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Optimization of NaOH thermo-chemical pretreatment for enhancing solubilisation of rice straw by Response Surface Methodology

Seyed Mohammad HOSSEINI^{†*}, Hamidi Abdul AZIZ *, SYAFALNI *, Meisam Valizadeh KIAMAHALLEH**

*Department of Environmental Engineering, School of Civil, University Sains Malaysia, 14300 Nibong Tebal, Pulau Pinang, Malaysia

(E-mail: envhosseini@gmail.com ; cehamidi@eng.usm.my ; cesyafalni@eng.usm.my)

+ Corresponding author. Tel: 0060174750193

** Department of Chemical Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Pulau Pinang, Malaysia

(E-mail: meisam488@gmail.com)

ABSTRACT

A municipal solid waste produced in urban and rural communities is a serious pollution source of water resource in developing country. One of the main problem in treatment of organic solid waste is its non-readily biodegradability due to the complexity of organic material especially hemicellulose. This study reports Sodium Hydroxide (NaOH) thermochemical pretreatment of rice straw as a indicators of cellulosic compound to enhance its biodegradability. Laboratory-scale experiments were carried out in completely mixed reactors, 500 ml capacity. In order to optimization of hydrolysis of rice straw, Response Surface Method (RSM) based on three factors and one response design was performed to optimize experimental conditions. The effects of reaction parameters including sodium hydroxide concentration (0-30 g/l), reaction time (10–180 min) and temperature (30–200 °C) were evaluated. The optimum conditions resulted by RSM were: NaOH concentration 29.9 g/l, reaction time 133.1 min. and temperature 157.6 °C. The actual experimental soluble chemical oxygen demand (SCOD) of rice straw soluble was 41200 mg/l under optimum condition, which compared well to the maximum predicted value of 41211 mg/l.

Keywords

Rice straw; thermochemical pretreatment; Response Surface Method; water resource; pollution

INTRODUCTION

Large amount of solid waste is produced each year in the world. Agricultural, municipal and industrial activities are three main generation sources of the solid waste. Lack of planning in this section can be caused pollution in water, soil and air resources . Organic fraction of municipal solid wastes (OFMSW) is composed of two main parts: readily and non-readily biodegradable. The part of non-readily biodegradable is formed a high content of lignocellulosics fiber (5-25% of the total waste mass) . Unlike readily bio degradable such as starch and sucrose that can be easily bio degradation into the monosaccharide, for the utilization of lignocellulosic materials in a bioconversion process, pretreatment is necessary . Therefore, the first step is integrated of chemical and physical treatment systems for dissolving hemicellulose and improving of accessibility of the cellulose for hydrolytic enzymes (Godliving, 2009). Alkaline thermo-chemical pretreatment process is a well know method that can be used to dissolving complex substrates before biological treatment. Compared with acid or oxidative reagents, alkali treatment appears to be the most effective method in breaking the ester bonds between lignin,

Design of experiment (DOE) is a structured, organized method that is used to determine the relationship between the different factors (X_i) affecting a process and the output of the process (Y). DOE uses the smallest possible number of experimental runs to discover and find the optimum settings for the process. DOE provides a cost-effective means for solving problems and developing new processes. The simplest, but most powerful, DOE tool is two-level factorial design, where each input variable is varied at high (+) and low (-) levels and the output observed for resultant changes. Statistics can then help to determine which inputs have the greatest effect on outputs. Response surface methodology (RSM) has been widely used in the empirical study of the relationship between one or more measured responses such as yield, on one hand, and a number of input variables such as time, temperature, pressure, and concentration on the other hand. RSM is a collection of mathematical and statistical techniques that are useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and the response surface can be explored to determine important characteristics such as optimum operating conditions.

In the present study, response surface methodology (RSM) was also employed to identify the optimum conditions for enhancing of rice straw dissolving by analyzing the relationships among a number of parameters that affect the overall process. Here we report on the chemical property of rice straw and the optimization of NaOH thermochemical pretreatment process parameters for maximum soluble chemical oxygen demand (SCOD) production. Maximum SCOD production means ability to produce maximum methane gas in anaerobic process or minimum residual of un-compostable materials in compost process. Another word, results of this study could enhance the biogas production or composting process from organic fraction of solid waste.

METHOD

Collection of sample

Around 10 kg of rice straw was collected from a local farm of Sungai Dua in Pulau Penang, Malaysia. Firstly, rice straw was cut to nominally 5-10cm length, washed thoroughly with tap water and then air-dried. It was then grinded to 2-3mm size and used for further treatment. Grinding of rice straw reduces the technical digestion time by 23–59% and increase methane production by 5–25%.

Analytical procedures

The selected physicochemical properties of the raw composting materials were measured prior to starting the thermochemical process experiment. The water-soluble extract was prepared by the following procedure: 5g of sample were first mixed with 100ml of deionised water, then shaken for 2h, and leave for 30 min. The supernatant was then filtered through a filter paper (Whatman No. 1). SCOD and TKN (total Kjeldahl nitrogen) were measured by dichromate digestion and Kjeldahl method, respectively (Paola Castaldi, 2008). SCOD of samples were determined by Chemical Oxygen Demand (COD) reactor-Hach, and DR2800 Spectrophotometer. pH values

were measured by using a pH digital meter (EUTECH, pH 510)), and an EC meter (VSI, Model 30M, 100FT, USA) in aqueous extract (weight : volume = 1:10). Moisture content of raw materials was determined by drying the samples at 105°C for 24h. Ash was determined in a muffle furnace at 550 °C for 24h, and the organic matter (OM) was calculated as the difference between ash and dry weight as a percentage . A typical hydrolysis mixture consisted of 5g of rice straw and 20ml of sodium hydroxide. The mixture was heated at 30-200 °C on the magnetic stirrer hotplate.

Response surface methodology

The experimental design for the selected process variables was carried out using Central Composite Design (CCD). In order to obtain the required data, the suitable range of values of each of the three variables was identified as shown in Table 1.

Table 1. Independent variables and their levels used for the Central Composite Design

Factor	variable	Coded levels of variables				
		-1	-0.5	0	0.5	1
Temperature, oC	A	30	72.5	115	157	200
time, min.	B	10	52.5	95	137	180
NaOH Concentration, g/l	C	0	7.5	15	22.5	30

For three variables (n=3) and five levels (low (−) and high (+)), the total number of experiments were 20 determined by the expression 2^n ($2^3=8$ factorial points), $2n$ ($2 \times 3=6$ axial points), 6 (centre points, six replications) as given in Table 2. Specific capacitance (SCOD) was selected as the response for the combination of the independent variables. Experimental runs were randomized to minimize the effects of unexpected variability in the observed responses.

Table 1. Complete experimental conditions tested and corresponding observed and predicted values of SCOD value

Run order	Type	Factors			SCOD value (Response)	
		A	B	C	Observed	Predicted
1	Fact	30.0	10.0	0.0	1654	4412
2	Fact	200.0	10.0	0.0	2167	365
3	Fact	30.0	180.0	0.0	2542	158
4	Fact	200.0	180.0	0.0	3838	5822
5	Fact	115.0	95.0	7.5	9233	9656
6	Fact	115.0	52.5	15.0	7567	4574
7	Fact	72.5	95.0	15.0	4617	2296
8	Fact	115.0	95.0	15.0	7333	8217
9	Axial	115.0	95.0	15.0	7300	8218
10	Axial	115.0	95.0	15.0	6983	8218
11	Axial	115.0	95.0	15.0	6967	8218
12	Axial	115.0	95.0	15.0	7433	8218
13	Axial	115.0	95.0	15.0	7183	8218
14	Axial	157.5	95.0	15.0	7917	8017
15	Centre	115.0	137.5	15.0	7717	8488
16	Centre	115.0	95.0	22.5	19750	17106
17	Centre	30.0	10.0	30.0	3300	1453
18	Centre	200.0	10.0	30.0	15517	18355
19	Centre	30.0	180.0	30.0	8250	10921
20	Centre	200.0	180.0	30.0	41200	38581

Statistical analysis

A quadratic polynomial equation was developed to predict the response as a function of independent variables and their interactions. In general, the response for the quadratic polynomials is described in Eq. (1).

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \sum \beta_{ij} x_i x_j \quad (1)$$

Here, Y is the response (SCOD value), β_0 is the intercept coefficient, β_i is the linear terms, β_{ii} is the squared terms and β_{ij} is the interaction terms, and x_i and x_j are the uncoded independent variables. The model evaluated the effect of each independent variable to a response. The fit quality of the models was judged from their correlation of determination (R^2) (Ahmadi et al., 2005; Jo et al., 2008).

Analysis of variance

Analysis of variance (ANOVA) was applied to estimate the effects of main variables and their potential interaction on the SCOD value. The ANOVA table can also be used to test for the statistical significance of the ratio of mean square due to regression and mean square due to residual error. Fisher F-test was used to check for the adequacy of the model, while p-value and Student's test were used to check for the significance of the equation parameters for a response. Three-dimensional response surfaces and contour plots were used for facilitating a straightforward examination of the influence of experimental variables on the responses. The analysis of the experimental design and calculation of the predicted data were carried out using Design Expert Software (version 6.0.6, Stat ease Inc., Minneapolis, USA) to estimate the response of the independent variables.

RESULTS AND DISCUSSION

Physicochemical property of rice straw

Physicochemical parameters were selected based on effective parameter on methane production process and composting process. The results obtained from analyzing the physical and chemical properties of the rice straw are shown in Table 3. Based on these results rice straw residues are brimful in organic matter (OM) content (88.4%) and total carbon (51.272%), have low total nitrogen (0.645%) and high Carbon/Nitrogen (C/N) ratio (79.49). This property is suitable as a carbon source in biogas production or composting process. The results of other studies, (Iranzo, et al., 2004), are slightly different because the climate and soil characteristics of rice filed in Malaysia are different from them solubility of carbon material in water is determined by SCOD. SCOD of rice straw, was determined after 4hr, 6hr, 16hr and 24hr stirring and the obtained results are shown in table 4.

Table 3. Selected physiochemical properties of rice straw

NO	Parameters	Rice Straw
1	Organic Matter (%)	88.4
2	Total Carbon (% dw)	51.272
3	Total Nitrogen (% dw)	0.645
4	Carbon/Nitrogen	79.49
5	PH	7.15
6	Electrical Conductivity (μSm^{-1})	2381
7	Moisture Content (%)	9.76

*All analysis was reported on a dry weight (dw) basis $\text{TC} = \text{OM}(\%) \times 0.58$

Table 4- SCOD of Water soluble of rice straw sample as function of time

sampling no.	Duration of Stirring (hr)	SCOD of Sample (mg/lit.)
1	2	1889
2	4	2580
3	6	3150
4	16	4185
5	24	4220

Pretest of thermochemical hydrolysis of rice straw by NaOH

In order to compare the solution rate of rice straw organic matter in water with NaOH, 10 runs of COD test were done. The results from Figure 1 shows SCOD extracted at 2hr by alkaline thermochemical(5g/l), equalled to 15240mg/l, while for water was 1889 mg/l which is 8 times lower in the same condition. It indicated that the alkaline pretreatment has a large effect on solubility of carbon compounds of rice straw in water for utilization. Also, the alkaline

pretreatment makes it more accessible for enzymes and bacteria in bio gas production or composting processes . In the study carried out by Penaud, et al (1999), max. SCOD, 63% was obtained at 5g NaOH/l. Heating enhanced the COD solubility to 85% at the higher concentration of sodium hydroxide (26.1g/l) when heated to 140°C for 30 min compared to that of 53.2% which was obtained by Penaud and co-workers at ambient temperature . The result of investigation from anaerobic digestion with waste activated sludge (WAS) by several of pretreatment indicated that when NaOH at 0 to 21g/l was added the SCOD increased from 17.6% to 86.5%

Model fitting and statistical analysis

The results obtained from the experiments conducted are summarized in table 2. The SCOD value ranged from 1654.17 to 41200 mg/l depending on the experimental conditions. Further analysis on the results showed that they developed highest order polynomials equation (in coded units) that could relate SCOD value to the parameters studied. The two factors interaction (2FI) model is given in Eq. (2).

$$Y = 8217.91 + 5720.59 A + 3913.72 B + 7450.00 C - 12246.72 A^2 - 6746.72 B^2 + 20653.26 C^2 + 2689.58 AB + 5419.79 AC + 3509.38 BC \quad (2)$$

The response functions are representing Y as the response for SCOD value, A as the coded value of variable temperature, B as the coded value of stirring time and C as the coded value of alkaline concentration. The closer the value of R^2 to unity, the better the empirical models fits the experimental data. On the other hand, the smaller the value of R^2 , the lesser will be the relevance of the dependent variables in the model has in explaining the behavior of variations .

Parity plot between the observed data versus predicted data using Eq. (2) for the SCOD value and the responses are graphically shown in Figure 2. As can be seen, the predicted values match the observed values reasonably well within the ranges of experimental conditions, with a R^2 value of 0.945. This result suggests the applicability and reliability of the equation in representing the reaction over a range of experimental conditions with sufficient degree of accuracy. Thus, it can be used to simulate the cellulose solublization reaction.

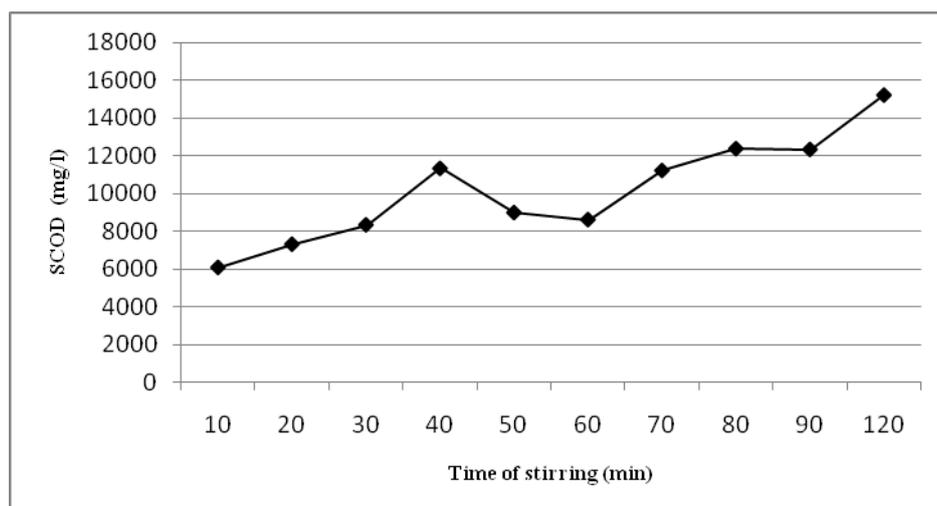


Figure 1: Variation of SCOD of alkaline thermochemical Hydrolysis as function of stirring time (NaOH, 5 g/l, t: 140°C)

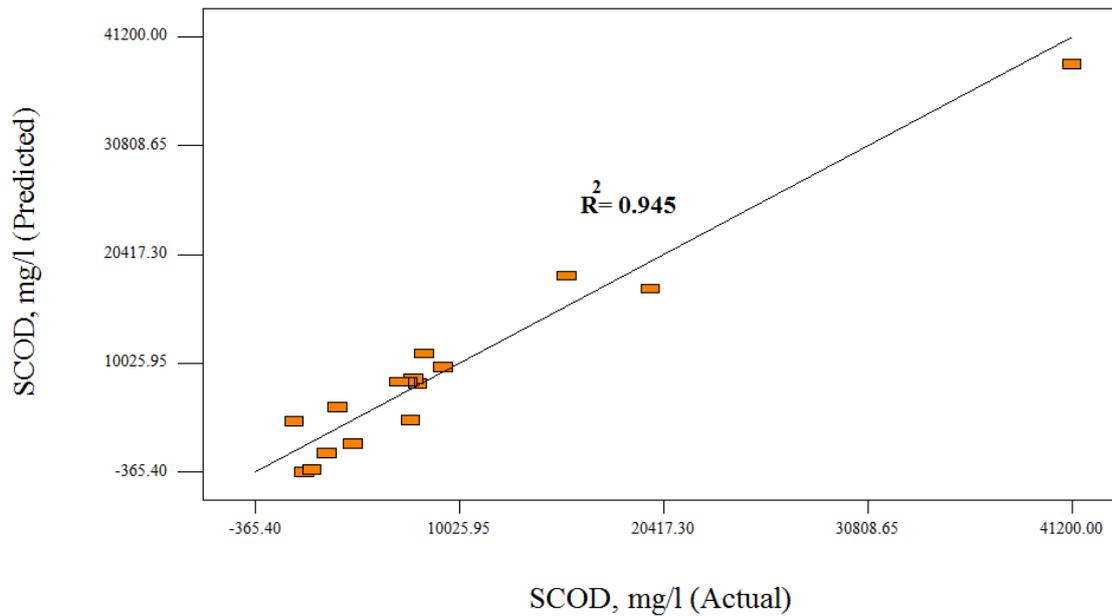


Figure 2: Parity plot for relation between observed and predicted SCOD value

Statistical analysis obtained from the analysis of variance (ANOVA) for the response surface 2FI model is shown in Table 5. The value of “PNF” for the models is less than 0.05 to indicate that it is significant and desirable as it indicates that the terms in the model that have a significant effect on the response. The value of $P < 0.0001$ indicates that there is only a 0.01% chance that a “model F-value” this large could occur due to noise in the experiment. Generally P-values lower than 0.01 indicates that the model is considered to be statistically significant at the 99% confidence level. Values greater than 0.1000 indicate the model terms are not significant. Therefore, A, B, C, AB, AC and BC are significant model terms to affect the SCOD value. The “Lack of Fit F-value” of 429.82 implies the Lack of Fit is not significant relative to the pure error. Therefore, there is a 19.09% chance that a “Lack of Fit F-value” this large could occur due to noise. Insignificant lack of fit is good as the primary objective was the model should fit the experimental data.

Table 5. ANOVA analysis for the quadratic model

Source	Sum of Squares	Degree of freedom (DF)	Mean Square	F Value	Prob > F	
Model	2.782E+008	9	1.513E+008	19.09	< 0.0001	significant
A	1.302E+008	1	2.782E+008	35.11	0.0001	
B	4.718E+008	1	1.302E+008	16.43	0.0023	
C	2.799E+007	1	4.718E+008	59.55	< 0.0001	
A2	8.495E+006	1	2.799E+007	3.53	0.0896	
B2	7.961E+007	1	8.495E+006	1.07	0.3248	
C2	5.787E+007	1	7.961E+007	10.05	0.0100	
AB	2.350E+008	1	5.787E+007	7.30	0.0222	
AC	9.853E+007	1	2.350E+008	29.66	0.0003	
BC	2.782E+008	1	9.853E+007	12.44	0.0055	
Residual	7.922E+007	1	7.922E+006			
Lack of Fit	7.904E+007	10	1.581E+007	429.82	< 0.0001	No significant
Pure Error	1.839E+005	5	36777.29			
Cor Total	1.441E+009	5	R-Squared	0.9450		
Std. Dev.	2814.65	19	Adj. R-Squared	0.8955		
Mean	8923.33		Pred. R-Squared	-0.6740		
Critical Value (C.V)	31.54		Adeq. Precision	19.568		
PRESS	2.412E+009		R-Squared	0.9450		

Effect of process conditions

Influence of individual effect

Most of results of studies indicated that pre-treatment can be implemented at low temperatures but with a relatively long time and high concentration of the alkaline . In this study the SCOD evaluated in vary temperature of 30-200 °C. SCOD value was enhanced by increasing of temperature as shown in Figure 3. The result indicated max. SCOD is obtained in around 157.5 °C. Increasing of temperature has inverse effect on production of SCOD.

DESIGN-EXPERT Plot

SCOD

X = A: Temperature

Actual Factors

B: time = 139.6

C: NaOH Concentration = 30.0

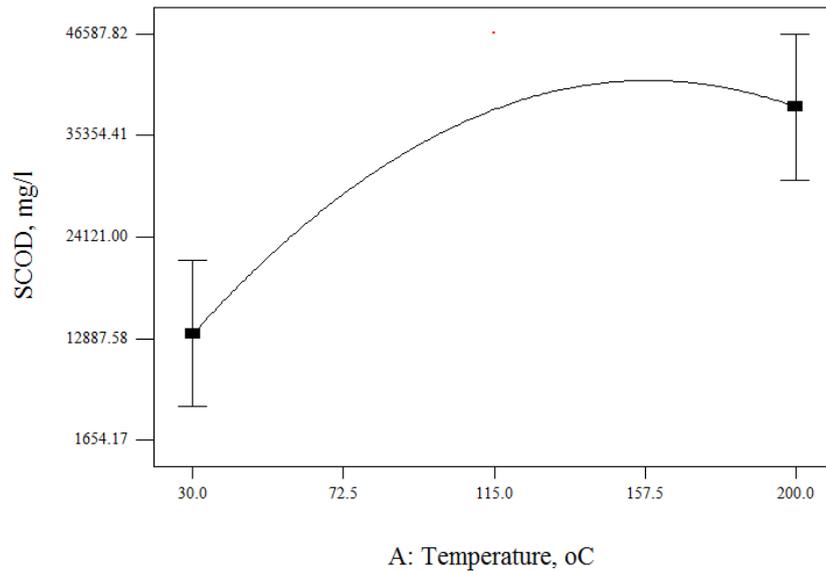


Figure 3: Influence of temperature and stirring time on SCOD

The increase of the SCOD was noticed by increasing the stirring time (Figure 4). Volume of thermochemical reactors is depended to stirring time. So, the selected stirring time should be based on desire rate of SCOD and economic limitation. In a study that was performed in detention time 2.5 to 6.5hr, the solubility of solid waste increased 8.5 to 12.5% . In some study experiments were performed in a fixed temperature and the influence of temperature on solubility have not been evaluated (Suna, et al. 2000; Zhang & Cai, 2008).

As shows in Figure 5, the minimum SCOD is obtained at around 7.5-10 g/l. the SCOD is increased as the concentration of sodium hydroxide solution increased from 15 to 30 g/l. Increasing of SCOD indicates that alkali pretreatment increases the rice straw solubility and its property is improved for bio gas generation process and composting process (Lo'pez Torres & Ma. del C. Espinosa, 2008 ; Fernandes, et al. 2009).

DESIGN-EXPERT Plot

SCOD

X = B: time

Actual Factors
 A: Temperature = 166.5
 C: NaOH Concentration = 30.0

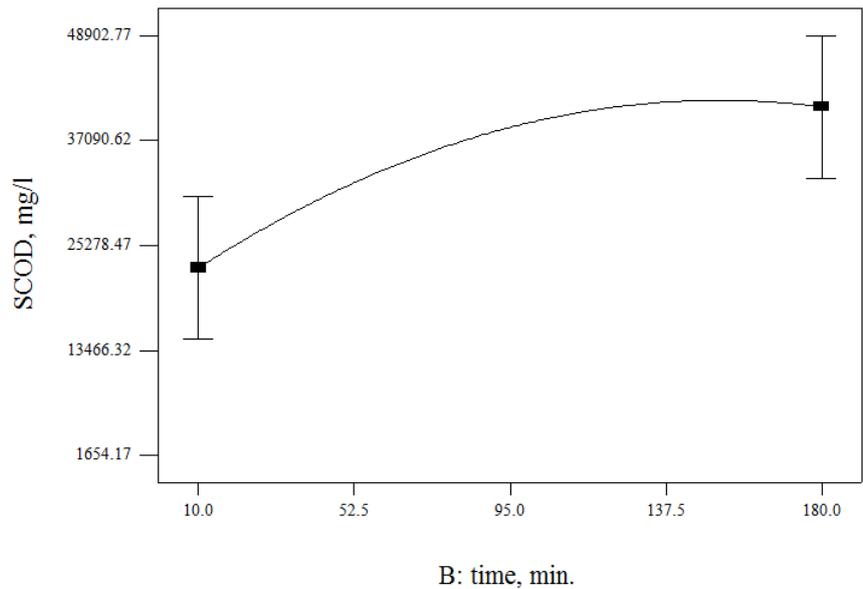


Figure 4: Influence of stirring time on SCOD.

DESIGN-EXPERT Plot

SCOD

X = C: NaOH Concentration

Actual Factors
 A: Temperature = 152.6
 B: time = 155.7

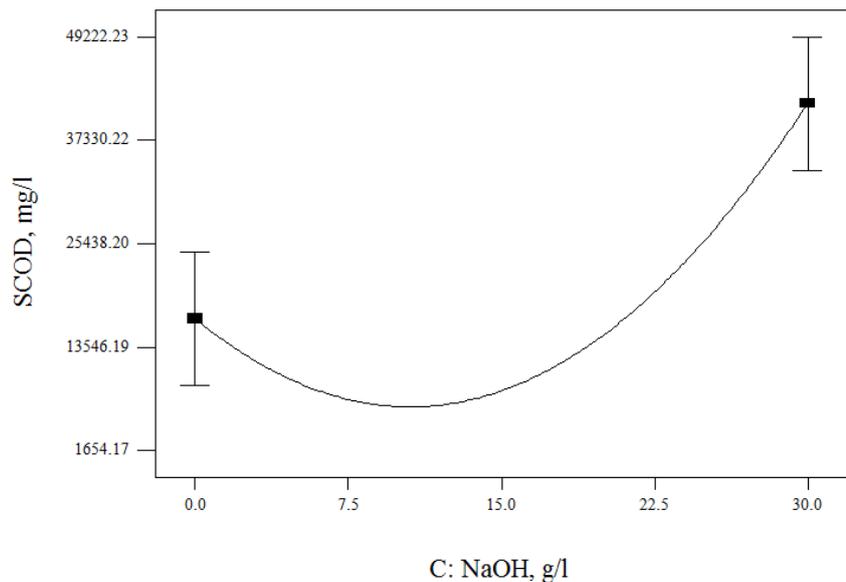


Figure 5: Influence of alkaline concentration on SCOD.

Interactions between the variables

Figure 6 shows the effect of temperature and stirring time on the SCOD production. From Figure 6, it can be seen that factors of temperature and stirring time are interactive while solubility of rice straw. The stirring time has positive effect on the SCOD produced when the temperature is high. For instance, increasing of the SCOD from 11510.49 mg/L to 41684.06 mg/L was noticed by increasing the stirring time from 10 min to 180 min and increasing the temperature from 30 °C to 157.5 °C. The NaOH concentration is fixed at 30 g/L.

DESIGN-EXPERT Plot

SCOD
X = A: Temperature
Y = B: time

Actual Factor
C: NaOH Concentration = 30.0

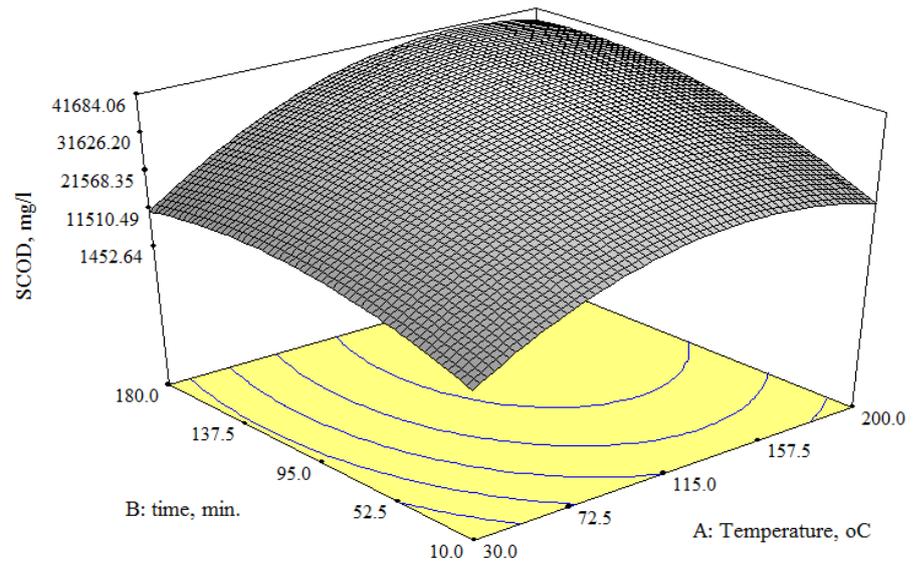


Figure 6: Influence of temperature and stirring time on SCOD.

Figure 7 shows the effect of the concentration of NaOH and temperature on SCOD produced.

DESIGN-EXPERT Plot

SCOD
X = A: Temperature
Y = C: NaOH Concentration

Actual Factor
B: time = 155.7

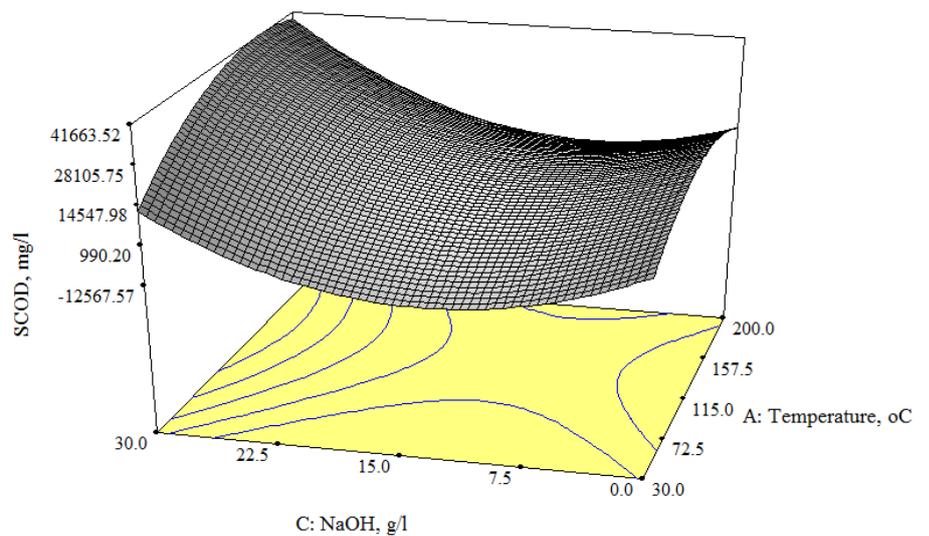


Figure 7: Influence of NaOH concentration and stirring time on SCOD.

From the plot in Figure 7, at higher NaOH concentration, the increase in temperature of reaction until 157.5 °C associates a increasing in solubility of rice straw. For example, at high NaOH concentration of 30 g/l, the SCOD increased from 14547.98 to 41663.52 mg/l when the temperature was increased from 30 to 157.5 °C. From another perspective, by fixing the stirring time, the SCOD also varies with NaOH concentration in two distinct ways, depending upon the extreme of operating conditions employed. Minimum SCOD is obtained in concentration of NaOH of 7.5 g/l with fixed stirring time at 155.7 min. Therefore, it is evident that in order to obtain optimum SCOD by fixing the stirring time, it is indispensable to analyze the relationships of factors A and C. This is because of the indefinite trend observed in investigating such variation in this study as observed in Figure 7.

Figure 8 shows the interaction between the concentration of NaOH and stirring time and their effects on the SCOD produced. Figure 8 indicates the 3D surface plot and the contour plot.

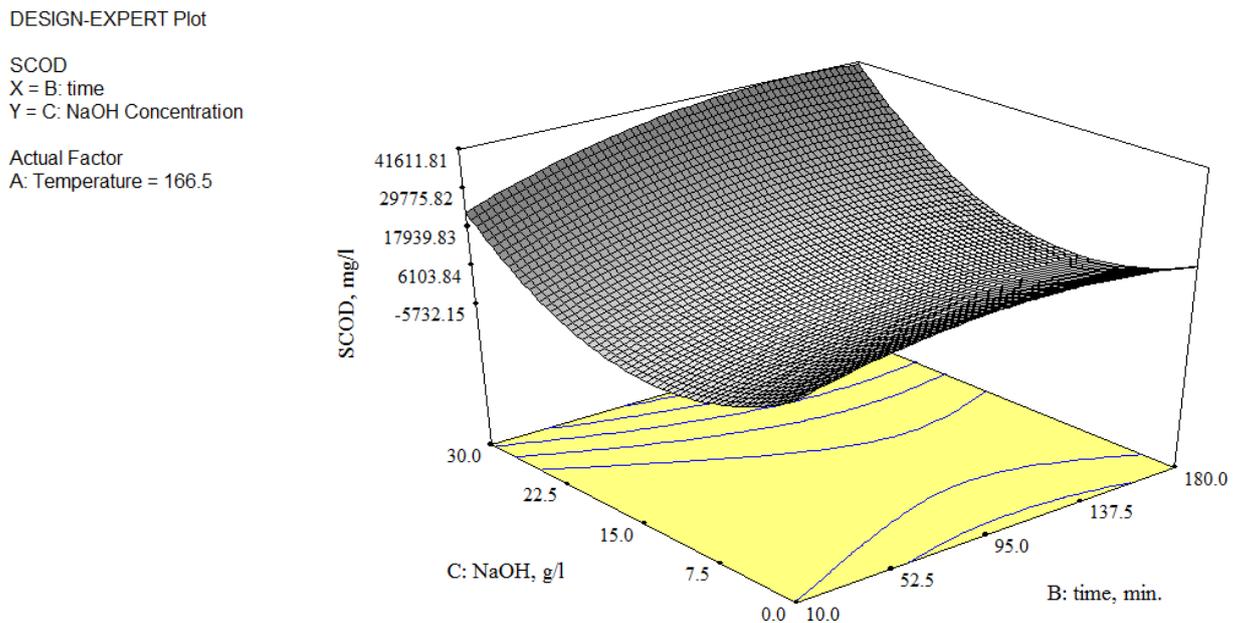


Figure 8: Influence of stirring time and NaOH concentration on SCOD.

In Figure 8, it is evident that the trend observed is the similar to those in Figures 6 and 7. This signifies that the variation of SCOD is highly indefinite and relies solely on the operating conditions such as stirring time and NaOH concentration. By analyzing the Figure, it can be easily seen that at high NaOH concentration at fixed temperature 166.5 °C, the SCOD rises proportionally with stirring time. For example, at 30 g/l of NaOH, when the sample was subject to an alkaline thermochemical process stirring time increment of 180 min from 10 min., it consequently led to a proportional increase of SCOD to 41611.81 mg/l by approximately 17939.83 mg/l.

Besides that, by analyzing the table 2 in another perspective, the increase of the SCOD from 3837.5 mg/l to 41200 mg/L was obtained when NaOH concentration was increased from 0 g/l to 30 g/l, more than 90% decrease. In the condition temperature and stirring time were 200 °C and 180 min. Hence, it has been elucidated that SCOD is influenced indefinitely by the concentrations of NaOH and stirring time, as well as the temperature of reactor.

PROCESS OPTIMIZATION

In the alkaline hydrolysis of rice straw process, its yield can be increased by manipulating the parameters such as the concentrations of alkaline, temperature and detention time. Nevertheless, optimization the response is difficult as the variation of each parameter with hydrolysis of rice straw is highly indefinite as discussed in the previous section. Therefore, in order to optimize the response, the function of desirability was applied using Design Expert software version 6.0.6. In this study, numerical optimization was chosen. Numerical optimization presents a comprehensive and up-to-date description of the most effective methods in continuous optimization. It responds to the growing interest of optimization in engineering, science, and business by focusing on the methods that are best suited to practical problems . To do so, the upper and lower limit of each variable (NaOH concentration, temperature and stirring time) and its response as predicted by the model were provided based on the contour and surface plot obtained previously. The ultimate goal of this optimization was to obtain the maximum response

that simultaneously satisfies all the variables properties. Table 6 below shows constraints of each variable and the desired response.

Table 6. Constraints of each variable for the numerical optimization of the SCOD

Type of variable	Goal	Lower Limit	Upper Limit
NaOH Concentration, g/l	is in range	0	30
Process temperature, °C	is in range	30	200
Stirring time, min	is in range	10	180
Specific capacitance	maximize	1654.17	41200

Table 7 exhibits the 6 possible solutions which fulfilled all specified conditions. All solution gave desirability value of 1 representing the ideal solution. Therefore, the optimum conditions in any of the given solutions can be chosen for further validation. In this study, solution number 1 (157.6 °C, 133.1 min and 29.9 g/l NaOH) was selected due to its highest prediction of response (41211 mg/l).

For validation purpose, comparisons were made between the predicted optimum condition and its subsequent response with the results obtained from experimental work. As observed the maximum SCOD production predicted for solution 1 was 41211 mg/l (157.6 °C, 133.1 min and 29.9 g/l NaOH). The maximum value of same response obtained from run 20 of experimental work with almost similar condition was 41200 mg/l (200 °C, 180 min and 30 g/l NaOH). The percentage difference between both experimental and predicted values was 0.00027%. The low percentage proved that the model was significant in predicting the response.

Table 7. Optimum conditions for maximum SCOD production

No.	Temperature , oC	Time, min	NaOH, g/l	SCOD, mg/l	Desirability
1	157.6	133.1	29.9	41211	1.000
2	142.9	145.7	30.0	41204	1.000
3	166.6	164.2	30.0	41387	1.000
4	163.4	136.1	30.0	41422	1.000
5	173.8	142.8	30.0	41243	1.000
6	122.8	115.8	30.0	38712	0.937
7	116.4	97.8	0.0	21430	0.500
8	116.8	98.6	0.0	21429	0.500
9	122.8	108.9	0.0	21273	0.496

SCOD extracted in optimum condition of this study is more than other study. For example The optimal solubilization condition for OFMSW was 2.3 g Ca (OH)₂/L) and 6.0 h . Under these conditions, dissolving reached levels up to 11.5%. Moreover, in optimal concentration, SCOD decreased from 19,359 to 14,664 mg/L, a finding that also supports the above results . Another study that develops an alkali pretreatment process prior to anaerobic digestion of pulp and paper sludge to enhanced the methane productivity. Maximum SCOD , 83% was achieved in 8 g NaOH/100 g TS sludge

CONCLUSION

A well dissolving of cellulosic compounds in rice straw is achieved by alkaline thermochemical hydrolysis. Alkaline thermochemical hydrolysis process leads to the increasing of transformation of non-solution complex cellulosic compounds to soluble simple compounds. The mathematical model developed could predict the SCOD yield at any point in the experimental domain as well as the determination of the optimal solubilization conditions with sufficient degree of accuracy.

The high accuracy of the model indicates that a two factors interaction (2FI) model could be used to optimize the SCOD value. The effect of alkaline and the thermochemical hydrolysis conditions to achieve an optimal response with 41211 mg/l of SCOD value were found to be 157.6 °C for the reaction temperature, 133.1 min for the stirring time and 29.9 g/l for the NaOH concentration. The interactions between reaction temperature, stirring time and NaOH concentration had been accurately demonstrated and elucidated. These results proved that alkaline thermochemical pretreatment was highly potential for increasing the SCOD value of rice straw and the use of RSM based on CCD was practical for the simultaneous study of effects by process variables on the SCOD value and the possible interaction between them. Also from the results, a process for the production of biogas or compost from organic solid waste shall have two main stages. The two-stage design must involve alkaline pretreatment, followed by anaerobic digestion or composting process unit. In this situation the rejected and risk of water and soil resource waste will be decreased to minimum.

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