

**OPTIMIZATION OF XYLOSE PRODUCTION BY ALKALINE HYDROLYSIS OF WATER
HYACINTH BIOMASS USING RESPONSE SURFACE METHODOLOGY AND ARTIFICIAL
NEURAL NETWORK**Premsunder Ghosh¹, Subhabrata Das³, Amit Ganguly², Kuntala Das¹ and Pradip Kumar Chatterjee^{2*}¹Department of Biotechnology, National Institute of Technology Durgapur, Durgapur 713209, India²Thermal Engineering, CSIR Central Mechanical Engineering Research Institute, Durgapur 713209, India³Department of Chemical and Biomolecular Engineering, National University of Singapore,
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ABSTRACT: This paper entails a comprehensive study on production of xylose by alkali pretreatment of water hyacinth biomass. Artificial Neural Network (ANN) and Response Surface Methodology (RSM) were used for optimization in order to enhance xylose yield by alkali pretreatment using NaOH. In the present study, water hyacinth (*Eichhornia crassipes*), a fast growing, aquatic lignocellulosic weed was exploited for the production of xylose. The application of RSM in the study for xylose yield by alkali hydrolysis process utilizing optimization and modeling facilitated the analysis of interaction effects of the process variables on the response. Artificial Neural Network modeling was also studied to validate and compare the results obtained from RSM. The optimized experimental result of the influencing parameters are NaOH concentration of 3%, agitation speed of 130 rpm, treatment time of 11 min, treatment temperature of 60 °C and soaking time of 3h which produced a xylose yield of 84.56 mg/g of dried biomass. The yield dropped drastically on increasing the temperature above a certain point which may be due to the degradation of xylose. The analysis was done by Design Expert 9.0.3 software and Matlab, R2009a (The Math Works, Inc., MA, USA) for optimization by Response Surface Methodology and Artificial Neural Networking.

Keywords: Water hyacinth, Pretreatment, Xylitol, Response Surface Methodology, Artificial Neural Networking

INTRODUCTION

Water hyacinth (*Eichhornia crassipes*), is considered as a noxious aquatic weed in different parts of the world. Owing to its exceptionally fast growing capacity it depletes the nutrient and oxygen content of the water system quite rapidly, which affects the flora and fauna of the ecosystem. The growth rate is about 17.5 metric tonnes per hectare per day under suitable conditions (Mehmood et al., 2009). This aquatic weed is found to be doubtful and debatable for various reasons, however its high content of hemicellulose (30–55% of dry weight) (Ganguly et al., 2012). It has the potential for the production of large quantities of biomass of aquatic floating species, which can be used as an untapped source for the production of xylose. Moreover, using low cost raw matter such as cellulose from agricultural residues is a boon to the environment (Rubio et al., 2012). For maintaining a self sustaining society, the possibility of converting water hyacinth for the production of xylose is a promising aspect. The efficiency of hydrolysis of pre-treated substrate depends on several process parameters such as NaOH concentration, soaking time, agitation speed, treatment time, treatment temperature and optimization of the factors affecting pre-treatment is necessary for achieving desired yields. This process aims to remove lignin and various uronic acid substitutions in hemicellulose which lowers the enzyme accessibility to carbohydrate polymers (Silverstein et al., 2008). NaOH pre-treatment is the most commonly studied alkaline pre-treatment process (Kumar et al., 2009) and it causes swelling of lignocellulosic materials which allow the separation of structural linkages between lignin and carbohydrate polymers, decreases cellulose crystallinity and cause lignin disruption which might lead to an increase in internal surface area (Fan et al, 1987).

The conventional approach is laborious and time consuming. Moreover, optimized conditions are seldom assured. To overcome these limitations the two most efficient methods for optimization of data in the present time are Response surface methodology and Artificial Neural Network

In our present study, Response surface methodology (RSM) is a Statistical analysis based approach implemented successfully to optimise the percentage of xylose yield% (Rao et al., 2000; Dutta et al, 2004; Rama Mohan Reddy et al., 2003). RSM fits the responses derived from design of experiments (DOEs) to a polynomial function. RSM includes statistical modelling to determine the interaction effects of the factors on the response, significance of the factors, evaluate the combined effect of two factors at a time on the response and finding out the optimum condition.

In the last twenty years, Artificial Neural Network (ANN) on the other hand evolved as one of the most efficient and attractive tool for modelling non-linear multivariate systems (Bourquin et al., 1998). The major advantage of ANN is that it is generic in structure and its ability to learn and train itself using historical data. ANN excels over RSM over the following points: (I) ANN is superior in compared to RSM (Basri et al., 2007) as it does not require preliminary information regarding a suitable objective function for fitting the response (ii) ANN can be used to universally approximate almost all kinds of non-linear functions whereas RSM is only competent for only quadratic non-linear correlation. (iii) ANN is more accurate and has a better optimization efficiency than RSM was incorporated recently in the prediction of values while calculating the amount of zinc (Moghaddam et al., 2011).

In this study, the optimal condition obtained empirically was verified experimentally and compared to determine the efficiency of both RSM and ANN technique for xylose yield from water hyacinth biomass.

MATERIALS AND METHODS

Preparation of water hyacinth

Fresh water hyacinth with long stem was collected from a natural pond inside the premises of CSIR-CMERI, Durgapur. The water hyacinth was thoroughly washed several times with tap water to remove adhering dirt, chopped into small pieces of size 2-3 cm (approx), and grinded to make a paste of water hyacinth biomass. The fresh biomass was used for NaOH hydrolysis.

Preparation of hemicellulose alkali hydrolysate

A sample of fresh water hyacinth (8 gm) was mixed with 2%, 3%, 4% (v/v) of sodium hydroxide to a final volume of 30 ml. The mixtures were soaked for 2, 3, and 4 h respectively. The alkali hydrolysis reactions were carried out in an incubator shaker in the temperature range of 30°C, 50°C, 70°C & agitation speed varying from 100 rpm, 130rpm, 160 rpm respectively after which the hydrolysate was autoclaved at 121 °C with treatment time varying from 7-15 min. The hydrolysate was filtered using Whatman paper no. 1 to remove the unhydrolysed material.

Determination of xylose content by Phloroglucinol assay

Xylose content was determined using the Phloroglucinol assay with the hydrolysate obtained from alkali hydrolysis. The colouring reagent mixture was heated in water bath and rapidly cooled to room temperature before measuring in a THERMO UV1 100 Double Beam scanning Spectrophotometer at 554 nm.

Experimental Modeling and Optimization Strategy

Artificial Neural Networking

Artificial neural network (ANN) is an attractive modelling approach which can serve as an excellent alternative to Response Surface Methodology (RSM) for representing non linear relationships between variables, as well as to solve multivariate regression problems. There is a basic difference even in case of terminology between ANN and RSM, where operating factors for RSM are termed as inputs in ANN, the experimental responses as targets and output indicate the ANN model predicted responses.

ANN architecture is in general made of highly interlinked bundles of elements known as neurons. The connections in between the neurons are defined by weights (w) and biases (b). The neurons are controlled by a defined transfer and a summing function. The most commonly used transfer functions for solving complex linear and non-linear regression problems in ANN architecture are: linear transfer function (purelin), log-sigmoid transfer function (logsig) and hyperbolic tangent sigmoid transfer function (tansig) the network topology basically demonstrates the interconnection between the neurons. Normally the neurons are arranged in groups, known as layers. A multi-layer neural architecture consists of input, output and hidden layer. Multi-layer feed-forward neural network also known as multiplayer perceptron.

MLP helps in effectively managing the neural architecture while solving non-linear regression models. In this study, the predictive model was built taking soaking time (h), concentration of NaOH, agitation speed (rpm), Treatment temperature and treatment time (min) as the input parameters and % yield of xylose (mg/g) as the output for the model. In the MLP network system, the data flows from the input layer domain of the hidden layer in the forward direction. The function of the input layer is to present the scaled input data to the hidden layer through weights. After this, the hidden layer performs two basic functions. First, the neurons in the hidden layer sum up the weighted inputs along with the biases as per the following equation:

$$\text{sum} = \sum_{i=1}^n x_i w_i + \theta \quad \text{--- (1)}$$

Where, w_i ($i=1, n$) represents the connection weights, θ is defined as the bias and x_i signifies the input parameter. After that, an activation function passes the weighted output, resulting shifts in non-linearity of input dataset. The logistic output function used in this study can be represented by the following equation:

$$f(\text{sum}) = \frac{1}{1 + \exp(-\text{sum})} \quad \text{--- (2)}$$

After following the two operations, the output data set produced by the hidden layer becomes the input for the output layer. The final predicted response by the ANN model is generated by the output layer following the same set of operations as in the hidden layer mentioned above.

Based on this, the calculated predicted response by the model and the actual experimental response an error function are developed. The next most important aspect of ANN modelling is the network training. Training is an iterative process, which involves the weights and biases of the neural architecture to minimise the developed error function. This is done by bringing the predicted output as close as possible to the experimental output. The most widely used error functions are mean-squared-error (MSE) and root-mean-squared-error (RMSE) which can be defined as follows:

$$\text{MSE} = \sum_{i=1}^N \sum_{n=1}^M (y_n^i - \hat{y}_n^i)^2 \quad \text{--- (3)}$$

$$\text{RMSE} = \sqrt{\sum_{i=1}^N \sum_{n=1}^M (y_n^i - \hat{y}_n^i)^2} \quad \text{--- (4)}$$

where, N refers the number of patterns that were tested during the training, M means the number of available output nodes; i indicates the pattern of input index and finally y_n^i and \hat{y}_n^i represents the desired experimental target and the model predicted output of the nth node of the output respectively. The construction of ANN models was performed in Matlab, R2009a (The MathWorks, Inc., MA, USA).

Table 1: Experimental range and levels of independent process variables

Name	Low	High
Soaking time(h)	2	4
Conc. Of NaOH%(v/v)	2	4
Agitation speed(rpm)	100	160
Treatment time(min)	7	15
Treatment temp(oC)	50	70

Response Surface Methodology (RSM)

RSM is an empirical statistical modelling technique, which builds a proper experimental design and then use that design to solve multivariate equations simultaneously. Based on the design of experiment, we have to perform the experiments to obtain the quantitative experimental responses. The quantitative experimental responses are used to perform multiple regression analysis to fit to the quadratic model of RSM. For our study RSM was used to develop an experimental design and then to evaluate the optimum condition for alkaline hydrolysis to maximize yield of xylose. Central composite rotatable design (CCRD) was implemented to investigate the interaction effects of the five independent process variables. Independent variables selected for our process are: soaking time, concentration of NaOH, agitation speed, treatment time treatment temperature. The input variables are scaled to coded levels based on the following equation:

$$x_i = \frac{X_i - X_{cp}}{\Delta X_i}, \quad i=1,2,3,\dots,k \quad \text{--- (5)}$$

where 'x_i' is a dimensionless parameter of the independent process variable, 'X_i' indicates the real value of the independent variable, 'X_{cp}' implies the real value of an independent variable at the centre point and 'ΔX_i' represents the step change in the real value of the variable 'i' upon an unit change in the dimensionless value of the variable 'i'. To gauge the variability in the measurements, all the experiments were performed in triplicates.

To check the absence of bias in-between several sets, experiments were repeated in centre points. A second order polynomial equation (eq.6) is used to estimate the relationship between the independent and the experimental responses:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i X_i + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} X_i X_j \quad \text{--- (6)}$$

where 'Y' is the predicted response, β₀ is constant, β_i represents the linear co-efficient, β_{ii} implies the co-efficient of the squared terms, β_{ij} expresses the co-efficient of the cross term products and 'k' indicate the number of independent factors considered for the optimisation study.

The second-order-polynomial co-efficient were calculated using Design Expert Version 9.0.3. The software was used to estimate the responses of the dependent variable and find out optimization efficiency. The range of the independent parameters for the alkaline hydrolysis and the experimental design setup of the RSM along with experimental responses are defined in Table 1 and 2 respectively.

RESULTS AND DISCUSSIONS

Chemical composition of water hyacinth

Chemical composition of the water hyacinth (root, leaf and stem) was determined individually. The result indicates that the composition of the water hyacinth varies in each plant body. It was found that the stem contains relatively more hemicellulose compared to leaf and root. Hence, root was excluded further from our study. The chemical composition of root, stem and leaf are shown in Figure 1. Stem biomass of water hyacinth was considered for pretreatment as it contained high hemicellulose which can be converted into xylan monomers.

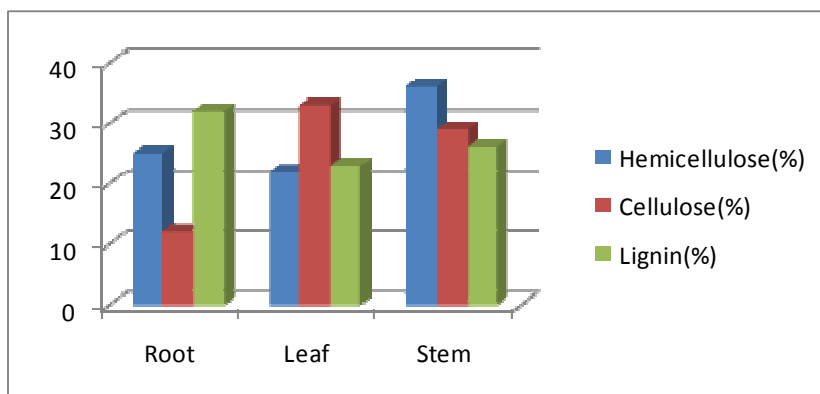


Figure 1: Chemical composition of water hyacinth

Alkali hydrolysis

Alkaline pre-treatment with sodium hydroxide is a viable, low-cost option for modifying the structure of lignocelluloses prior to hydrolysis and fermentation of the carbohydrate fractions. The advantage of using alkali over acidic methods is the milder conditions (near or at atmospheric temperature and pressures) and the removal of the lignin fraction without the degradation of the other major constituents. Alkaline pre-treatment can play an important role in the conversion of lignocelluloses. Lignin is the main component of lignocelluloses affected by NaOH pre-treatment. The alkaline pre-treatment reacts with the ester bonds linking the lignin to the hemicellulose in the LCC network. As these bonds are broken, the LCC networking is disrupted, allowing lignin components to be solubilised. Hemicellulose, with its branched and somewhat irregular structure, tends to be the most sensitive of the three lignocelluloses fractions to changes in pre-treatment conditions. The attraction of alkali pre-treatment is the removal of acetyl and various uronic acid substitutions on hemicellulose that lower the accessibility of the enzymes to the hemicellulose and cellulose surface.

Table 2: Comparative result of observed and predicted results of xylose yield as a response using RSM and ANN model

Run	Soaking time(h)	Conc. of NaOH % (v/v)	Agitation speed (rpm)	Treatment time (min)	Treatment temp (oC)	Experimental Xylose Yield (mg/g)	RSM Predicted xylose yield (mg/g)	ANN predicted xylose yield (mg/g)
1	2	2	100	15	70	64.2717	63.9744	64.3817
2	2	4	100	15	50	52.691	52.7879	52.801
3	3	3	130	11	60	83.1772	82.4295	83.2872
4	4	4	160	7	70	53.899	54.1939	54.01
5	3	5.37841	130	11	60	50.9148	50.4679	51.0248
6	3	3	130	11	60	84.5643	82.4295	84.675
7	3	3	58.6476	11	60	53.5408	53.681	53.66
8	3	3	201.352	11	60	46.7326	45.4646	46.6129
9	4	2	160	15	70	62.5996	62.5211	62.7026
10	2	2	160	15	50	46.6122	47.1585	46.8426
11	4	4	160	7	50	50.4057	50.7952	50.5157
12	2	2	160	15	70	64.3048	65.1235	64.4148
13	0.621586	3	130	11	60	56.4529	55.6387	56.563
14	2	2	100	7	50	43.4688	43.9535	43.5788
15	4	4	160	15	70	56.12	56.8159	56.25
16	3	3	130	11	60	81.4277	82.4295	81.5377
17	3	3	130	11	60	83.3439	82.4295	83.454
18	2	4	160	7	70	55.2102	56.0427	55.3201
19	2	4	100	15	70	60.13	61.3026	60.25
20	3	3	130	11	83.7841	59.3361	58.6769	59.46
21	3	3	130	11	60	83.3467	82.4295	83.513
22	4	4	160	15	50	50.6016	50.2277	50.731
23	2	2	100	7	70	57.5257	58.2495	57.67
24	3	3	130	11	36.2159	34.31	33.8414	34.223
25	2	4	160	15	50	51.5905	51.9953	51.71
26	3	0.621586	130	11	60	47.4276	47.7467	47.54
27	3	3	130	11	60	81.1315	82.4295	81.26
28	4	2	160	7	50	44.8721	44.9943	44.79
29	4	2	160	7	70	57.0172	57.2599	57.17
30	4	2	100	7	50	51.9564	51.6942	52.14
31	3	3	130	11	60	81.1315	82.4295	81.26
32	4	2	160	156	50	46.2241	47.066	46.36
33	2	4	160	15	70	61.4515	61.0935	61.35
34	2	2	160	7	50	42.6771	42.6578	42.71
35	4	2	100	15	70	66.7156	66.7762	66.825
36	4	4	100	7	50	58.8439	58.8534	58.904
37	4	4	100	15	50	56.0936	52.8	56.11
38	3	3	130	1.48634	60	53.4019	52.8924	53.36
39	2	2	100	15	50	51.5711	51.8009	51.70
40	2	4	160	7	50	49.7263	50.1339	49.84
41	4	2	100	7	70	63.4801	63.2503	63.3103
42	2	2	160	7	70	57.4335	57.40	57.46
43	4	4	100	15	70	62.4292	62.601	62.661
44	2	4	100	7	50	52.7879	52.553	52.603
45	5.37841	3	130	11	60	59.757	60.01	59.867
46	3	3	130	20.5137	60	59.0257	59.595	59.1357
47	4	4	100	7	70	61.7724	61.67	61.8324
48	2	4	100	7	70	58.2169	59.251	59.311
49	2	2	100	15	50	46.4891	46.751	46.811
50	3	3	130	11	60	82.42.95	82.103	82.163

The present study aims at applying a simple and reliable pre-treatment process with dilute alkali hydrolysis for the conversion of water hyacinth for maximum xylose yield. Estimations of xylose which is a major fermentable sugar have been performed by spectrophotometric determinations. Moreover, different parameters like treatment temperature, treatment time, concentration of the NaOH, agitation speed, soaking time of fresh biomass have been considered and detailed studies were carried out with water hyacinth to establish the variation of xylose yield. However, classical approach does not depict the combined effect of all the process parameters. It is also time consuming and requires a number of experiments to determine the optimum levels, which may be unreliable thereby elevating the overall cost of the process. These limitations of the classical method can be eliminated by optimizing all the process parameters collectively by statistical experimental design such as Response Surface Methodology (RSM). RSM offer certain advantages like a higher percentage yield, reduced process variability, closer confirmation of output in response to nominal and target achievement. Hence, the combined effects of various parameters for the xylose yield by alkali hydrolysis from water hyacinth, viz. Treatment temperature, concentration of NaOH, treatment time, soaking time, agitation speed was done using Central Composite Response Design Model in Response Surface Methodology (RSM) in Design Expert Version 9.0.3. The results obtained from RSM are compared through ANN. Even with the limited number of experiments with ANN predicted model was highly accurate and better generalization capability than RSM.

ANN Modelling and Optimization

To construct and train the neural network model the set of data provided in the design of experiment (Table 2) have been used. A Feed Forward Network Architecture as shown in Figure 2 of five Inputs namely Soaking Time, Concentration, Agitation Speed and Treatment Time, Treatment Temperature. 12 Neurons were present Hidden Layer. Among the total data set, 80% of the total experimental data set was used for training the network while the rest 10% was kept for testing and 10% for validation of the model. The Output of the model was the Total Xylose Yield % of alkali pre-treated WHB.

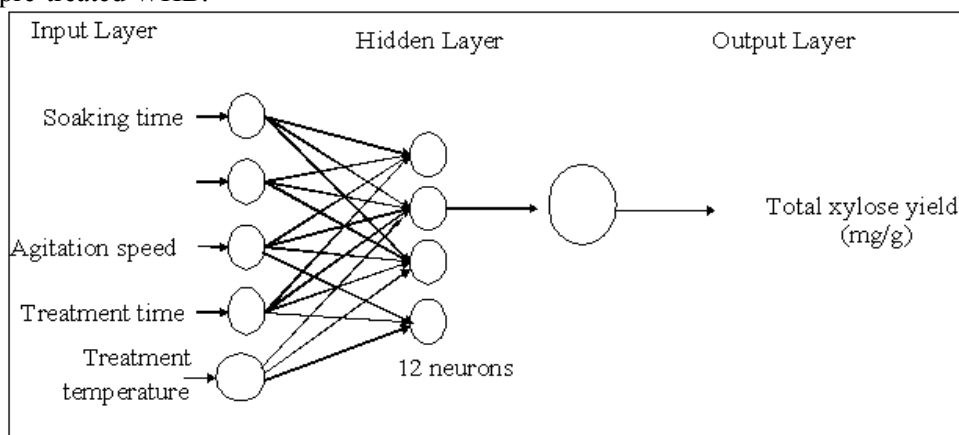


Figure 2: Finalized Neural Network Architecture used for prediction of xylose yield by water hyacinth biomass

The Neural Network Toolbox in MATLAB has been used for building the artificial neural network model. The performance plot Figure 3 tells about the fitness and best validation performance of the data set.

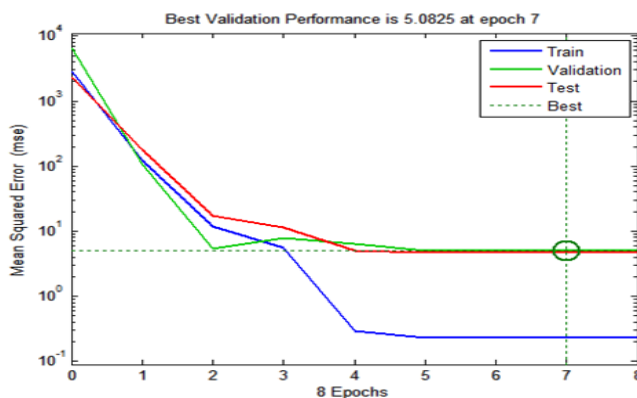


Figure 3: Development of MSE During training phase of ANN MODEL

From the regression plot, Figure 4, we observe that all points are present close to the straight line implying ANN predicted results were in close agreement with the experimental results providing a suitable fitting. A very high value of correlation co-efficient indicates a good agreement between the experimental and predicted data. The y axis represents the xylose yield (experimental data) and the x axis represents xylose yield (simulated results) respectively in Figure 4.

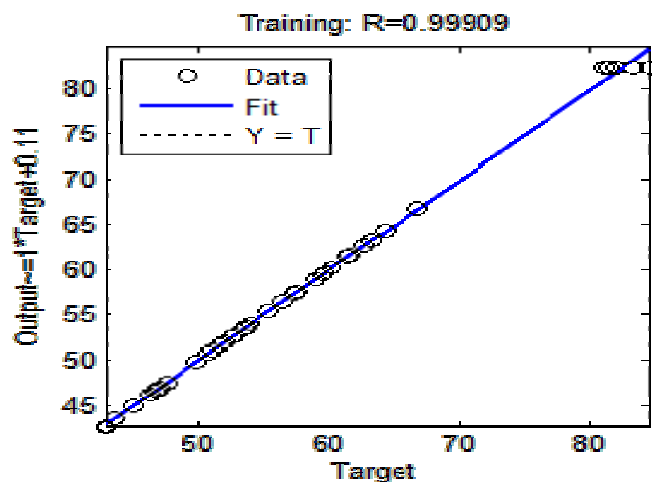


Figure 4: The plot of experimental data vs. ANN model predicted data

Response Surface Methodology

50 experiments were designed for the estimation of the combined effect of the independent factors-soaking time, concentration of NaOH, agitation speed treatment time and treatment temperature affecting the xylose % yield. In order to perform the regression analysis and obtain the co-efficient of the second order polynomial involving the independent parameters of the xylose% yield Design Expert Version 9.0.3 (Stat Ease, USA) was used. Table 3 summarises results obtained for the ANOVA study of the second order response surface model. The statistical significance of the model was determined by F-test ANOVA. The results in Table 3 suggest that the independent parameters viz. soaking temperature, soaking time, agitation speed and treatment time, have a significant influence on the yield of total xylose during alkaline hydrolysis. The non-significant value of lack of fit and a significant value for model proved validity and significance the quadratic model. For understanding the interaction effect between the process parameters ANOVA study was very helpful. Our developed model for RSM proved to be highly significant due to its high Fisher's F-value (458.86) with a low probability value ($p < 0.0001$) [13]. Regression co-efficient was determined to additionally understand the goodness of fit. Regression co-efficient came out to be $r^2 = 0.9968$, implying that 99.68% of the total variance was explained by this RSM model. The predicted R-squared (0.9916) and the adjusted R-squared (0.9947) were in reasonable argument with each other indicating the efficiency of the model. The residual variation is measured using co-efficient of variance (CV) relative to the size of the mean. A very low (1.52%) value of CV implies a sufficient precision and reliability on the experimental results (Lazic et al., 2004). Predicted Residual Sum of Squares (PRESS) is another check to express the fitness of the model and the experimental value. The smaller the PRESS statistic, the better the model fits the data points. In the present study the calculated value of PRESS is 62. The model shows the values of standard deviation and means as 58.84 and 0.89 respectively. The corresponding second order model obtained after ANOVA study:

$$\begin{aligned} \text{Xylose Yield} = & -482.96847 + 39.64344 * \text{Soaking time} + 54.49865 * \text{Concentration} \\ & + 1.72042 * \text{Agitation Speed} + 5.97846 * \text{Treatment Time} + 8.77701 * \text{Treatment Temperature} - 0.41879 * \text{Soaking} \\ & \text{time} * \text{Concentration} - 0.045035 * \text{Soaking time} * \text{Agitation Speed} - 0.15180 * \text{Soaking time} * \text{Treatment Time} - \\ & 0.062751 * \text{Soaking time} * \text{Treatment Temperature} - 0.011319 * \text{Concentration} * \text{Agitation Speed} - 0.16495 * \\ & \text{Concentration} * \text{Treatment Time} - 0.22167 * \text{Concentration} * \text{Treatment Temperature} + 4.09398\text{E-}003 * \text{Agitation} \\ & \text{Speed} * \text{Treatment Time} + 3.99752\text{E-}004 * \text{Agitation Speed} * \text{Treatment Temperature} + 0.019934 * \text{Treatment Time} * \\ & \text{Treatment Temperature} - 4.37197 * \text{Soaking time}^2 - 5.97897 * \text{Concentration}^2 - 6.45366\text{E-}003 * \text{Agitation Speed}^2 \\ & - 0.29246 * \text{Treatment Time}^2 - 0.063941 * \text{Treatment Temperature}^2 \end{aligned}$$

Table 3: Analysis of Variance of quadratic model for xylose yield % of alkali pre-treated WHB

ANOVA for Response Surface Quadratic model						
Analysis of variance table [Partial sum of squares - Type III]						
	Sum of		Mean	F	p-value	
Source	Squares	df	Square	Value	Prob > F	
Model	7326.09	20	366.30	458.86	< 0.0001	significant
A-Soaking time	32.47	1	32.47	40.67	< 0.0001	
B-Concentration	26.51	1	26.51	33.20	< 0.0001	
C-Agitation Speed	129.23	1	129.23	161.88	< 0.0001	
D-Treatment Time	72.01	1	72.01	90.20	< 0.0001	
E-Treatment Temperature	1180.70	1	1180.70	1479.02	< 0.0001	
AB	5.61	1	5.61	7.03	0.0129	
AC	58.41	1	58.41	73.17	< 0.0001	
AD	11.80	1	11.80	14.78	0.0006	
AE	12.60	1	12.60	15.78	0.0004	
BC	3.69	1	3.69	4.62	0.0400	
BD	13.93	1	13.93	17.45	0.0002	
BE	157.24	1	157.24	196.97	< 0.0001	
CD	7.72	1	7.72	9.67	0.0042	
CE	0.46	1	0.46	0.58	0.4538	
DE	20.34	1	20.34	25.49	< 0.0001	
A ²	1062.15	1	1062.15	1330.53	< 0.0001	
B ²	1986.48	1	1986.48	2488.40	< 0.0001	
C ²	1874.69	1	1874.69	2348.37	< 0.0001	
D ²	1216.75	1	1216.75	1524.19	< 0.0001	
E ²	2271.89	1	2271.89	2845.92	< 0.0001	
Residual	23.15	29	0.80			
Lack of Fit	11.91	22	0.54	0.34	0.9761	not significant
Pure Error	11.24	7	1.61			
Cor Total	7349.24	49				

The Figure 5 gives clear evidence that model predicted results are very close in agreement with the experimental ones. As most experimental results lie on the 45 degree line implying the model had fitted the experimental data with an excellent accuracy resulting in lesser deviation from predicted values.

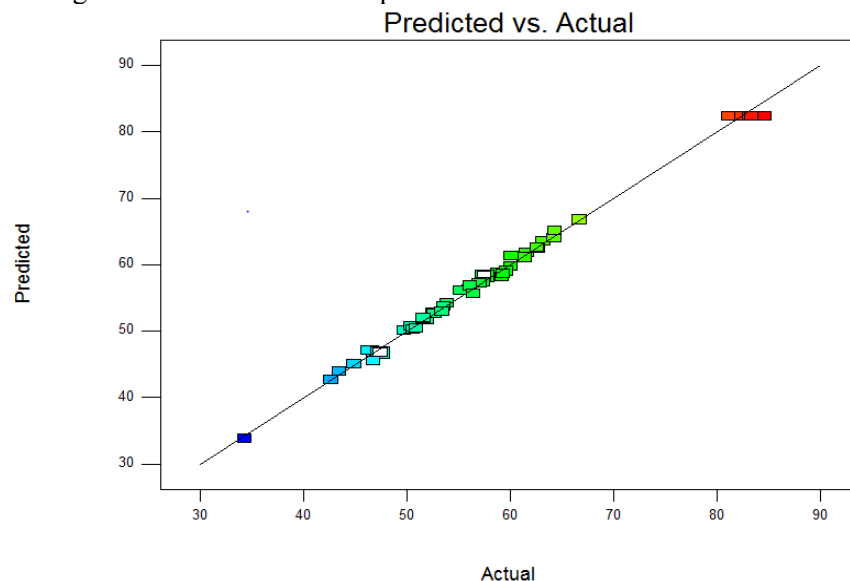


Figure 5: Experimental data plotted against RSM model predicted data for alkaline hydrolysis of WHB.

Contour plots

The contour plot determines the interaction of the components and optimum level of each component for maximum response. These plots were obtained from the pair-wise combination of independent factors, while keeping other factors at its center point level. The elliptical contour plot shown in Figures 6 (a) – (f), it clearly indicates that the mutual interaction is prominent among factors.

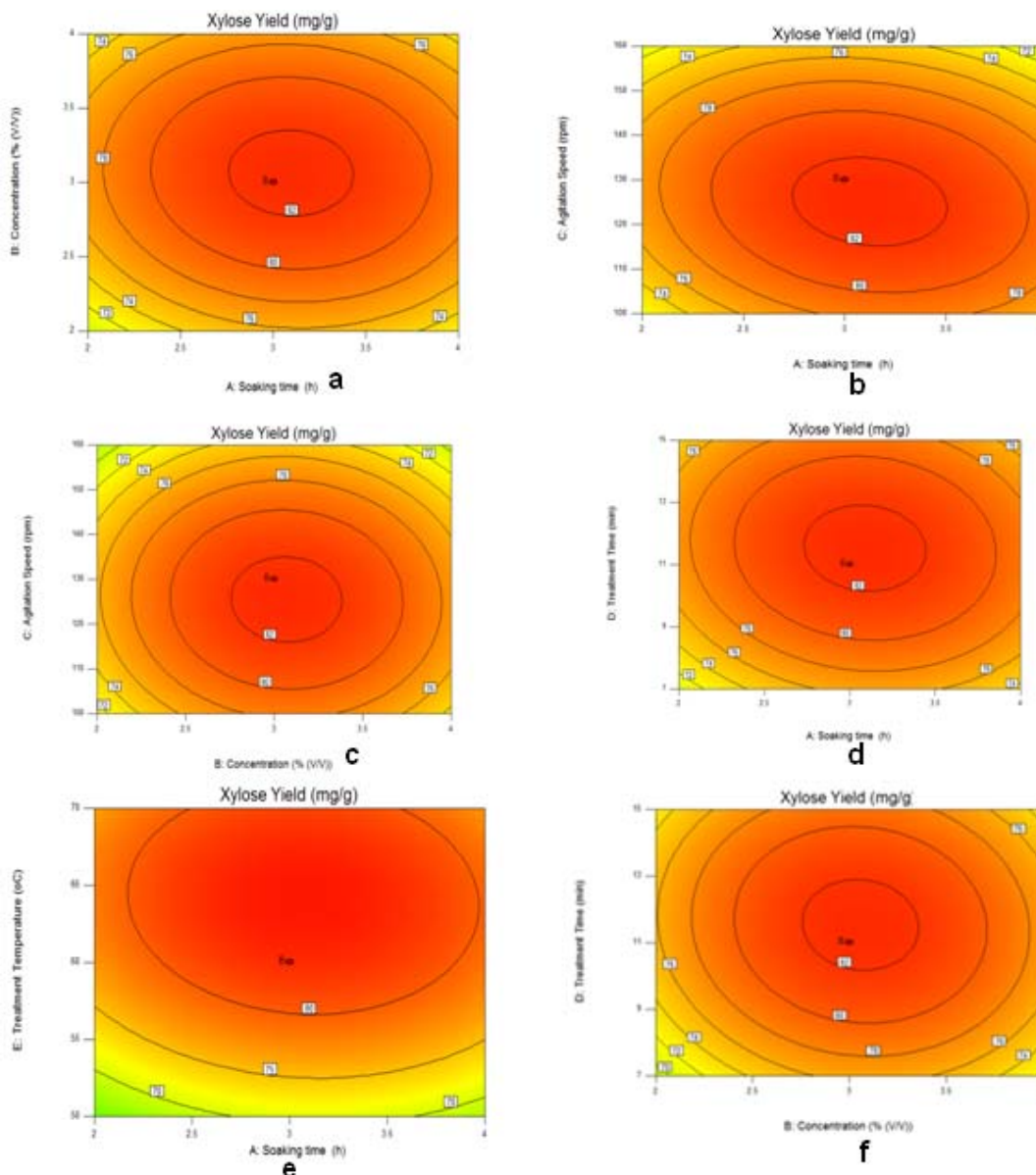


Figure: 6 Elliptical Counter Plots showing mutual interaction between the factors (a) Effect of concentration of NaOH and Soaking Time on xylose yield, (b) Effect of agitation speed and soaking time on xylose yield, (c) Effect of treatment time and soaking time on xylose yield,(d)Effect of treatment temperature and soaking time on xylose yield,(e) Effect of agitation speed and concentration of NaOH on xylose yield,(f) Effect of treatment time and concentration of NaOH on xylose yield.

Response Surface Plots

Design Expert 9.0.3 was also used for plotting response surface plots. The surface plots were beneficial to determine the interaction between process parameters on the total xylose yield % for the alkaline hydrolysis of water hyacinth biomass. The Response surface plots shown in Figure 7 (a) – (f)

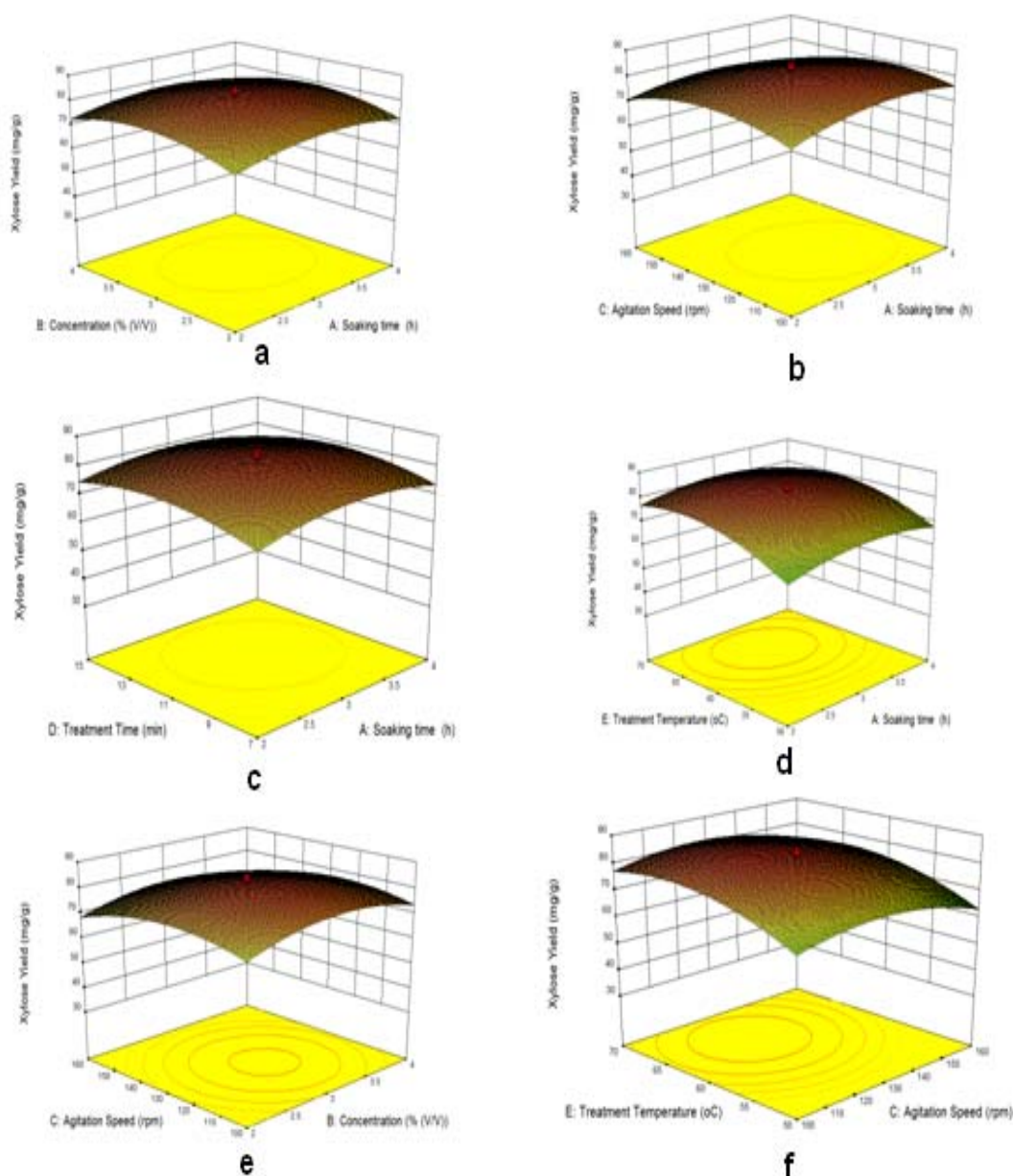


Figure 7: Response Surfaces showing interactions for xylose yield response between (a) Concentration of NaOH and Soaking Time (b) Agitation speed and Soaking time (c) Treatment time and soaking time (d) Treatment temperature and Soaking time (e) Agitation speed and Concentration of NaOH (f) Treatment temperature and Agitation speed.

Sensitivity analysis

ANN modelling is not that much serviceable for studying the effect of individual components and their interaction on the system as compared to RSM. As because the variables in the quadratic equation are already normalised in RSM, the coefficients measured from the equation provide a direct measure of the contribution of each of the factors present in the system. All the factors soaking time, concentration of NaOH, agitation speed, treatment time, treatment temperature have a very important role on the alkali pre-treated WHB. ANN being a black box model, we cannot estimated insight of the system directly. But using various numerical methods, ANN can inherently give the sensitivity analysis of the system. ‘Perturbation method’ Figure 8 is one of the numeric techniques which are employed in our study to estimate the sensitivity analysis of the ANN model. The rate of change response per unit change in each of the independent variable is represented in each of the series of the Figure 8 Higher the slope and the range of change in the response, greater is the expected influence of that very independent variable on the xylose yield. As evident from Figure 8, the treatment temperature has the highest slope and hence can be considered as the most significant factor which influencing the process. This observation is quite similar to that of RSM. Thus ANN can also be used as an effective model for sensitivity analysis.

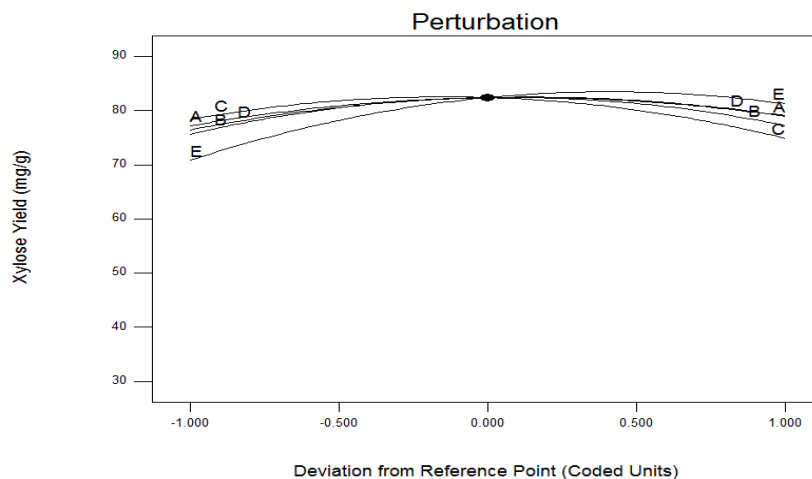


Figure-8: Sensitivity analysis for xylose yield of alkali pre-treated WHB

Optimization

The optimized experimental result of the influencing parameters are NaOH concentration of 3%, agitation speed of 130 rpm, treatment time of 11 min, treatment temperature of 60 oC and soaking time of 3h which produced a xylose yield of 84.5643 mg/g. In comparison to the experimental result, RSM and ANN predicted xylose yield at optimized condition are 82.4595mg/g and 84.675mg/g respectively. The deviation or prediction error of RSM was more in comparison to ANN, RSM over predicted the yield resulting in deviation. The predictive capacity of the model can be determined by the difference between the predicted and experimental yield, lesser the extent of deviation, more is the model accurate. As a result, ANN is better equipped for finding out optimum condition, since it is more accurate and more generalized model than quadratic RSM. Although, there is no statistical difference in maximum experimental yield obtained by ANN and RSM. ANN has predicted the optimum condition and yield more accurately than RSM.

CONCLUSION

In the present work, we used of CCRD design matrix for conducting experiments. Two models response surface methodology (RSM) and artificial neural network (ANN) were developed for finding out the optimum condition and predicting optimum yield. ANN showed better accuracy than RSM even with limited number of experiments. First, RSM was used for predicting the amount of xylose yield from WHB by alkali pre-treatment. Then, the independent variables, soaking time, concentration of NaOH, agitation speed, treatment time, treatment temperature were fed as inputs to an artificial neural network while the output of the network was the amount of xylose yield. A multilayer feed-forward network was trained and used to build an efficient predictive model. Finally, two methodologies were compared for their predictive capabilities. ANN showed better results than RSM in predicting the amount of xylose yield in our present study. Thus, ANN has consistently performed better than RSM in all respects. THUS, we can come to the conclusion the even though RSM is widely used for optimization of xylose yield from WHB, ANN significantly proved to be a better alternative and effective for optimization.

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