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Evaluation of Significant Parameters on Alkaline Pretreatment Process of Rice Straw

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Abstract

Rice straw can be used in the production of energy or organic fertilizer. However, one of the main problems in the transformation of rice straw is its low-biodegradability. This study reports a comparison on the effectiveness of the Sodium Hydroxide (NaOH) and Potassium Hydroxide (KOH) pre-treatment methods on the enhancement of rice straw solubilization to increase its biodegradability. Laboratory-scale experiments were carried out in reflux completely mixed reactors of 500 mL capacity. The Response Surface Method (RSM) was used to optimize experimental conditions. The respective optimum conditions determined from RSM for NaOH and KOH were as follows: concentration of 29.99 and 30 g/L, reaction time of 165.7 and 151.23 min, and temperature of 164.37 and 200°C. The actual and predicted results are clearly indicated that the solubility of rice straw is significantly increased under NaOH pretreatment in comparison with KOH. The actual results of solubility of rice straw under optimum conditions are compared well with the maximum predicted values.

Keywords: *rice straw, solubilization, agricultural wastes*

1. Introduction

A reliable source of biomass is necessary for the production of biogas. Rice straw is the largest available biomass feedstock in the world (i.e., 7.31×10^{14} of dry rice straw per year), and 90% of the annual global rice straw is produced in Asia (Kim and Dale, 2004). Compounds such as cellulose are hardly decomposed by biological processes because of their recalcitrant and heterogeneous structure, primarily consisting of cellulose, hemicelluloses, and lignin (Chandra *et al*., 2007). Cellulose digestibility is enhanced by the alkali pre-treatment process. This process is more effective for lignin solubilization than the acid or hydrothermal processes (Alvira *et al*., 2010).

The first step is the integration of the chemical and physical treatment systems for dissolving hemicelluloses, followed by the improvement in the accessibility of cellulose for hydrolytic enzymes (Godliving, 2009). Alkaline pre-treatment involves the application of alkaline solutions like NaOH or KOH to remove lignin and a part of the hemicelluloses, and an increase in the accessibility of the enzyme to cellulose (Tarkov and Feist, 1969). Alkali pre-treatment can result in a sharp increase in saccharification yields. The main effect of sodium hydroxide pre-treatment on lignocellulosic biomass is delignification by breaking the ester bonds cross-linking lignin and xylan, thus increasing the porosity of biomass (Tarkov and Feist, 1969).

Design of Experiment (DOE) is a structured and organized method to determine the relationship between the different factors (Xi) 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 of the process (Yang and El-Haik, 2003). It provides a cost-effective means of solving problems and developing new processes. The simplest but most powerful DOE tool is the two-level factorial design where each input variable is varied at high (+) and low (–) levels while observing the output for any resulting changes. Statistics then helps determine which input has the greatest effect on the output (Anderson and Kraber, 1999; Aziz *et al*., 2009). 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 (Aziz *et al*., 2009; Chow and Yap, 2008; Ko *et al*., 2009; Kumar *et al*., 1999).

In the present study, response surface methodology was employed to identify the optimum conditions for enhancing rice straw solubility by analyzing the relationships among a number of parameters that affect the overall process. RSM was also used

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to compare the efficiency of NaOH and KOH in enhancing the maximum solubility of rice straw. In the present work, Soluble Chemical Oxygen Demand (SCOD) is considered a suitable indicator for evaluating the solubility of the cellulosic compound of rice straw (Torres and Lloréns, 2008).

2. Method

2.1 Collection of the Sample

Rice straw samples were taken from a local farm of Sungai Dua located in Pulau Penang, Malaysia. First, rice straw was cut nominally to 5-10 cm in length, washed thoroughly with tap water, and then air-dried. It was then grinded to 2-3 mm size and was used for further treatment.

2.2 Analytical Procedures

The selected physicochemical properties were measured prior to the start of the thermochemical process experiment. The water-soluble extract was prepared using the following procedure: 5 g of the sample was initially mixed with 100 mL of deionised water, shaken for 2 h, and left for 30 min. Next, the supernatant was filtered through a filter paper (Whatman No. 1) (Goyal *et al.*, 2005). SCOD and total Kjeldahl nitrogen (TKN) were measured using dichromate digestion and the Kjeldahl method, respectively (Paola Castaldi, 2008). The SCOD of samples was determined using COD reactor-Hach and DR2800 Spectrophotometer. The pH values were measured using a pH digital meter (EUTECH, pH 510) and an EC meter (VSI, Model 30M, 100FT, USA) in aqueous extract (weight: volume = 1:10) (Sylla and Kuroda, 2006). The moisture content of the raw materials was determined by drying the samples at 105°C for 24 h. Ash was determined in a muffle furnace at 550°C for 24 h, and Organic Matter (OM) was calculated as the difference between ash and its dry weight as a percentage (Yamada and Kawase, 2006). A typical hydrolysis mixture consisted of 5 g of rice straw and 20 mL of sodium hydroxide. The mixture was heated at 30-200°C on a magnetic stirrer hotplate.

2.3 RSM

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

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

Table 2. Selected Physiochemical Properties of Rice Straw

NO	Parameters	Rice Straw
1	OM(%)	88.4
2	TC (% dw)	51.272
3	TN (% dw)	0.645
4	C/N	79.49
5	PH	7.15
6	EC (μSm^{-1})	2381
7	Moisture Content (%)	9.76
8	SCOD (mg/L)	1889

*All analysis was reported on a dry weight (dw) basisTC = OM(%) × 0.58 (Rashad *et al.*, 2010)

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

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum\sum \beta_{ij} x_{ij} x_j \tag{1}$$

Y is the response (SCOD value), β_o is the intercept coefficient, β_i is the linear term, β_{ii} is the squared term, β_{ij} is the interaction term, and x_j and x_j are the uncoded independent variables. The model evaluated the effect of each independent variable to the response. The fit quality of the models was evaluated from their correlation of determination (R^2) (Ahmadi *et al.*, 2005; Ghafari *et al.*, 2009; Jo *et al.*, 2008).

2.4 ANOVA

ANOVA was applied to estimate the effects of the 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 the mean square due to regression and the mean square due to residual error. Fisher F-test was used to check the adequacy of the model, whereas the p-value and Student's test were used to check for the significance of the equation parameters for the response. Three-dimensional response surfaces and contour plots were used for facilitating a straightforward examination of the effect of the experimental variables on the responses (Jo *et al.*, 2008). The analysis of the

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

experimental design and the 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.

3. Results and Discussion

3.1 Physicochemical Property of Rice Straw

Physicochemical parameters were selected based on the effective parameters of biodegradation process. The solubility of carbon material in water is determined by SCOD. The SCOD of rice straw was determined after 2 h of stirring, 1:20 (w:v). The results obtained are shown in Table 2. Rice straw residues are found to be rich in OM (88.4%) and total carbon (51.272%), and they have a low total nitrogen (0.645%) and high C/N ratio (79.49). These properties make rice straw a suitable carbon source for biogas production or the composting process. The results of other studies (Rashad *et al*., 2010), (Iranzo *et al*., 2004), (Abdelhamid *et al.*, 2004) are slightly different because the climate and soil characteristics of the rice field in Malaysia are different.

3.2 Model fitting and Statistical Analysis

The results obtained from the experiments are summarized in Table 3. The $SCOD_{NaOH}$ value ranged from 1654.17 to 41200 mg/L, and the $SCOD_{KOH}$ value ranged from 1675 to 27000 mg/L depending on the experimental conditions. Further analysis of the results showed that they developed the highest order polynomial equation (in coded units) that can relate the SCOD value to the parameters studied. The Two-Factor Interaction (2FI) models are given in Eq. (2), (3):

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

$$Y(SCOD_{KOH}) = 11573.73+3642.16A+3302.94B+4400.00C -1171.23A_2-5671.23B_2+2428.77C_2+1631.25AB +2887.50AC^{+}+2553.13BC \quad (3)$$

The statistical analysis obtained from ANOVA for the response surface 2FI model is shown in Table 4. The value of “PNF” for the models is less than 0.05, indicating that it is significant and desirable because it indicates that the terms in the model have a significant effect on the response. The values $P < 0.0001$ for $SCOD_{NaOH}$ and $P = 0.0014$ for $SCOD_{KOH}$ indicate that there are only 0.01% and 0.14% chances, respectively, that a “model F-value” this large can occur due to noise in the experiment. Generally, P-values lower than 0.01 indicate that these models are statistically significant at 99% confidence level (Ravikumar *et al*., 2005). The values greater than 0.1000 indicate that the model terms are not significant. Therefore, A, B, C, A^2, B^2, C^2, AB, AC, and BC are significant model terms that affect the $SCOD_{NaOH}$ and $SCOD_{KOH}$ values.

3.3 Process Analysis

The response surface plots for NaOH and KOH are shown in Figs. 3 and 4, respectively. The two-dimensional representation of the responses on the time-temperature, time-alkaline concentration,

Table 3. Complete Experimental Conditions Tested and Corresponding Observed and Predicted Values of SCOD Value

Run order	Type	Factors			$SCOD_{NaOH}$ value ($Response_1$)		$SCOD_{KOH}$ value ($Response_2$)	
		A	B	C	Observed	Predicted	Observed	Predicted
1	Fact	30	10	0	1654.17	4412.18	1675.00	2886.82
2	Fact	200	10	0	2166.67	365.40	2100.00	1133.64
3	Fact	30	180	0	2541.67	158.29	2520.83	1123.95
4	Fact	200	180	0	3837.50	5822.47	3845.83	5895.77
5	Fact	30	10	30	3300.00	1453.84	3066.67	805.58
6	Fact	200	10	30	15516.67	18355.44	9416.67	10602.39
7	Fact	30	180	30	8250.00	10920.87	8500.00	9255.21
8	Fact	200	180	30	41200.00	38580.80	27000.00	25577.02
9	Axial	72.5	95	15	4616.67	2295.93	5233.33	9459.85
10	Axial	157.5	95	15	7916.67	8016.52	13950.00	13102.01
11	Axial	115	52.5	15	7566.67	4574.36	6000.00	8504.46
12	Axial	115	137.5	15	7716.67	8488.09	10933.33	11807.40
13	Axial	115	95	7.5	9233.33	9656.22	10933.33	9980.92
14	Axial	115	95	22.5	19750.00	17106.22	10050.00	14380.93
15	Centre	115	95	15	7333.33	8217.91	14150.00	11573.73
16	Centre	115	95	15	7183.33	8217.91	12200.00	11573.73
17	Centre	115	95	15	6966.67	8217.91	13016.67	11573.73
18	Centre	115	95	15	7433.33	8217.91	12266.67	11573.73
19	Centre	115	95	15	7300.00	8217.91	13816.67	11573.73
20	Centre	115	95	15	6983.33	8217.91	13283.33	11573.73

Table 4. ANOVA for the Quadratic Model

Response	Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	Note
$SCOD_{NaOH}$	Model	1.36E+09	9	1.513E+008	19.09	< 0.0001	Significant
	A	2.78E+08	1	2.782E+008	35.11		Significant
	B	1.3E+08	1	1.302E+008	16.43		Significant
	C	4.72E+08	1	4.718E+008	59.55		Significant
	A^2	27989978	1	2.799E+007	3.53		Significant
	B^2	8494701	1	8.495E+006	1.07		Significant
	C^2	79605062	1	7.961E+007	10.05		Significant
	AB	57870832	1	5.787E+007	7.30		Significant
	AC	2.35E+08	1	2.350E+008	29.66		Significant
	BC	98525703	1	9.853E+007	12.44		Significant
	Residual	79222388	1	7.922E+006			
	Lack of Fit	79038501	10	1.581E+007	429.82		
	Pure Error	183886.4	5	36777.29			
	Cor Total	1.44E+09	5	R-Squared	0.9450		
	Std. Dev.	2814.647	19	Adj. R-Squared	0.8955		
	Mean	8923.334		Pred. R-Squared	-0.6740		
	C.V.	31.54255		Adeq. Precision	19.568		
	PRESS	2.41E+09					
$SCOD_{KOH}$	Model	6.01E+08	9	66731374	8.289875	0.0014	Significant
	A	1.13E+08	1	1.13E+08	14.00729		Significant
	B	92729985	1	92729985	11.51962		Significant
	C	1.65E+08	1	1.65E+08	20.4429		Significant
	A^2	256003.3	1	256003.3	0.031803		Significant
	B^2	6002295	1	6002295	0.74565		Significant
	C^2	1100867	1	1100867	0.136758		Significant
	AB	21287813	1	21287813	2.644533		Significant
	AC	66701250	1	66701250	8.286132		Significant
	BC	52147578	1	52147578	6.478166		Significant
	Residual	80497446	10	8049745			
	Lack of Fit	77339300	5	15467860	24.48884		
	Pure Error	3158145	5	631629.1			
	Cor Total	6.81E+08	19	R-Squared	0.8818		
	Std. Dev.	2837.207		Adj. R-Squared	0.7754		
	Mean	9697.917		Pred. R-Squared	-0.4364		
	C.V.	29.25584		Adeq. Precision	12.347		
	PRESS	9.78E+08					

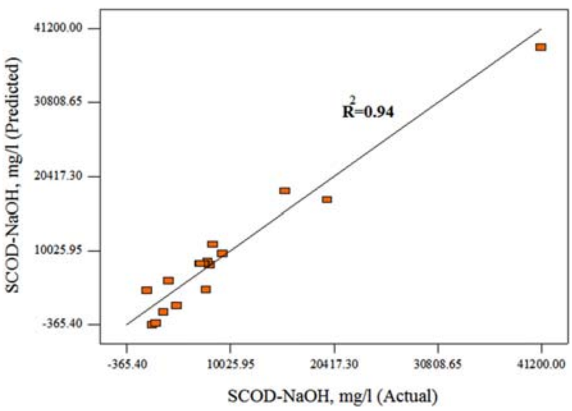

Fig. 1. Parity Plot for Relation between Observed and Predicted $SCOD_{NaOH}$ Value

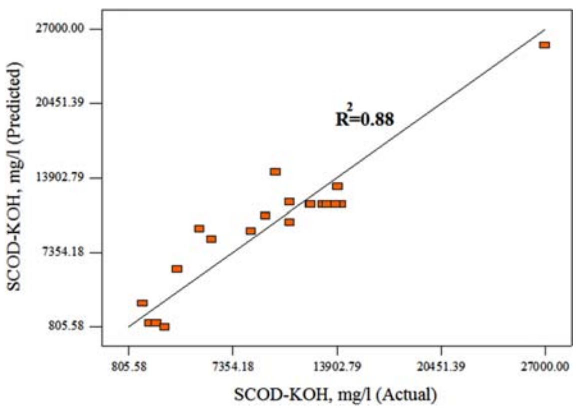

Fig. 2. Parity Plot for Relation between Observed and Predicted $SCOD_{KOH}$ Value

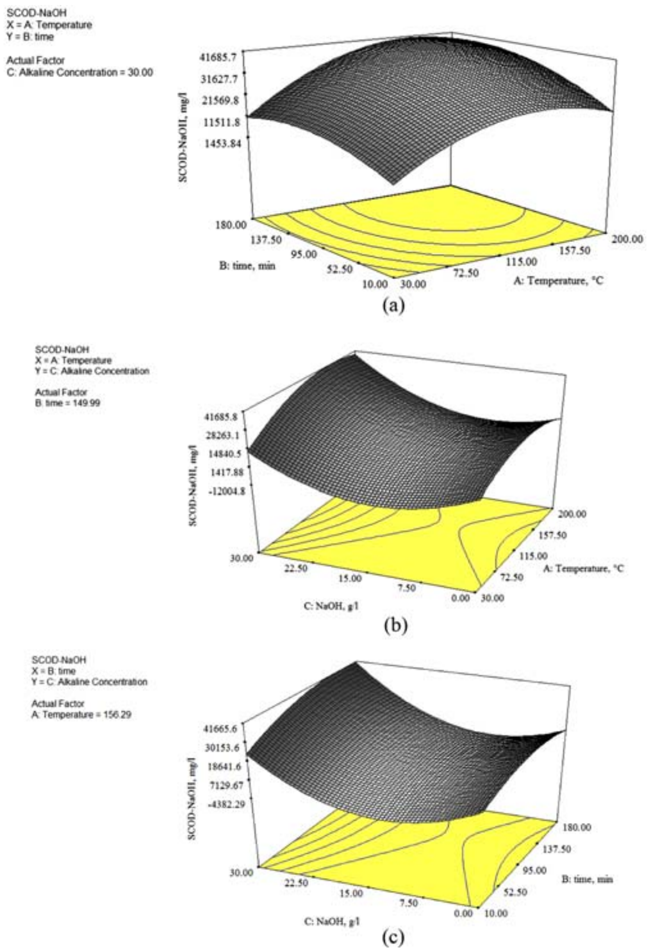

Fig. 3. Expert Plot; Response Surface Plot for Influencing of Stirring Time and Temperature: (a) NaOH Concentration and Temperature, (b) and NaOH Concentration and Stirring Time, (c) on SCOD

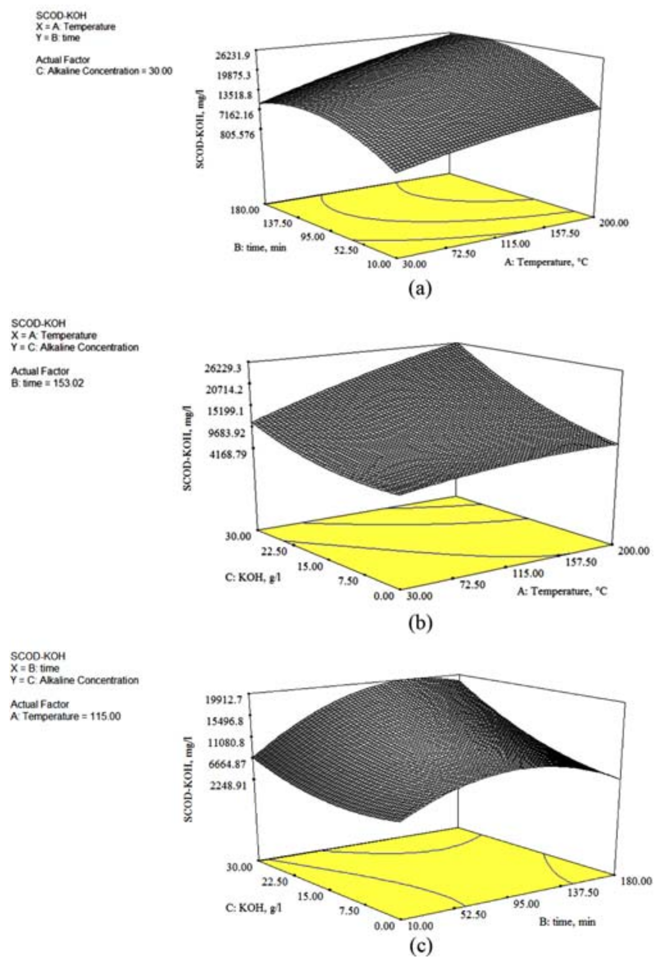

Fig. 4. Expert Plot; Response Surface Plot for Influencing of Stirring Time and Temperature: (a) KOH Concentration and Temperature, (b) and KOH Concentration and Stirring Time, (c) on SCOD

and temperature-alkaline concentration planes (contour plot) shows concentrically closed curves whose centers represent the optimum conditions.

The response surface plots in Fig. 3 indicate that the optimum points occur at a temperature of 154°C, time of 145 min, and dosage of 30 g/L for NaOH. Likewise, Fig. 4 demonstrates that the optimum solubilization of rice straw occurs at around a temperature of 200 °C, time of 151 min, and dosage of 30 g/L for KOH.

An increase in soluble COD of rice straw was noticed with an increase in stirring time (Figs. 3(a) and 4(a)) for NaOH and KOH hydrolysis. These Figs. show that temperature and stirring time interact in affecting the solubility of rice straw. Stirring time has a positive effect on the SCOD produced when the temperature is high. For instance, in Fig. 3(a), an increase in SCOD from 11500 to 41700 mg/L was noticed with an increase in stirring time from 10 to 180 min and with an increase in temperature from 30 to 157.5°C. Likewise, in Fig. 4(a), an increase in SCOD from 8000 to 26232 mg/L was noticed with an increase in stirring time from 10 to 180 min and with an increase in temperature from 30 to 200°C. Given that the NaOH concentration is fixed at 30 g/L in the same concentration, the efficiency of NaOH is 1.37 times higher than that of KOH. In a study conducted at detention times 2.5-6.5 h, the solubility of solid waste increased from 8.5% to 12.5% (López Torres and Ma. del C. Espinosa, 2008). In experiments involving fixed temperatures, the effect of temperature on solubility has not been evaluated (Sun *et al*., 2000; Zhang *et al.*, 2008).

As shown in Figs. 3(b) and 4(b), the minimum SCOD is obtained at around 7.5-10 g/L. SCOD increases as the concentration of sodium hydroxide solution increases from 15 to 30 g/L. An increase in SCOD indicates that the alkali pre-treatment increases rice straw solubility, and its property is improved for the bio gas generation and composting processes (Torres and Espinosa, 2008; Fernandes *et al.*, 2009).

From the plot in Figs. 3(b) and 4(b), at a higher NaOH concentration, the increase in reaction temperature up to 157.5 and 200°C for NaOH and KOH, respectively, corresponds to an increase in the solubility of rice straw. For example, at a 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. The minimum SCOD was obtained at a NaOH concentration of 7.5 g/L, with the stirring time fixed at 155.7 min, whereas at a KOH concentration of 30 g/L, the SCOD increased from 10000 to 26200 mg/L when the temperature was increased from 30 to 200°C. From another perspective, by making the stirring time constant, the SCOD also varies with the NaOH and KOH concentrations in two distinct ways depending on the extremity of the operating conditions used. Therefore, in order to obtain the optimum SCOD by making the stirring time constant, there is a need to analyze the relationships of factors A and C. This is due to the indefinite trend observed in examining such variation in the current study, as observed in Figs. 3(b) and 4(b).

Figures 3(c) and 4(c) show the 3D surface plot and the contour plot of the interaction between the alkaline concentration and stirring time, as well as their effects on the SCOD produced. In these Figs., the variation in SCOD is highly indefinite and relies solely on the operating conditions such as the stirring time and NaOH and KOH concentrations. Analysis of Fig. 3(c). shows that at a high NaOH concentration at a fixed temperature of 156.29°C, the SCOD increases proportionally with the stirring time. For example, at 30 g/L NaOH, when the stirring time was increased from 10 to 180 min, the SCOD increased proportionally to 41670 mg/L (increased by approximately 25000 mg/L). At 30 g/L KOH, an increase in the stirring time from 10 to 137.5 min increased the SCOD from 6670 to 19900 mg/L. SCOD is decreased with an increase in stirring time from 137.5 to 180 min. Hence, SCOD is affected indefinitely by the concentrations of NaOH, the stirring time, and the temperature of the reactants. Additionally, NaOH is more effective than KOH in dissolving rice straw.

4. Process Optimization

In the alkaline hydrolysis of rice straw, the yield can be increased by manipulating parameters such as the concentration of alkaline, the temperature, and the detention time. However, optimization of the response is difficult because the variation in each parameter with the 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 the present study, numerical optimization was chosen. This process 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 the fields of engineering, science, and business by focusing on the methods that are best suited to practical problems (Nocedal and Wright, 2006). To do so, the upper and lower limits of each variable (NaOH and KOH 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 optimization is to obtain the maximum response that simultaneously satisfies all variables' properties and the relationships among them. Table 5 shows the constraints of each variable and the desired response. Table 6 shows the six and four possible solutions that satisfy all the specified conditions for NaOH and KOH, respectively. The solutions that gave a desirability value of 1 represent those that can be ideal solutions. Therefore, the optimum conditions in any of the selected solutions can be chosen for further validation. In the current study, the solution number one for NaOH (164.37°C, 165.77 min, and 29.99 g/L NaOH) and the solution number one for KOH (200°C, 151.23 min, and 30 g/L KOH) are selected due to their highest prediction of responses (41425.9 mg/L and 26232 mg/L for NaOH and KOH, respectively).

For validation purpose, comparisons were made between the predicted optimum condition and its subsequent response with

Table 5. 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
KOH 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-NaOH, mg/L	Maximize	1654.17	41200
Specific capacitance-KOH, mg/L	Maximize	1675	27000

Table 6. Optimum Conditions for Maximum SCOD Production by NaOH and KOH

Type of Alkaline	No.	Temperature , °C	Time, min	Alkaline Con. g/L	COD, mg/L	Desirability
NaOH	1	164.37	165.77	29.99	41425.9	1.000
	2	156.96	146.99	29.87	41215.1	1.000
	3	143.55	152.33	30.00	41224.5	1.000
	4	153.07	154.87	29.96	41426.6	1.000
	5	164.93	150.89	29.93	41387.4	1.000
	6	116.30	97.83	0.00	21430.3	0.500
KOH	1	200.00	151.23	30.00	26232.1	0.970
	2	199.90	152.12	29.95	26181.6	0.968
	3	198.55	148.60	30.00	26137.7	0.966
	4	167.48	150.39	30.00	24051.7	0.884

the results obtained from an earlier experimental work. 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 the same response obtained from run 20 of the 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 difference proved that the model was significant in predicting the response. The percentage difference between the experimental and predicted values for KOH is 0.000284%.

The SCOD extracted from the optimum condition of the present work is more than those obtained from other studies. For example, the optimum solubilization condition for the organic fraction of solid waste was 2.3 g Ca(OH)2/L at 6.0 h (López Torres and Ma. del C. Espinosa, 2008). Under these conditions, the solubility reached levels up to 11.5%. Moreover, in the optimum concentration, the SCOD increased from 14664 to 19359 mg/L, a finding that also supports the above results (López Torres and Ma. del C. Espinosa, 2008). Another study developed an alkali pre-treatment process prior to anaerobic digestion of pulp and paper sludge to enhance methane productivity. A maximum SCOD of 83% was achieved in 8 g NaOH/100 g TS sludge (Yunqin *et al.*, 2009).

5. Conclusions

A comparison between the effectiveness of NaOH and KOH in terms of the hydrolysis of non-soluble complex cellulosic compounds in rice straw shows that the use of NaOH results in greater solubility compared with the use of KOH. The use of RSM based on CCD is practical for the simultaneous study of the effects of process variables on the SCOD value and the possible interaction between them. Further, the results indicated that a process for the production of biogas or compost from rice straw entails two stages: (1) alkaline pre-treatment followed by (2) anaerobic digestion or the composting process.

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