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ORIGINAL ARTICLE

Comparison of response surface methodology and artificial neural network to enhance the release of reducing sugars from non-edible seed cake by autoclave assisted HCl hydrolysis

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Abstract

In the current investigation, statistical approaches were adopted to hydrolyse non-edible seed cake (NESC) of *Pongamia* and optimize the hydrolysis process by response surface methodology (RSM). Through the RSM approach, the optimized conditions were found to be 1.17%v/v of HCl concentration at 54.12 min for hydrolysis. Under optimized conditions, the release of reducing sugars was found to be 53.03 g/L. The RSM data were used to train the artificial neural network (ANN) and the predictive ability of both models was compared by calculating various statistical parameters. A three-layered ANN model consisting of 2:12:1 topology was developed; the response of the ANN model indicates that it is precise when compared with the RSM model. The fit of the models was expressed with the regression coefficient R^2, which was found to be 0.975 and 0.888, respectively, for the ANN and RSM models. This further demonstrated that the performance of ANN was better than that of RSM.

Keywords Hydrolysis · Hydrochloric acid · Non-edible seed cake · Artificial neural network

Introduction

The rapid depletion of fossil fuels as well as the severe environmental problems associated with the increasing combustion of fossil fuels has encouraged the need toward the development of alternative fuel sources (Wang 2013). Bioenergy is the renewable source of energy using natural resources for the production of sustainable biofuels (Popp et al. 2011). Due to their low cost and abundance, lignocellulosic materials have attracted attention as renewable feed stocks for the production of biofuel as an alternative to petroleum fuel (Choi et al. 2015). Unlike other edible de-oiled seed cakes like groundnut, sunflower and soya, the use of *Jatropha* and *Pongamia* de-oiled seed cake is restricted for direct animal feeding and feed applications due to the presence of toxic phorbol esters, curcin, pongamin and other anti-nutritional factors, such as phytate, saponins and trypsin inhibitors (Rakshit et al. 2008). However, biodiesel production from *Jatropha* and *Pongamia* seed oil generates large quantity of seed cake after oil extraction from seeds. Therefore, subsequent utilization of the *Jatropha* de-oiled seed cake must be addressed to improve the outcome value of *Jatropha* and *Pongamia* biodiesel production. *Pongamia pinnata*, commonly known as honge or karanja, is a promising fast growing, leguminous non-edible oil-rich seed tree. The *Pongamia pinnata* trees are found in tropical Asia, Indian Ocean Islands, Australia and widely distributed in most of the Indian states. It can grow in drought condition and is also capable of fixing atmospheric nitrogen. After 4–6 years of maturity, *Pongamia* trees can yield 900–9000 kg seeds/hectare and hence in the present situation it is recommended to be a best source to utilize because of its ready availability of seeds (Valente et al. 2011). Carbohydrates and lignin make up a major portion of biomass samples (Sluiter et al. 2008).The proximate analysis of *Pongamia pinnata* seed cake reported by Muktham et al. (2016) is: ash 6.8%, carbohydrate 42%, protein 22.3% and lignin 29%. The cake yield

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is approximately 700–750 g per kg of *Pongamia* seeds. Various pretreatment methods can be adopted including alkali, dilute acid, steam explosion, mechanical, ammonia and fiber explosion (Mosier 2005) with different degrees of strength and weakness (Chandel and Singh 2010). To hydrolyse biomass containing cellulose and hemicelluloses to sugars, dilute or concentrated acid can be used (Wyman 1994). Acid hydrolysis is an effective method to break down polymerized cellulose with a partial removal of lignin and hemicellulose. It helps primarily in partial solubilization of hemicellulose after pretreatment and, in specific, increases the digestibility of cellulose (Hu and Wen 2008). Acid hydrolysis is much faster compared to the enzymatic method. Using concentrated acid for pretreatment of biomass, 90% yield of monosaccharides could be achieved (Heinonen et al. 2011).

In the current investigation, non-edible seed cake (NESC) of *Pongamia* was hydrolyzed using HCl to obtain reducing sugars. Release of reducing sugars is affected by various factors involved in the hydrolysis process. To achieve the release of maximum reducing sugars, a statistical approach was adopted. Initially, one factor at a time (OFAT) method was adopted to screen the significant factors and its range. A two-factor-five-level central composite design (CCD) was chosen and the optimum conditions for the release of reducing sugars obtained. ANN is a powerful technique which can model a complex non-linear process with limited prior knowledge about a process performance (Rajendran and Thangavelu 2007). In general, ANN topology consists of three layers, namely, input layer (receives input data), hidden layer and output layer (desired response). A feedforward back-propagation algorithm was used in the present study. A comparison between RSM and ANN models was done. To the best of our knowledge, this is the first research article which deals with the comparison of RSM and ANN methods for the study of release of reducing sugars from NESC by autoclave assisted HCl hydrolysis.

Methods

Raw material and chemicals

The raw material NESC (Fig. 1) of *Pongamia* was collected from a biodiesel production plant situated in the institutional campus at NMAMIT, Nitte, Udupi District, Karnataka, India, a pilot plant for biodiesel production of 50-l capacity. A biofuel demonstration and information dissemination center has been established under the supervision of the Department of Biotechnology Engineering funded by the Karnataka State Biofuel Development Board, Bengaluru. *Pongamia* seeds were procured from Mandya District situated in Karnataka State. All reagents, solvents and chemicals used were of analytical grade for the study.

Fig. 1 NESC of *Pongamia*

Processing of NESC

NESC was homogenized using a blender and mixed with distilled water in the ratio 1:2. To separate the residual oil contents from the NESC, the slurry was maintained in a convection oven at 70 °C for 45 min and cooled to room temperature. The top layer of separated oil was skimmed out. Removal of water-soluble non-structural materials may include inorganic material, non-structural sugars and nitrogenous material (Sluiter et al. 2005) Excess distilled water was drained and NESC was dried in a hot air oven at 100 °C until the moisture was removed. NESC then grounded to a fine powder and sieved through Taylor mesh number 10 having 2 mm opening to achieve uniform particle size. It was stored in closed containers under refrigeration temperature of 4 °C until further use.

Optimization of NESC hydrolysis

Selection of significant parameters and their levels by OFAT

The OFAT approach was used for the selection of significant parameters wherein the effectiveness of the parameters were checked by varying one parameter at a time while keeping the other parameters and process conditions constant. Significant physical parameters and the levels selected from the OFAT approach for the hydrolysis process are given in Table 1. OFAT was carried out to screen the significant physical parameters and the initial test range of the three variables, i.e., time (X_1; min), HCl concentration (X_2, %v/v) and weight of NESC (X_3; %w/v) for the hydrolysis processes. All the experiments were conducted in 250 mL Erlenmeyer flask with working volume

Table 1 Selected parameters and their levels for OFAT studies of NESChydrolysis

Parameter	Notation	Low/high value
Time (min)	X_1	10–60
HCl (%v/v)	X_2	2–10
NESC concentration (% w/v)	X_3	5–15

of 100 mL in triplicate using autoclave at temperature of 121 °C. Each flask containing the acid-hydrolyzed sample was neutralized using sodium hydroxide and the pH of all the samples were adjusted to around 7.0. Released fermentable sugars were quantified by the DNSA method (Miller 1959). The parameter levels at which maximum reducing sugars were released were chosen as the center point values to enhance the hydrolysis process by RSM.

Central composite design

Two experimental factors, time (X_1; min) and HCl concentration (X_2, %v/v), were selected for RSM optimization. These factors showed significant effect on the hydrolysis process during OFAT studies and their levels were optimized for maximum release of reducing sugars from NESC using the central composite design (CCD). The concentration of the released reducing sugars due to hydrolysis was considered as the response Y and designated as Y_1. Factors effecting Y_1 and its levels are given in Table 2. The CCD consisting of 12 experimental runs are given in Table 3. The experiments were conducted in random order and tabulated in standard order. The levels of other non-significant factors were kept constant at the tested center values during the OFAT studies. These optimization experiments were designed by Design-Expert (9)-software. The data of hydrolysis studies obtained was subjected to analysis using analysis of variance (ANOVA) from Design-Expert (9)-software. A second-order polynomial model was utilized to obtain the mathematical response between the response and the independent variables.

$$Y = \beta_0 + \sum \beta iXi + \sum \beta iiXi^2 + \sum_{i \neq j} \beta ijXiXj, \qquad (1)$$

Table 3 CCD for two variables and experimental RRS compared with RSM & ANN predictions

Run#	X_1	X_2	Y_{Exp}	Y_{RSM}	Y_{ANN}
1	2	30	43.66534	46.94805	43.80225
2	2	50	53.10757	53.76802	51.21441
3	6	30	32.43028	35.97799	33.57468
4	6	50	35.95618	36.88163	36.22526
5	1.17	40	55.07968	53.16298	55.9144
6	6.82	40	35.75697	33.4655	36.09035
7	4	25.85	44.7012	40.7429	44.95662
8	4	54.14	46.45418	46.20432	47.77323
9	4	40	44.04382	45.30876	45.7455
10	4	40	47.251	45.30876	45.7455
11	4	40	44.96016	45.30876	45.7455
12	4	40	44.98008	45.30876	45.7455
AARD (%)				4.170386	2.042697
RMSE				2.139162	1.078685
SEP (%)				4.858176	2.449763

X_1: Time (min), X_2: HCl concentration (%v/v)

Y_{Exp}: Concentration of RRS determined by experiments expressed as g/L

Y_{RSM}: Concentration of RRS predicted by RSM

Y_{ANN}: Concentration of RRS predicted by ANN

where Y is the dependent response and β_o, β_i, β_{ii}, β_{ij} are the estimates of the polynomial coefficients and X_i, X_j represent independent variables.

The optimization experiments were carried out in 250 mL Erlenmeyer flasks containing 100 mL of acid as per Table 2. The weight of NESC was fixed at 14%w/v. The Erlenmeyer flasks were taken out at the time (X_1) mentioned in the design (Table 3) and the estimation of released reducing sugars was done by the DNSA method.

Estimation of released reducing sugars by the DNSA method

The acid-hydrolyzed samples were neutralized to pH 7.0. The expected products of hydrolysis were glucose and saccharides. Since the saccharides are reducing sugars (Brummer et al. 2014), the concentration of the released reducing sugars (RRS) was estimated by UV–visible spectrophotometer at 540 nm using 3, 5-dinitrosalicylic acid (DNS) reagent (Miller 1959).

Table 2 Independent parameters and their coded levels for hydrolysis of NESC by acid hydrolysis optimized by CCD

Factors	Notations	Levels				
		− α	− 1	0	1	+ α
Time (h)	X_1	1.17	2	4	6	6.82
HCl concentration (%v/v)	X_2	25.85	30	40	50	54.14

Validation of the second-order polynomial model

The second-order polynomial model obtained from RSM was validated by conducting experiments at the optimized condition generated by the software.

Artificial neural network modeling

ANN is a mathematical model which provides the best possible relationship between the input and output. A feedforward back-propagation algorithm was used in the present study (Fig. 2). The non-linearity and stability of the network depend on the type of transfer function chosen for the different layers. The data entered in the input layer are passed to the hidden layer and processed by a hyperbolic tangent sigmoid transfer function called *tansig*. The data from the hidden layer are transferred to the output layer using a linear transfer function called *purelin*. The connections between the three layers and neurons are given by weights (W) and biases (b).

The neural network model was trained using Levenberg–Marquardt back-propagation algorithm and the gradient descent method was used for the adaptation (Aghav et al. 2011). The experimental CCD data were employed to train, test and validate the ANN model and MATLAB 2016a (The Mathworks Inc., USA) was used for the modeling purpose. Based on the predicted output of the ANN model, the various fitness parameters such as regression coefficient (R^2), root mean square error (RMSE), standard error of prediction (SEP) and average absolute relative deviation (AARD) were calculated and compared with the RSM model (Speck et al. 2016).

$$R^2 = 1 - \frac{\mathrm{SSE}}{\mathrm{SST}}, \tag{2}$$

where SSE is the sum of squared error and SST is the sum of squared total.

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{i=1}^{n}\left(Y_{i,\mathrm{exp}} - Y_{i,\mathrm{pred}}\right)^2}{n}}, \tag{3}$$

where "i" represents the experiment number, "n" is the total number of experiments, "Y" the concentration of RRS, and Y_{exp} and Y_{pred} represent the concentration of the experimental and predicted RRS, respectively.

$$\mathrm{SEP}(\%) = 100 * \frac{\mathrm{RMSE}}{Y_{\mathrm{ave}}}, \tag{4}$$

where Y_{ave} represents the average concentration of RRS.

$$\mathrm{AARD}(\%) = 100 * \frac{\sum_{i=1}^{n}\left[\frac{Y_{i,\mathrm{exp}} - Y_{i,\mathrm{pred}}}{Y_{i,\mathrm{exp}}}\right]}{n}. \tag{5}$$

.

Results

Selection of significant parameters through OFAT

The concentration of the acid was varied from 2, 4, 6, 8 and 10% (v/v) by keeping other factors constant ($X_1 = 20$ min and $X_3 = 5$%w/v). Autoclave assisted HCl hydrolysis was carried out at 121 °C. The maximum reducing sugar of 20.75 g/L was released at 4% (v/v) acid (Fig. 3). The weight of NESC was varied from 5, 10 and 15% (w/v) by keeping the other factors fixed ($X_2 = 4$% (v/v) and $X_1 = 20$ min) (Fig. 4). At 14%w/v, maximum sugar of 39.96 g/L was released. Beyond

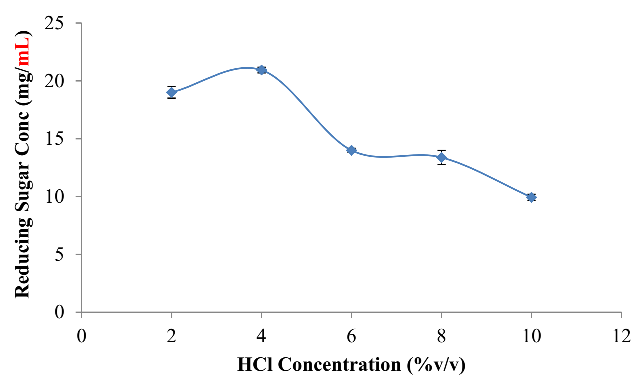
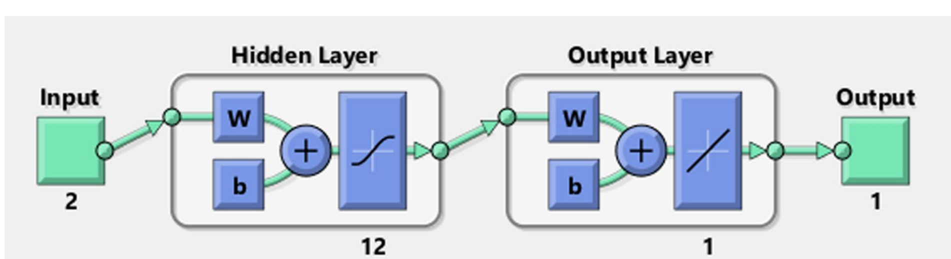


Fig. 3 Release of reducing sugars on acid hydrolysis of NESC at different concentrations of HCl. Factors kept constant were time and weight of the NESC ($X_1 = 20$ min and $X_3 = 5$%w/v)

Fig. 2 Structure of ANN used in the present study W: weights, b: biases



14% w/v of NESC concentration, liquid was found to be absorbed by NESC even after a well mixed condition, resulting in the reduction of hydrolysate volume. Hence, X_3 was fixed at 14%w/v for the rest of the experiments. The time was varied from 10, 20, 30, 40, 50 and 60 min by keeping other factors constant (X_3 = 14%(w/v) and X_2 = 4% (v/v)). The maximum reducing sugar of 50.69 g/L was released at 40 min. Thus, 40 min (X_1) was considered as a significant value in this study (Fig. 5). The center points of the OFAT analysis were further used for the central composite design (CCD).

Central composite design

The influence of time (X_1) and concentration of acid (X_2) on the release of reducing sugars was determined by CCD results, as shown in Table 3. This table represents the experimental values for RRS concentration by acid hydrolysis (Y_{Exp}) at different combinations of the independent parameters along with RSM-predicted (Y_{RSM}) and ANN-predicted responses (Y_{ANN}).

The regression equation for the release of reducing sugars by autoclave assisted acid hydrolysis of NESC, as a function of the two independent variables (X_1 and X_2) and their linear and quadratic interactions, is represented as follows:

$$Y_1 = 21.01039 + 1.470634 * X_1 - 0.24932 * X_1^2 + 1.222971 * X_2 - 0.00918 * X_2^2 - 0.07395X_1 * X_2 \quad (6)$$

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Table 4 indicates the values obtained by ANOVA for the release of reducing sugars on acid hydrolysis of NESC. Basically, a smaller p value stands for higher influence and only factors having p values < 0.05 can be considered as statistically important (Guo et al. 2009).

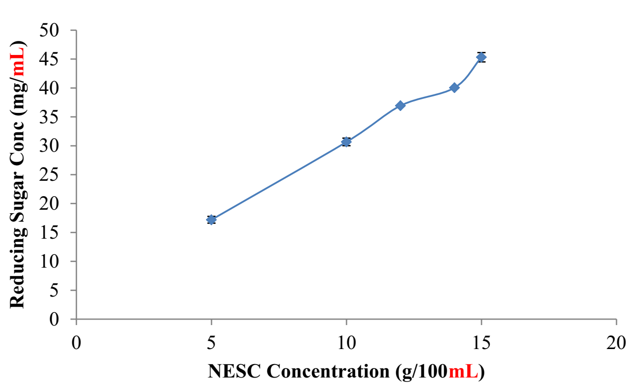

Fig. 4 Release of reducing sugars on acid hydrolysis of NESC at different NESC concentrations. Factors kept constant were HCl concentration and time (X_2 = 4% (v/v) and X_1 = 20 min)

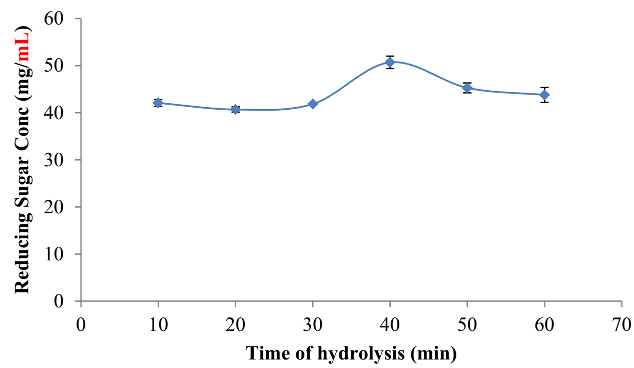

Fig. 5 Release of reducing sugars on acid hydrolysis of NESC at different time intervals. Factors kept constant were NESC and HCl concentration (X_3 = 14% (w/v) and X_2 = 4% (v/v))

It also shows that the linear effects of the independent variable time showed highly significant effect on the reducing sugar concentration released by acid hydrolysis of NESC. In addition, the low value of P for the model affirmed the significance of the model.

The response surface graph for RRS as a function of time and HCl concentration is depicted in Fig. 6. As the time of autoclaving was increased, the release of reducing sugars also increased. The increasing trend of reducing sugar release may be probably due to the chemical hydrolysis of starch with the increase in acid concentration.

The optimized levels of variables X_1 and X_2 for the maximum release of reducing sugars by hydrolysis were determined by desirability profiles (Fig. 7). Based on the desirability plots, the optimized factors for releasing maximum sugars (Y) were found to be 1.17% of HCl and 54.14 min of autoclave treatment. Further experiment was conducted

Table 4 ANOVA table for release of reducing sugar on acid hydrolysis of NESC as affected by time and HCl concentration

	SS	df	MS	*F*	*p*
Model	**436.3717**	**5**	**87.27434**	**9.536067**	**0.0080***
X_1 (L)	**387.9908**	**1**	**387.9908**	**42.39398**	**0.000626***
X_1 (Q)	6.364988	1	6.364988	0.695473	0.436245
X_2 (L)	29.8271	1	29.8271	3.259071	0.121062
X_2 (Q)	5.388496	1	5.388496	0.588776	0.471998
X_1 by X_2	8.750754	1	8.750754	0.956155	0.365921
Error	54.91216	6	9.152026		
Lack of fit	49.31026	3	16.43675	8.802425	0.0536
Total SS	491.2838	11			

Bold values indicate the significant parameter

X_1 Time (min), X_2 HCl concentration (%v/v), *L* Linear, *Q* Quadratic

*Values less than 0.05 indicate significance at 95% confidence interval

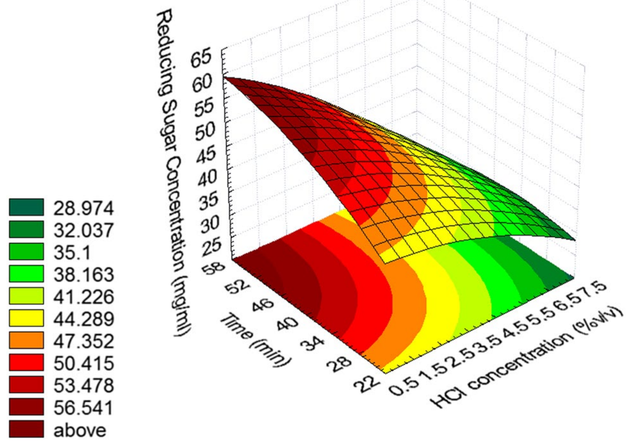

Fig. 6 Response surface plot showing the effect of acid concentration and time duration on the acid hydrolysis of NESC for the release of reducing sugar

at optimized condition and the concentrations of reducing sugars were found to be 53.03 g/L, respectively. Previous reports have shown that 8.71 g/L glucose was released from *Pongamia pinnata* de-oiled seed cake using 2% HCl at 100 °C (Muktham et al. 2016). The comparative yield of reducing sugar data (Table 5) clearly reveals that significantly higher reducing sugar yield of 0.378 g per gram of seed cake was achieved from the current study.

ANN modeling

The data from the CCD matrix were divided into training data (70%), validation (15%) and testing data (15%). The Levenberg–Marquardt algorithm was adopted for training and the training process, which was stopped when the mean squared error (MSE) reached a minimum value. The number of hidden layers and of hidden neurons plays a significant role in ANN modeling. In the present study, it was found that one hidden layer with 12 hidden neurons was optimum (MSE = 1.16) and the selected optimal structure of the ANN topology was 2:12:1.

Comparison of RSM and ANN

The ANN model developed was used to simulate the experimental data achieved through the central composite design and compared with the RSM-predicted value (Y_{RSM}) and ANN-predicted output (Y_{ANN}) (Table 3).The correlation between the predicted responses and experimental values from the RSM and ANN model for maximum RRS is shown in Figs. 8 and 9, respectively. The performance of RSM and ANN models was statistically measured. The predicted output based on this ANN topology and the various fitting parameters R2, RMSE, SEP and AARD were calculated and are shown in Table 6.

The regression coefficient (R^2) was also determined to evaluate the goodness of fit of the model. It is evident from Table 6 that the ANN model has low values of RMSE (1.07), SEP (2.44) and AARD (2.04) when compared with the RSM model. The R^2 value of the RSM model was only 0.8882,

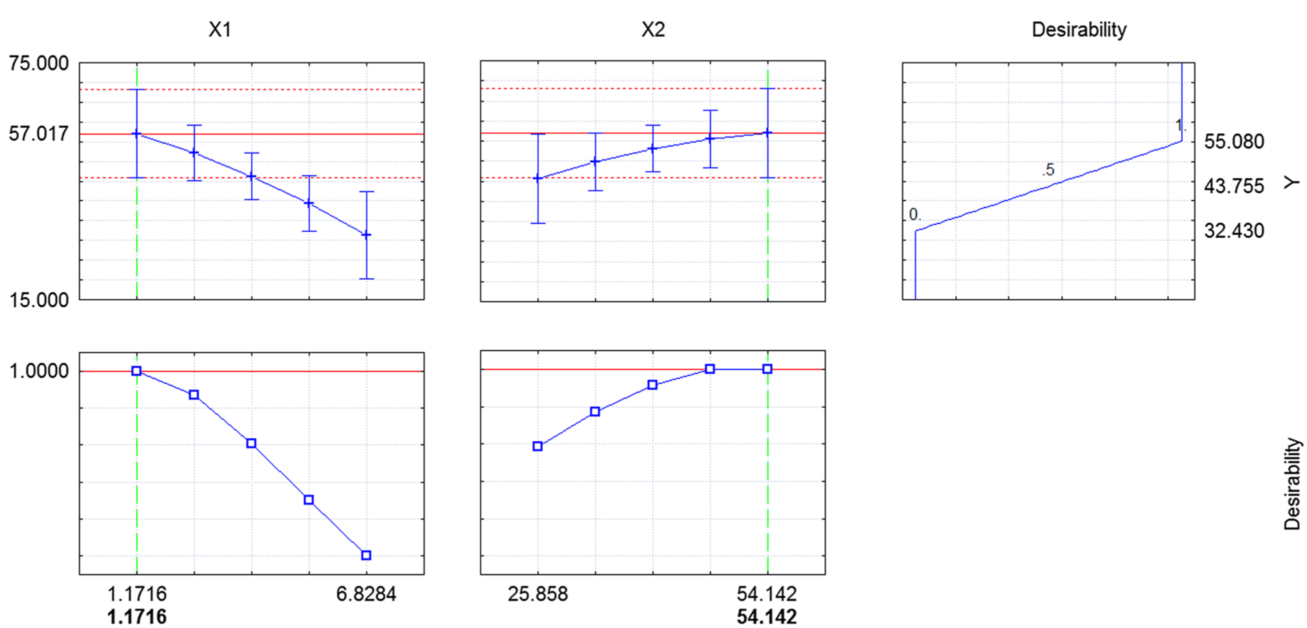

Fig. 7 Profiles for the predicted pretreatment process and the desirability levels for different parameters for optimum hydrolysis

Table 5 Comparison of reducing sugar yield

	Feed	Reaction conditions	Reducing sugar yield	Reference
1	De-oiled *Pongamia* seed cake	Acid: 2% HCl Temperature:100 °C Time: 60 min	0.132 g of glucose per gram of seed cake.	(Muktham et al. 2016)
2	*Pongamia* seed cake	Acid:7.5% H_2SO_4 Acid to seed cake weight ratio: 15 Temperature: 120 °C Time: 60 min	0.245 g of glucose per gram of seed cake.	(Radhakumari et al. 2014)
3	*Pongamia* seed cake	*1st step: Pretreatment of the residues* Acid: 0.5% H_2SO_4 Temperature:121 °C Time: 90 min *2nd step: Acid hydrolysis of the pretreated residues* Acid: 5% H_2SO_4 Temperature:50 °C Time: 70 h	0.034 g of glucose per gram of seed cake.	Doshi and Srivastava 2013
4	*Pongamia* seed cake	Acid: 1.17% of HCl Temperature:121 °C Time:54.14 min	**0.378 g** of reducing sugars per g of seed cake.	Current study

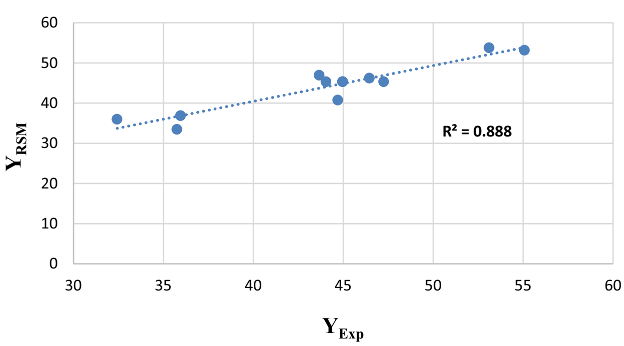

Fig. 8 Scatter plot of RSM-predicted response versus experimental values of RRS

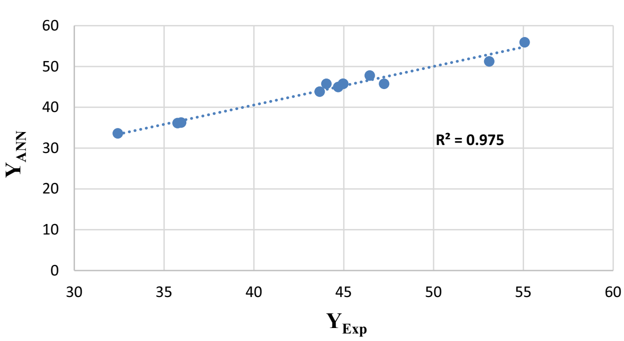

Fig. 9 Scatter plot of ANN-predicted response versus experimental values of RRS

Table 6 Comparison of RSM and ANN predicted output

	DOE	ANN
AARD(%)	4.170386	2.042697
MSE	4.576013	1.16356
RMSE	2.139162	1.078685
SEP (%)	4.858176	2.449763
R^2	0.888227	0.975342

AARD average absolute relative deviation, *MSE* mean square error, *RMSE* root mean square error, *SEP* standard error of prediction, R^2 Regression coefficient, *DOE* design of experiment/RSM

but for ANN model, the R^2 value was observed to be 0.9753, close to 1 which indicated the reliability and suitability of the ANN model. Also, only 0.111% and 0.025% of the total variations was not explained by the RSM and ANN model, respectively.

These findings corroborate that the deviation between the experimental and predicted values of the ANN model was very less as compared to the RSM model (Speck et al. 2016). The results clearly suggest that both models fit well with the experimental data. However, ANN was found to have better predictive power with a more realistic approach to experimental data when compared with RSM. The advantage of the RSM is to identify the significant factors and provide a regression equation for the prediction, thereby reducing the complexity of the problem in comparison with ANN. The only limitation of the RSM is that it assumes only quadratic non-linear correlation and this technique requires suitable ranges for each factor involved in the response(s)

under consideration. However, ANN can capture almost any form of non-linearity and easily overcome the limitations prevailed upon by RSM (Desai et al. 2008).

Conclusions

Acid hydrolysis was found to be a potential method for the release of reducing sugars from NESC. RSM was found to be an efficient methodology for the determination of conditions leading to effective hydrolysis of NESC using HCl. However, the ANN model predicted the response in a better way. The significance of this work is that it includes the use of cheap chemical aides (HCl) which in turn makes the process of biochemical production cost-effective. Moreover, the optimized conditions obtained from this study can be further used for large-scale hydrolysis of NESC to ensure the maximum release of reducing sugars before using it as a substrate for biochemical production.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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