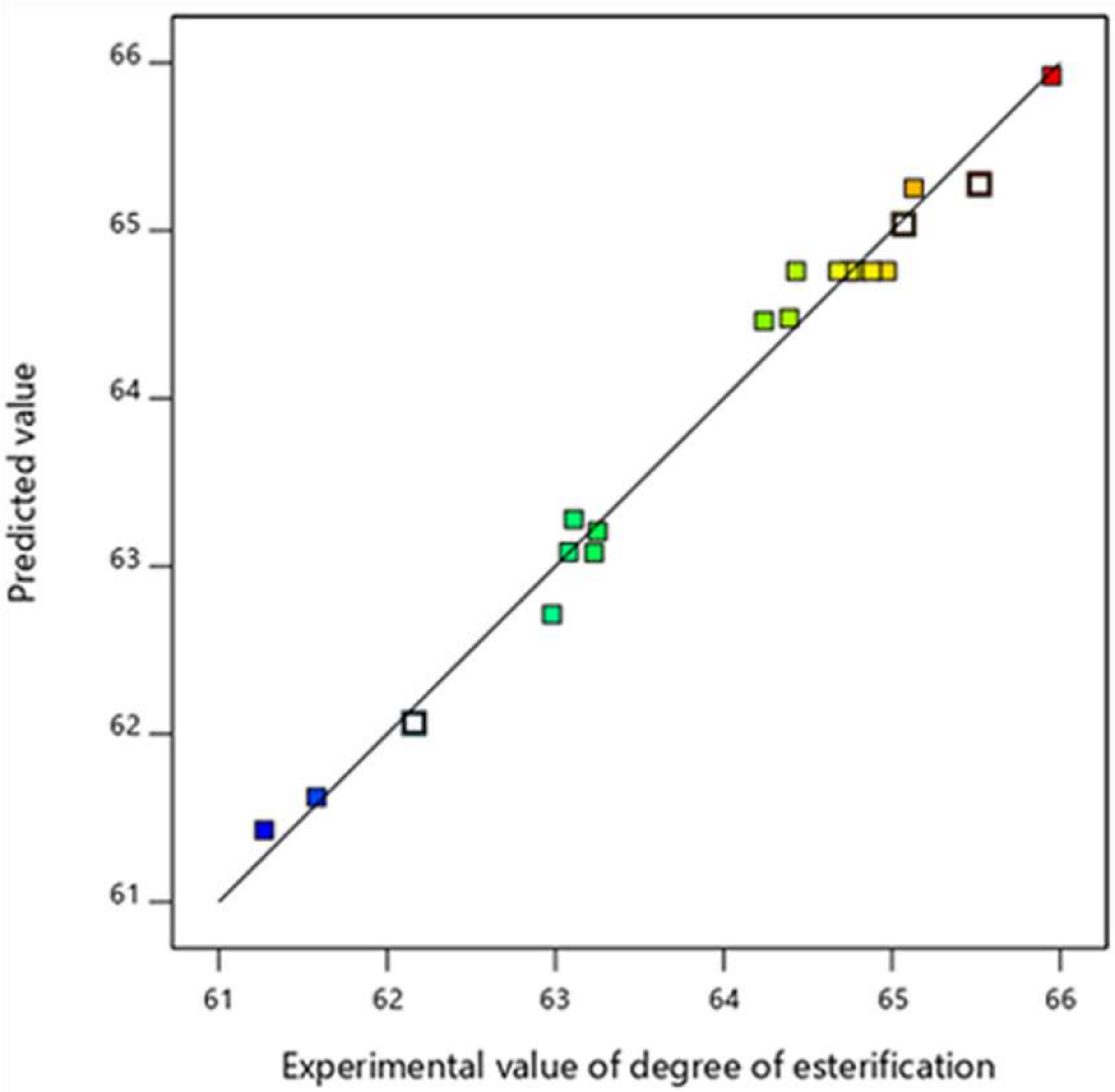

Figure 3 Correlation between experimental and predicted value of DE

3.4 Response surface analysis of extraction process

The three-dimensional response surfaces plots help to understand the main and interaction effects on the responses in a straightforward way. The regression models developed in this study have four independent variables; two variables are held constant at the optimum level, whereas the other two factors are varying within their experimental range. Based on the analysis of variance, the pectin yield and degree of esterification were significantly affected by linear, interaction and quadratic process variables. The most significant interactive effects on each response are demonstrated in Figs. 4 and 5 through 3D response surface plots.

3.5 Effect of extraction condition on the pectin yield

The present work was carried out under different experimental conditions (temperature, pH and extraction time) shown in Table 3. Pectin yield obtained in this experiment was found to be in the range of10.52-15.87% (Table 3), which is comparable to the ripe mango peel pectin (10.76-30.43%) (Nahar et al. 2017), *Citrus sinensis* peels (12.93–29.05%)(Fakayode and Abobi, 2018) and mango peel pectin (6.1-16.3%) (Girma and Worku, 2016; Sangheetha et al. 2018). Pectin yield obtained in this study is in agreement with previous studies(Sangheetha et al., 2018). Pectin extracted from banana peel was lower than Artocarpus integer (15.80 - 39.05%) (Sundarraj et al. 2018b), *Azanza garckeana* (24.38 and 26.75%) (Joel et al., 2018), grapefruit peels (25%)(Mohamed, 2016) and Ubá mango peel (18.8 - 32.1%) at different cooking conditions (Liew et al. 2014b), but higher than that of *Durio zibethinus* (2.27-9.35%,w/w) (Wai et al. 2009) and passion fruit peels (7.12-7.16%) (Wai et al. 2009). According to (Happi Emaga et al. 2008) reported that the yield of pectin extracted from the banana peel ranges from 2.4-21.7% while (Khamsucharit et al. 2018) reported that the yield of banana peel pectin ranged from 15.89 to 24.08%. These differences may be due to the nature of the fruits and extraction processing conditions.

Generally, compared to the data obtained from different sources, pectin yield lies in the accepted limits of pectin extracted conditions as suggested by (Khamsucharit et al. 2018; Liew et al. 2014b) According to the results presented herein, it is evident that the pectin yield of banana peel was comparable to values obtained from the conventional sources of pectin (i.e. apple pomace, sugar beet and citrus peel) thus, signifying the potential use of banana peel as an alternative source for the commercial-scale pectin production. Pectin yield was directly proportional to temperature and extraction time and indicated that increasing any of those parameters would result increase the pectin yield until the optimum value was achieved (Eq.10). The yields always increased if temperature and extraction time increased (with the other remaining constant), because each of these factors increases the solubility of the extracted pectin, giving a higher rate of extraction. However, further increase in temperature and extraction time decreasing the tendency of pectin yield, since too high extraction time and temperature would lead to breaking down of pectin molecules as pectin is composed of α-(1-4) inked units of galacturonic acid or methyl ester resulting in pectin of lower molecular size which is not perceptible with alcohol. Besides, the colors of the pectin extract became dark brown which in turn required a higher number of alcoholic washing of the precipitate. At lower temperatures, the lower viscosity of pectin might cause poor diffusion between the phases that will lead to a slower rate of extraction.

The result shows that the yield increases with an increase in extraction time as the prospecting naturally present in cells takes time to solubilize and go into the solution. The temperature, pH and extraction time show a significant (p < 0.0001) effect on the pectin yield. The pH value has the most significant effect on the pectin yield whose F value is 475.98, followed by extraction temperature and time (Table 4). The pH value was inversely proportional to pectin yield and indicated that increasing the value of pH would result in a decreasing percentage of pectin yield (Eq.10). A similar effect was noted in the extraction of pectin from durian rind (Sangheetha et al. 2018). The pectin yield decreases with increasing pH value; this is might be due to some pectin that might still be attached to the cell wall components although, pectin molecules can be partially solubilized from plant tissues without degradation in a weak acid solution.

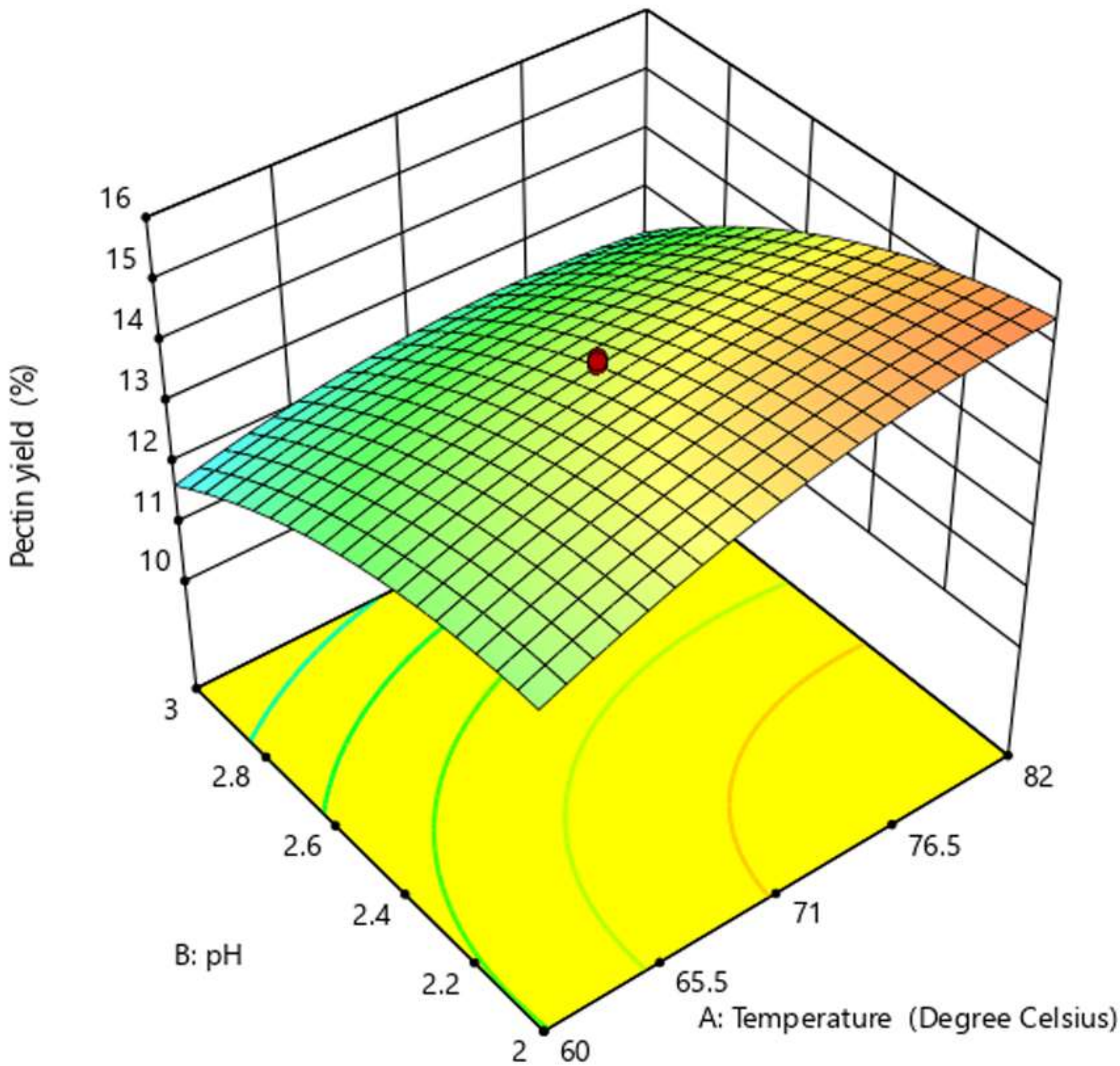

Figure 4 Three-dimensional response plots for yield as a function of temperature and pH at constant extraction time

The interaction effects between temperature and time have a significant ($p < 0.0014$) effect on the pectin yield. Compared to the other process variable, pH value had the most significant effect on the pectin yield and the effect is within the order of B > A > C > B^2 > A^2 > AB > C^2 (Table 4). Figure 4 shows a 3D response surface plot of the pectin yield as a function of temperature and pH at a fixed extraction time. Increasing the combined effect between extraction temperature and pH generally decreased the pectin yield as can be seen from Figure 4; the highest yield was achieved when both variables were at the minimum point. Relatively long period of temperature and extraction time would cause a thermal degradation effect on the extracted pectin, thus causing a decrease in the amount perceptible by alcohol. The effect of temperature, pH and period in this study is similar to previous work of(Oliveira et al., 2016).

3.6 The effect of process variable on the degree of esterification

The degree of esterification obtained in the experiment is found to be in the range of 61.27 – 65.95 (Table 3). Based on the degree of esterification pectin can be classified as low methoxyl pectin with $\leq$ 50% and high methoxyl pectin with > 50%. The presence of high methoxyl pectin (DE > 50 %) in the extracted banana peel pectin was evident (Table 3). These results were consistent with previous work of 76.30 % DE in citrus maxima and 79.51%DE in premature lemon pomace pectin(Azad, 2014) and 63.15 -72.03%DE of extracted pectins from various banana varieties, indicating that banana peel pectins have been classified as high methoxyl pectin similar to those from the citrus peel (62.83%) and apple pomace (58.44%)(Khamsucharit et al. 2018).

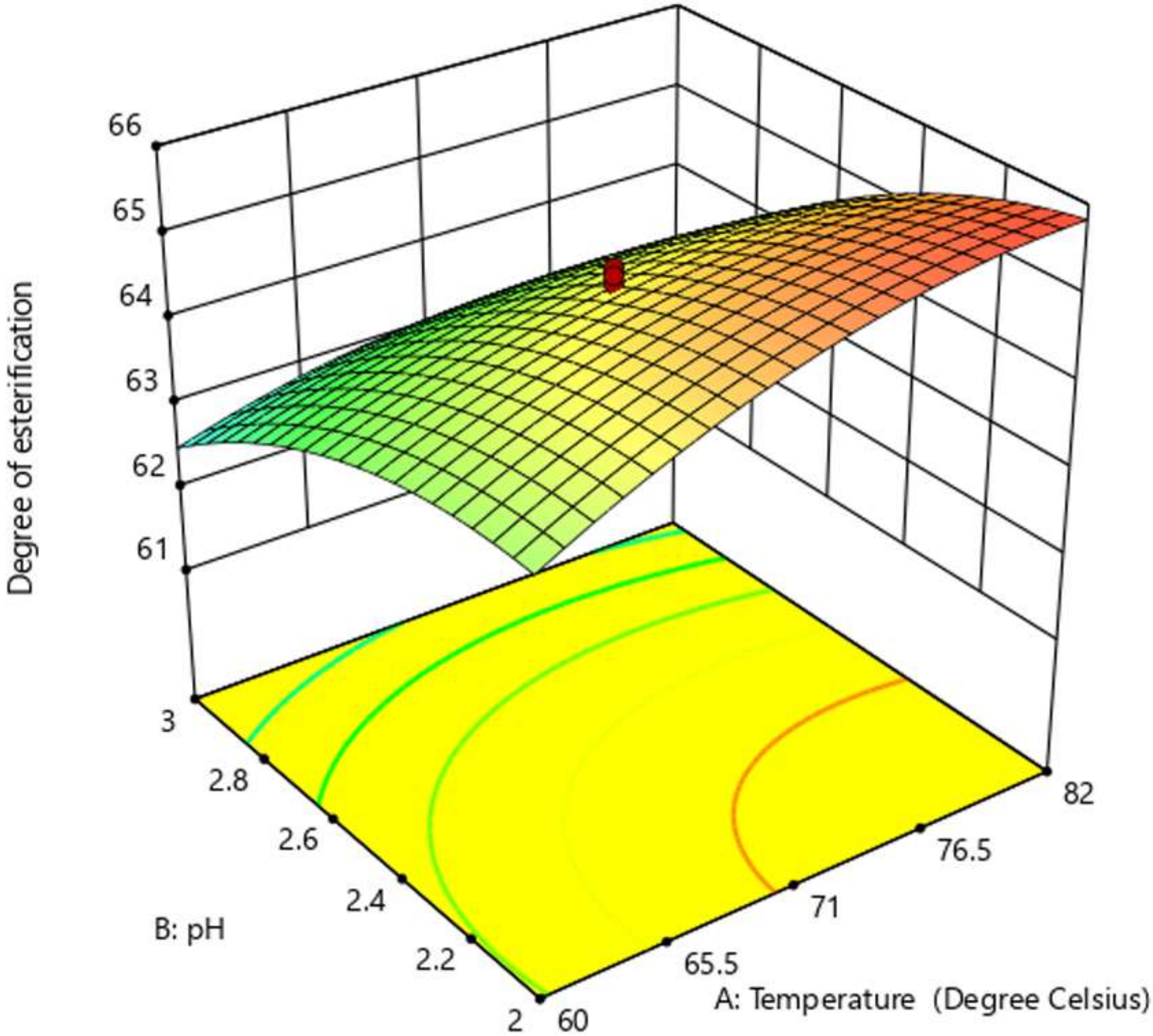

Figure 5 Three-dimensional response plots for degree of esterification as a function of temperature and pH at constant extraction time

Based on the analysis of variance, the DE of pectin was significantly affected by linear, interactions and quadratic between process variables. The pH, extraction time and temperature exhibited a significant (p < 0.0001) effects on the DE of banana peel pectin (Eq. 11). The DE was positively influenced by extraction time and temperature, while pH has a negative effect. It is clear that increasing temperature and extraction time would result in increasing in DE if other factors remained constant (Eq. 11). The results obtained from the ANOVA showed that pH has the most significant effect on the DE, followed by extraction time and temperature. The maximum DE of pectin (65.95) was obtained at a temperature of 82 °C, pH of 2, and extraction time of 105 min.

The interaction between temperature and pH exhibited a strong significant ($p < 0.0014$) effect on the DE of pectin. Significant interaction indicates that the factors work independently, whilst the presence of interaction indicates that the difference in DE at different levels of a factor is not the same at all levels of another factor. It is to note that the interaction between temperature and extraction time, as well as pH and time, did not exhibit a significant effect on DE (Table 4). In order to visualize the relationship between the response and process variables 3D response surface plots were generated from the model equation (Eq. 11) developed in this study. The 3D dimension response surface showed mutual interactions between pH and temperature as shown in Figure 5.

The three-dimensional response surface model obtained reflects a linear correlation between the DE with that of pH and temperature. This result is in agreement with the observations made by other authors (Wai et al. 2010). Figure 5 shows a 3D response surface plot of the DE of pectin as a function of temperature and pH at fixed extraction time. DE was generally decreased as the interaction between temperature and pH increased (Eq.11). The quadratic of temperature, pH and time have a significant effect on the DE of pectin. Similar findings were reported by other researchers for banana peel (Happi Emaga et al. 2008) and durian rind pectin (Wai et al. 2010).

3.7 Artificial neural network based results

Feed-forward with backward propagation neural network 3-10-2 is used in the present investigation to train the experimental data given in Table 3. In the present study, different training algorithms were tested by varying the number of hidden layers and neurons by training the different feed-forward with backward propagation networks of various topologies, in order to select the optimal architecture based on the minimization of the performance function the mean square error (MSE) and R-values of the data set with 20 samples. The ANN model depends on the decisive optimal neuron numbers. Figure 6 shows the spread plot of the experimental versus the computed ANN data in both training, testing and validation networks. The correlation coefficients (R) values for training (0.99823), validation (0.99851), test (0.99937) and all prediction set (0.99837) indicating that the ANN model shows better regression and fitting compared to RSM model. Nearly each and every data points have been scattered around the 45° line indicating remarkable compatibility between the experimental and predicted output data values by ANN. Therefore, the ANN prediction for training, validation, and testing is highly substantial and meritorious in terms of correlation and implies that the predicted model was more precise in predicting the responses.

The value of MSE obtained from the ANNs for both batch and continuous modes was 0.00098, which is close to the acceptance limit for the MSE, set to zero. The closeness of the training and testing errors validates the accuracy of the model. The linear regression analysis between the values predicted by ANN and RSM showed that the values predicted by the ANN model were much closer to experimentally measured data, suggesting that the ANN model was better modeling ability for both simulation and predicted values. Therefore, in the case of data sets with a limited number of observations in which regression models fail to capture reliably, advanced soft computing approaches like ANN may be preferred. ANN model had fitted the experimental data with an excellent accuracy.

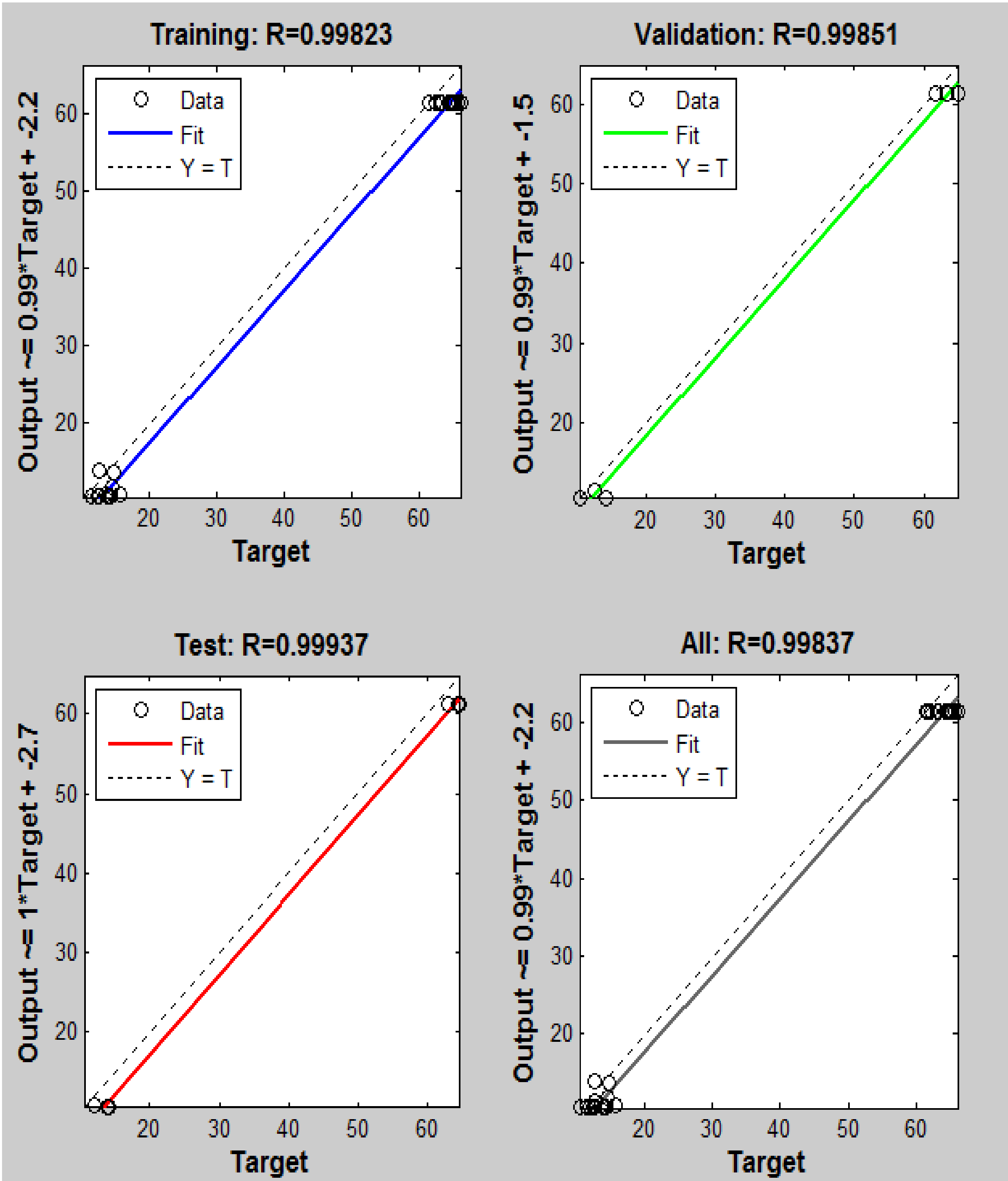

Figure 6 Neural Network model with training, validation, test and all prediction set

Table 6 Validation data set for experimentally determined ANN and RSM predicted values of pectin yield (%) and DE

Exp. No	Pectin yield (%)			Degree of esterification		
	Experimental	Predicted value		Experimental	Predicted value	
		RSM	ANN		RSM	ANN
1	12.08	11.99	12.09	62.98	62.71	62.99
2	14.32	14.18	14.28	64.97	64.76	64.98
3	14.65	14.84	14.66	65.13	65.25	65.13
4	12.84	12.77	12.85	63.25	63.21	63.26
5	14.26	14.18	14.26	64.88	64.76	64.89
6	11.67	11.49	11.68	62.16	62.07	62.19
7	10.52	10.70	10.53	61.27	61.43	61.27
8	12.74	12.77	12.74	63.11	63.28	63.11
9	14.49	14.70	14.50	65.07	65.04	65.09
10	14.19	14.18	14.19	64.75	64.76	64.75
11	14.86	14.75	14.87	65.52	65.28	65.53
12	14.23	14.18	14.25	64.79	64.76	64.79
13	13.98	14.18	14.00	64.43	64.76	64.44
14	12.81	12.66	12.83	63.23	63.08	63.23
15	15.87	15.65	15.88	65.95	65.92	65.95
16	13.98	14.12	13.99	64.39	64.48	64.39
17	14.13	14.18	14.14	64.68	64.76	64.69
18	13.78	13.92	13.79	64.24	64.46	64.25
19	12.53	12.30	12.55	63.08	63.08	63.08
20	10.62	10.79	10.63	61.58	61.62	61.59

3.8 Comparative evaluation of ANN and RSM models

The predictive competence of the ANN and RSM models were determined and compared based on prediction accuracy and various parameters such as RMSE, R^2, SEP, MAE and AAD. Table 7 shows the predictive indices for RSM and ANN models comparison for the pectin yield and degree of esterification. Both the models performed reasonably well, but ANN models have the superiority modeling capability compared to the RSM models for both pectin yield and degree of esterification. Figure 7 and 8 depicted the experimental values and those of predicted values of RSM and ANN. As can be observed, the ANN predicted value is much closer to that of the experimentally measured data, suggesting that the ANN model has superior prediction ability than the RSM model. RSM modeling is easier compared to ANN, as ANN needs a higher number of inputs than RSM for better predictions. This proves the applicability of the ANN and RSM in the prediction and optimization of pectin yield and DE with a minimal experimental setup which maximize the yield and DE.

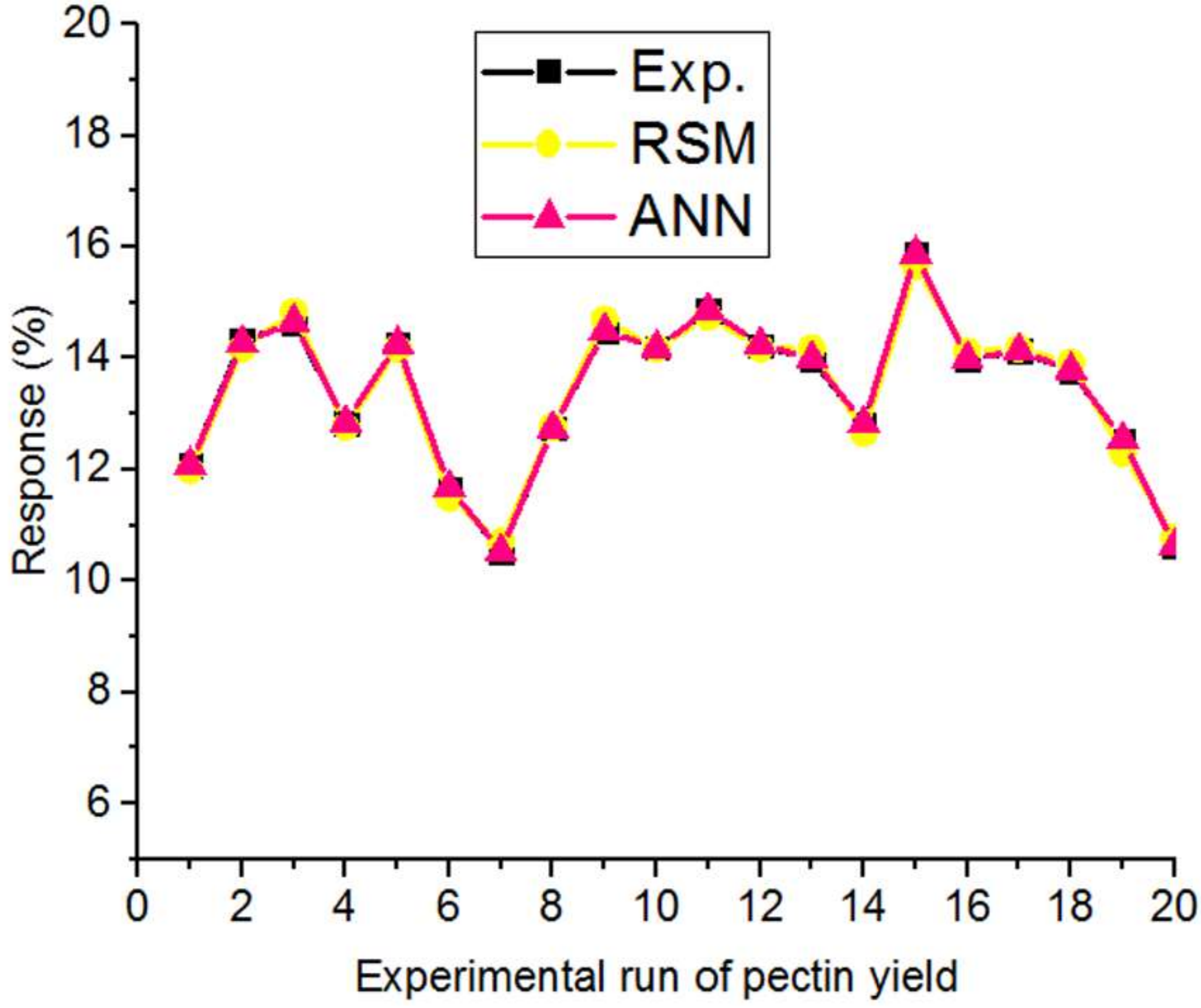

Figure 7. Comparison of experimental with predicted value obtained by the RSM and ANN model for the prediction of pectin yield

Table 7 Comparison of predictive abilities of RSM and ANN models

	Pectin yield (%)		Degree of esterification	
Parameters	RSM	ANN	RSM	ANN
RMSE	0.1473	0.0148	0.1527	0.0105
R^2	0.9884	0.9998	0.9856	0.9999
AAD (%)	1.001	0.0898	0.1899	0.0109
MAE	0.1320	0.0120	0.1215	0.0070
SEP	1.097	0.1105	0.2387	0.0164

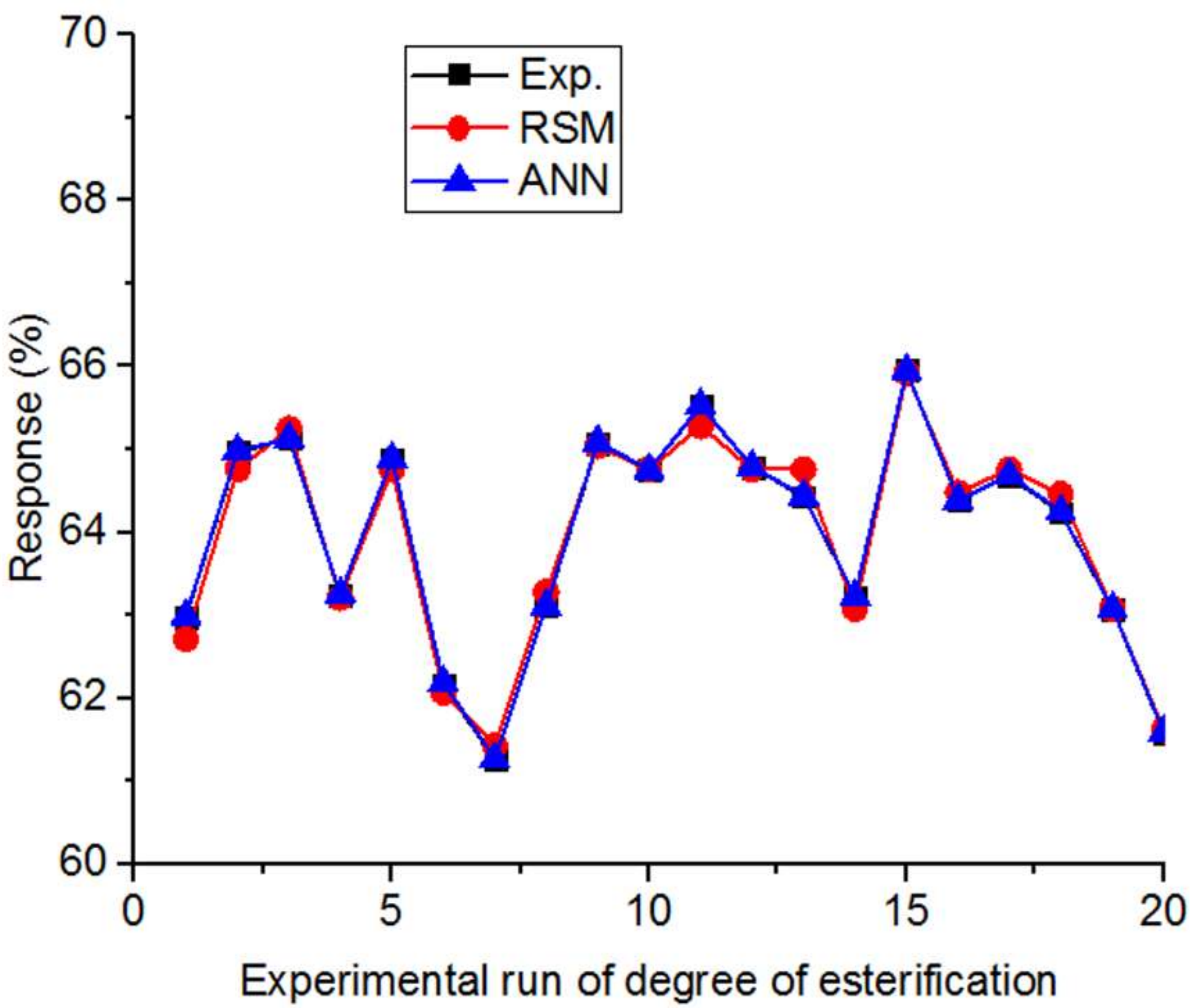

Figure 8 Comparison of experimental with predicted value obtained by the RSM and ANN model for the prediction of degree of esterification

3.9 Physicochemical characterization of banana peel pectin

The characterization of extracted pectin obtained from banana peel was carried out to evaluate its suitability in food industries. The physiochemical characterization of pectin was carried out at optimized operating conditions. The yield and properties of pectin are dependent on the source and are also affected by the nature of the extraction process used. The result shows that the anhydrouronic acid and methoxyl contents were found to be dependent on the pH while equivalent weight depends on extraction time. The extraction time and pH had significance effect on the degree of esterification and moisture content of banana peel pectin. However, moisture, anhydrouronic acid, degree of esterification, ash, equivalent weight and methoxyl contents of banana peel pectin were independent of the extraction variables. The physicochemical characterizations of pectin depend mainly on the raw material source and conditions selected for isolation and purification of pectin.

Moisture content of pectin extracted in this experiment was found to be 7.87%, which is slightly higher than banana peels of different varieties (4.54 – 6.24%) and apple pomace (4.54%) but slightly lower than citrus peel (7.92%) (Khamsucharit et al. 2018). Low moisture content is necessary for safe storage because they inhibit the growth of microorganisms and pectinase enzymes that adversely affect pectin quality (Mohamadzadeh et al. 2010).

The ash content of pectin extracted from banana peel was found to be1.44% (Table 8) which was in similar range to that obtained from the conventional pectin sources, apple pomace (1.96%) and citrus peel (3.46%). The current finding was in agreement with an earlier finding of varies banana peel pectin (1.43-2.76%) (Khamsucharit et al. 2018). Low ash content (below 10%) and maximum limit of ash content 10% are one of the good criteria for gel formation (Manh et al. 2019). Lower ash content means higher purity. Therefore, the ash content found in this experiment indicates the purity of the pectin.

The Anhydrouronic acid content of pectin extracted from banana peel was found to be 67.43% (Table 8), which is comparable to pectin extracted from banana peels of different varieties (34.56– 66.67%) while lower than citrus peel and apple pomace (Khamsucharit et al. 2018). The content of anhydrouronic acid (AUA) indicates the purity of the extracted pectin with a recommended value of not less than 65% for pectin used as food additives or for pharmaceutical purpose(May, 1990). In this study the highest AUA content of banana peel pectin was obtained which lies in the acceptable limits of pectin purity. This requirement has limited the potential sources of food and pharmaceutical pectin. Based on the AUA content the extracted pectin from banana peel had higher than 65% and met the criteria for commercial pectin; thus, banana peel can be an alternative source of high methoxyl pectin.

Methoxyl content is an important factor in controlling the setting time of pectin and the ability of the pectin to form gels (Constenla and Lozano, 2003). The methoxyl content of pectin extracted from banana peel was found to be 8.52% (Table 8), which is comparable to pectin extracted from pomelo peel (8.57%), passion (8.81%-9.61%), (Azad, 2014), banana peels of different varieties (3.86– 8.46%) while lower than citrus peel (9.06%) and higher than apple pomace (7.92%) (Khamsucharit et al.2018). Spreading quality and sugar binding capacity of pectin are increased with increase methoxyl content (Azad, 2014). Based on methoxyl content value in this study indicates that banana peel pectin was categorized as high methoxyl pectin (HM). HM pectin requires a minimum amount of soluble solids and a pH within a narrow range, around 2.0–3.5, in order to form gels (Azad, 2014).

The equivalent weight of pectin extracted from banana peel was found to be 956.49 which was higher than citrus peel (577) and apple pomace (551) but comparable to other varieties of banana peel pectin (943-1456) (Khamsucharit et al. 2018) and lemon pomace peel pectin (368 - 1632) (Azad, 2014). Viscosity of pectin extracted from banana peel was found to be 6.53x 10^{-3} N s m^{-2} (Table 8). The physicochemical characterizations of pectin depend mainly on the raw material source and conditions selected for isolation and purification of pectin.

Table 8 Physicochemical characterization of pectin extracted from banana peel

Moisture (%)	Ash (%)	Total anhydrouronic acid (%)	Methoxyl (%)	Equivalent weight (g/ml)	Viscosity N s m^{-2}
7.87	1.44	67.43	8.52	956.49	6.53x 10^{-3}

3.10 Validation of the optimized condition by response surface modeling

The main objectives of this study were to determine the optimal operating parameters for the maximum pectin yield and DE from banana peel using sulfuric acid. The numerical optimization of extraction of pectin was performed by using Design Expert 11.0 (Stat-Ease, Inc., Minneapolis, MN, USA, Trail version) statistical package by setting the desired goal for each process variable and responses. Pectin yield and DE were set at maximum values while the value of process variables was set in the range under study. To validate the statistical experimental strategies, the duplicate was performed under the predicted process conditions. Table 9 shows the model validation for pectin yield and DE. Optimized parameters were selected based on the highest desirability.

The maximum predicted pectin yield and DE were achieved at a temperature, pH and extraction time of 82^{o}C, 2, and 102 min, respectively. Pectin yield and DE were 15.64% (w/w) and 65.94, respectively. Model desirability (0.977) approaching unity and with low error value portrays the applicability of the model toward the responses. The validity of the estimation models built through the statistical experimental design was verified by the small differences (< 4%) between the experimental and the predicted responses. The result indicates that there was good agreement between the predicted and experimental results verified the validity of the model and confirmed the existence of the optimal point. Compared to the data obtained from the literature, the optimum extraction conditions of the pectin in the accepted limits of banana peels (Musa AAA) as reported by (Happi Emaga et al. 2008).

Table 9 Model validation for optimization of pectin yield and degree of esterification

Model	Pectin yield			Degree of esterification		
Desirability	Experimental (%)	Predicted (%)	Error (%)	Experimental (%)	Predicted (%)	Error (%)
0.977	15.85	15.64	0.69	65.95	65.94	2.30

Conclusions

In this study, the modeling, predictive and generalization capabilities of RSM and ANN models were compared for extraction of pectin from banana peel. The performance of both the models was compared based on prediction accuracy of the pectin yield and degree of estrification.The study revealed that all the three variables linearly affect the pectin yield and DE significantly compared to the combined and squared effect. Based on the values of R^2, RMSE, SEP, MAE, AAD for validation data sets, ANN model was demonstrated to be more efficient than RSM model both in data fitting and prediction capabilities. The optimum conditions of the pectin yield and DE were achieved at temperature, pH and exxtraction time of 82^{o}C, 2, and 102 min, respectively, with the desirability of 0.977. Under these conditions the maximum pectin yield and DE of 15.64% and 65.94, respectively. The extracted banana peel pectins were classified as high methoxyl type similar to citrus peel and apple pomace pectins. Based on the value of AUA content, pectin from banana peel had high purity which met the criteria for use as food additive, signifying its potential use as an alternative source of commercial pectin production.

Abbreviations

AAD: Absolute average deviation; ANN: Artificial neural network; ANOVA: analysis of variance; AUA: Content of anhydrouronic acid; CCD: Central composite design; CE: Insignificant coefficients; DE: Degree of esterification; HCl: Hydrochloric acid; H_2SO_4 : Sulfuric acid; LMP: Low methoxyl pectin; MAE: Mean absolute error; RSM: Response surface methodology; R^2 : Coefficient of determination; RMSE: Root mean square error; SEP: Standard error of prediction; Y: Pectin yield.

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Authors' contributions

The Author performed all the experiments and wrote this paper and also participated in experiment design and research supervision. The author read and approved the final manuscript.

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Availability of data and materials

All data analyzed during this study are included in this research article.

Competing of Interest

The author certifies that NO affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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