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Alkaline pre-treatment of rice hull and coconut hull using Response Surface Methodology

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Abstract

In this research, the rice hull and coconut hull from agriculture waste were investigated. The lignin content within the rice and coconut hull were determined before and after pre-treatment. Response Surface Methodology (RSM) used to predict the optimal condition for the treatments and Fourier Transform Infrared (FTIR) to identify the lignin content. The interaction of NaOH concentration, contact time (CT), and sample weight was investigated to optimize the lignin removal percentage (%). The correlation coefficient, R^2 for a quadratic model of rice hull lignin removal was 0.8863 while for coconut hull lignin removal in the linear model was 0.7998, as well as the 2FI model was 0.8892. The optimum condition for rice hull lignin removal predicted by RSM were10 M NaOH concentration, 1-hour CT and 0.5 g sample produced 32.45% lignin removal. While for coconut hull lignin removal were 10 M NaOH concentration, 12 hours CT, 0.5 g sample produced 59.47% removal of lignin. Results show that alkaline pre-treated rice hull and coconut hull able to be used to remove higher percentage of lignin.

1. INTRODUCTION

As the human population growing annually, rice has become a staple food among Asia countries due to its economic and it has become a dietary habit. Since there is unmilled rice or commonly known as paddy, there is a rice by-product (rice bran, hull, and germ). According to Ludueña *et al.* (2011), the composition of the rice hull for cellulose is 38%, hemicellulose is 20%, and lignin is 22%. Countries like Malaysia treat rice hull as agricultural waste and rice milling company did not take further action to manage the rice hull but just left it to decompose in the field or burnt it in open space. Meanwhile, since the coconut hulls or exocarp or coir are readily and easily available as waste from green coconut production in the hawker stall, thus numerous coconut hull fibre could be obtained. Zafar (2015) mentioned that there are 30% coconut fibres out of 40% of the coconut hulls. The composition of the coconut hull for lignin is 32.8%, hemicellulose is 56.3%, and cellulose is 4.2% according to Khalil *et al*. (2006). Lawrence (2010) stated that the major issue in the livestock industry is the feed cost, which occupies 60-70% of the total production cost. Besides, the rising cost in feed, as well as the feed shortage also gives rise to chaos among Asian farmers (Ahuja, 2012) and FAOSTAT (2010) stated that Asia has heavily import tonnes of maize as livestock feed 20 years previously. These troubles the farmer and they had to search for alternative feed to cope with this problem. Thus, rice hull and coconut hull were selected in this study. However, rice hull and coconut hull does undergone pre-treatment technology and the result could be used by some farmers as animal feedstuff. Pre-treatment which meant to enhance digestibility will somehow affect the fraction of the cellulose, hemicellulose, and lignin (Harmsen & Huijgen, 2010). Sodium hydroxide (NaOH) is proven to break lignin structure by degenerate both ester and glycosidic chains and modify lignin structure, cause cellulose to enlarge and interrupt crystalline structure in cellulose and hemicellulose (Mcintosh & Vancov, 2010). For this study, Central Composition Design (CCD) used because this design has three input factors responsible for the designing objective as well as the selected value according to the preliminary study which are diverged over five levels: minimum value (-1), middle value (0), maximum value (+1), and two outer points (-α and α) (Cho and Zoh, 2007).

2. MATERIALS AND METHODS

The rice hull and coconut hull were washed with tap water and dried in oven at 70°C for 24 hours and stored in airtight zipper bag under dry environment. This experiment used NaOH concentrations (A) with a range of 1-10 molar (M) to pre-treat 0.5-5.0-gram (C) rice hull and coconut hull samples at 1-12 hours contact time (B) (Pouteaua *et al,* 2003). The total numbers of experimental runs are 20 runs with different operating conditions. Each experiment will be run once, and the final weight of the pre-treated sample was measured. The determined values of each parameter were inserted into Design Expert software (Version 10.0) and the response surface methodology (RSM) were used by employing central composite design (CCD). Fourier Transform Infrared

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Spectroscopy (FTIR) technique was used to determine the chemical functional groups by identify the peak between the particular gap and band contained in rice hull and coconut hull after pre-treatment process. For rice hull and coconut hull sample, powdered form of the sample after pre-treatment and dried, the powder samples were used for FTIR analysis. Transmission for FTIR spectra of rice hull and coconut hull were recorded using Perkin Elmer spectrum in the 400-4000 cm^{-1} wavelength region. In this experiment and other alkali solution which include potassium hydroxide (KOH) and calcium hydroxide ($Ca(OH)_2$) were used to replace sodium hydroxide (NaOH) to confirm the alkali solution choose in this study was the best solution to remove lignin contain in rice hull as well as coconut hull (Gonçalves *et al.,* 2016). Analysis of variance (ANOVA) was used to support the relationship between the process parameters and the responses.

3. RESULT AND DISCUSSION

3.1 Optimization of lignin removal from rice hull

Optimization of lignin removal from rice hull was studied by using CCD where 20-experimental runs were carried out. NaOH concentration (A), contact time (B), and weight of sample (C) were selected as the parameters to investigate the most important factor that contributed to the lignin removal. Equation 1 demonstrated a complete empirical polynomial equation for lignin removal.

Rice hull lignin removal:

$$Y\ (\%) = 16.76 + 1.28A + 0.93B - 5.61C - 3.29AB - 5.93AC + 0.40BC - 3.98A^2 + 9.18B^2 - 5.08C^2 \quad (1)$$

This equation also showed factor A (NaOH concentration), factor B (contact time), and interaction factors of BC (contact time and weight of sample) have a positive response towards the lignin removal percentage. Factor A contributes the most towards the lignin removal due to it is the largest positive coefficient and the individual variation has more influence on the response apart from the interaction among the variation. Table 1 shows non-significant in lack of fit in this model which represent this model fit the experiment data well. There were 3.35 F-value in the lack of fit showed pure error was not significant and 0.1054 p-value in the lack of fit showed only 10.54% chance for F-value this large to appear because of noise. Hence, this model is appropriate for this experiment.

Based on Table 2, the most outstanding percentage of the lignin removal was 33.2% with the optimum condition of 10 M of NaOH concentration, 1 hours of contact time, and 0.5 g of sample weight in the actual value whereas the predicted value, 32.45% lignin removal. Furthermore, Figure 1 showed the residuals descended approximately to the straight which means that the errors distributed normally. Figure 2 showed that the residuals descended approximately to the straight which means that the errors distributed normally.

Table 1: ANOVA table for response surface quadratic model equation of rice hull

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	960.53	9	106.73	8.66	0.0011*
A	16.33	1	16.33	1.32	0.2765
B	8.67	1	8.67	0.70	0.4213
C	314.27	1	314.27	25.49	0.0005*
AB	86.59	1	86.59	7.02	0.0243
AC	281.32	1	281.32	22.82	0.0007*
BC	1.28	1	1.28	0.10	0.7539
A^2	43.61	1	43.61	3.54	0.0894
B^2	231.89	1	231.89	18.81	0.0015
C^2	71.03	1	71.03	5.76	0.0373
Lack of Fit	94.92	5	18.98	3.35	0.1054

* Represents that the value is significant
A-NaOH concentration; B-Contact time; C-Weight of sample in gram

Table 2: Results for actual values and predicted values of lignin removal from rice hull

Run Order	Coded Factors A	B	C	Lignin Removal (%) Actual Value	Predicted Value
1	0	0	0	13.31	16.76
2	0	0	0	20.58	16.76
3	0	0	-1	19.4	17.28
4	-1	1	-1	16.6	19.09
5	-1	1	1	19.44	20.54
6	0	0	0	16.33	16.76
7	1	-1	1	10.72	8.58
8	0	0	0	15.60	16.76
9	0	0	0	15.53	16.76
10	0	-1	0	24.98	25.01
11	-1	0	0	17.96	11.50
12	1	-1	-1	33.2	32.45
13	-1	-1	1	9.88	11.30
14	1	0	0	8.98	14.05
15	-1	-1	-1	10	11.45
16	0	0	1	5.34	6.07
17	0	0	0	16.4	16.76
18	1	1	1	5.76	4.66
19	0	1	0	28.29	26.87
20	1	1	-1	28	26.93

A-NaOH concentration; B-Contact time; C-Weight of sample in gram

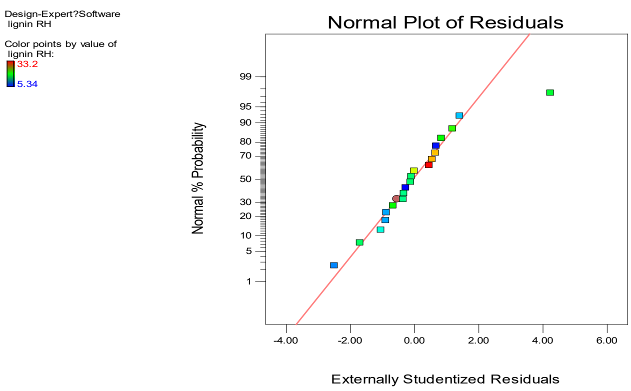

Figure 1: Plot of normal % probability versus residual error of lignin removal of rice hull.

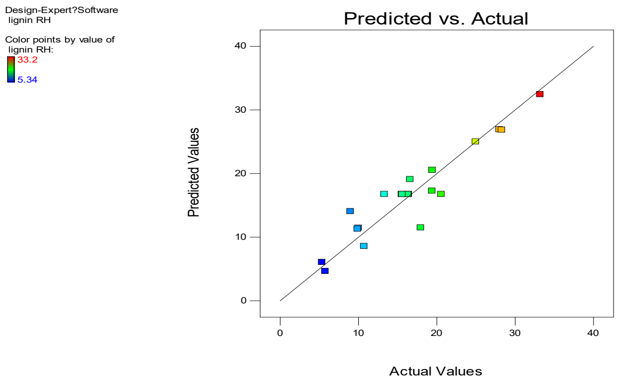

Figure 2: Diagnostic plot for predicted versus actual values for lignin removal of rice hull.

3.2 Effect of NaOH concentration, and contact time and weight of sample on lignin removal for rice hull

According to Figure 3 the NaOH concentration increase from 1 to 10 M provide more chances to exposed adsorption site in the rice hull interact with NaOH solution which eventually increase the rice hull lignin removal percentage (Ofomaja, 2008). The results show 10 M NaOH concentration able remove rice hull lignin percentage efficiently which indicated equilibrium system had achieved at 10 M thus further higher concentration gradually retarded (Banerjee & Chattopadhyaya, 2013). The maximum rice hull lignin removal percentage for both NaOH concentration and contact time interaction effect was 33.2% at the optimum condition of 10 M NaOH concentration and 1 hours contact time with 0.5 g constant weight of the sample.

Figure 4 shows the rice hull lignin removal gradually increases along the increase of NaOH concentration (M) from 1 M to 5.5 M and sample weight (g) from 0.5 g to 2.75 g, respectively. NaOH concentration after 5.5 M does not show an increase in the lignin removal percentage indicates that the alkali solution had saturated the rice hull binding site and eventually become a barrier for free chemical particles in the alkali solution to be absorbed which led to low lignin removal (El-Wakil *et al.*, 2015). On the other hand, the sample weight (g) after 2.75 g do not have shown the increase in lignin removal percentage indicated there is more vacant adsorption site in total surface area obtain in rice hull than provided by the adsorbate in NaOH solution (Nuengmatcha *et al.,* 2014). The maximum rice hull lignin removal percentage for both NaOH concentration and sample weight (g) interaction effect was 20.58% at the optimum condition of 5.5 M NaOH concentration and 2.75 g sample weight with 6.5 hours constant contact time.

Figure 5 displayed the rice hull lignin removal percentage increase gradually with the increase of contact time (hours) from 1 hour to 12 hours and sample weight (g) from 0.5 g to 2.75 g, respectively. This indicated that at 2.75 g of the sample had reach equilibrium even in longest contact time in this study and further contact time may be needed to obtain better lignin removal. The maximum rice hull lignin removal percentage for both contact time (hours) and sample weight (g) interaction effect was 28.29% at the optimum condition of 12 hours contact time and 2.75 g sample weight with 5.5 M constant NaOH concentration.

The numerical optimisation of rice hull using the desirability function was illustrated in Figure 6. The results demonstrated the highest value of desirability represented by red colour is more than 0.8 desirability which is approximately to 1.

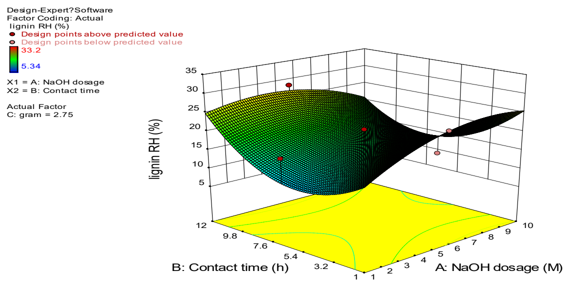

Figure 3: 3D response surface graph of the interaction effect of NaOH concentration (M) and contact time (hours) on lignin removal (%).

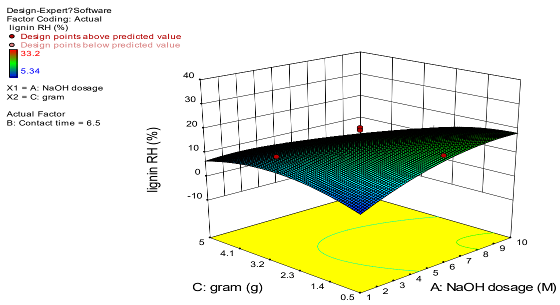

Figure 4: 3D response surface graph of the interaction effect of NaOH concentration (M) and sample weight (g) on lignin removal (%).

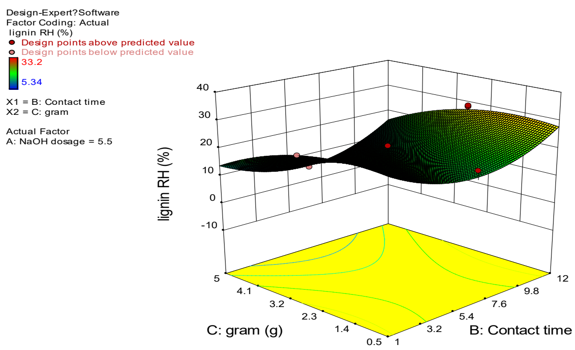

Figure 5: 3D response surface graph of the interaction effect of contact time (hours) and sample weight (g) on lignin removal (%).

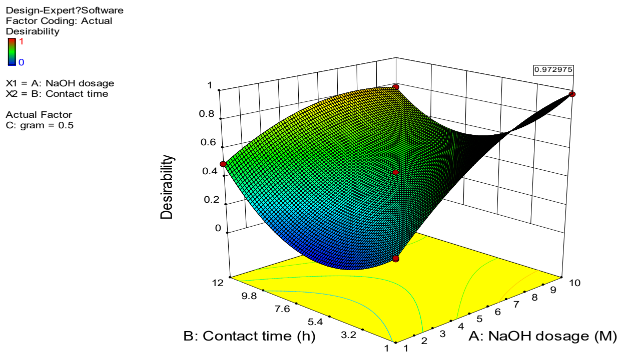

Figure 6: 3D response surface graph of the interaction effect of NaOH concentration (M) and contact time (hours) on lignin removal (%).

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3.3 Optimization of lignin removal from coconut hull

Optimization of lignin removal from coconut hull was also studied by using CCD where 20-experimental runs were carried out and the three parameters were similar with previous experiment.

Coconut hull lignin removal:

$$Y\ (\%) = 24.87 - 0.12A + 5.94B - 15.92C + 0.88AB - 3.66AC - 5.12BC \quad (2)$$

According to Equation 2, the positively sign coded of factor B (contact time) and factor AB (NaOH concentration and contact time) has significance positive impact towards the lignin removal. Among the positive sign factors, individual coded factor B has more influence on the response apart from interaction among the variation.

Table 3 shows smaller p-value than 0.05 (95% confidence level) in this study supported the statistical significance of the model terms. Besides, factor B, C, and BD less than 0.05 show that the model terms were significant. The value excess 0.1000 is insignificant for the model. Table 3 presented 17.39 F-value and less than 0.0001 p-values which means there was less than 0.01% chance for large F-value to noise. From the table, factor C (gram) has the largest F value which is 82.37 indicated this was the most vital factor in this model.

Based on Table 4, the most outstanding percentage of lignin removal was 63.60% with the optimum condition of 10 M of NaOH concentration, 12 hours of contact time, and 0.5 g of sample weight. In the actual value whereas the predicted value, 56.26% lignin removal. Furthermore, Figure 7 and 8 showed that the residuals descended moderately which means that the errors distributed normally. It defined that the predicted value was fitted to the actual experimental run value.

Table 3: ANOVA table for response surface quadratic model equation of coconut hull

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	3210.03	6	535.01	17.39	< 0.0001*
A	0.15	1	0.15	4.918E-003	0.9452
B	353.07	1	353.07	11.48	0.0049
C	2533.83	1	2533.83	82.37	< 0.0001*
AB	6.16	1	6.16	0.20	0.6619
AC	107.31	1	107.31	3.49	0.0845
BC	209.51	1	209.51	6.81	0.0216
Lack of Fit	387.72	8	48.47	19.93	0.0022*

* represents that the value is significant
A-NaOH concentration; B-Contact time; C-Weight of sample in gram

Table 4: Results for actual values, predicted values of lignin removal from coconut hull.

Run Order	Coded Factors A	B	C	Lignin removal (%) Actual Value	Predicted Value
1	0	0	0	19.96	24.87
2	0	0	0	20.91	24.87
3	0	0	-1	47.8	40.78
4	-1	1	-1	44.4	47.43
5	-1	1	1	19.54	12.68
6	0	0	0	20.69	24.87
7	1	-1	1	11.16	3.46
8	0	0	0	23.82	24.87
9	0	0	0	23.31	24.87
10	0	-1	0	21.75	18.92
11	-1	0	0	24.91	24.99
12	1	-1	-1	30.2	32.39
13	-1	-1	1	10.12	12.79
14	1	0	0	16.18	24.74
15	-1	-1	-1	31	27.06
16	0	0	1	9.4	8.95
17	0	0	0	22.44	24.87
18	1	1	1	7.6	6.86
19	0	1	0	28.51	30.81
20	1	1	-1	63.6	56.26

A-NaOH concentration; B-Contact time; C-Weight of sample in gram

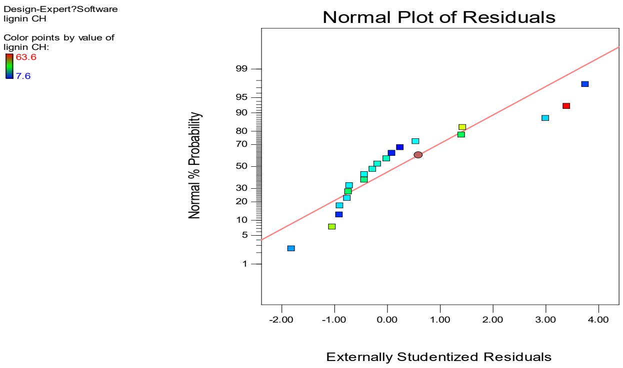

Figure 7: Plot of normal % probability versus residual error of lignin removal of coconut hull.

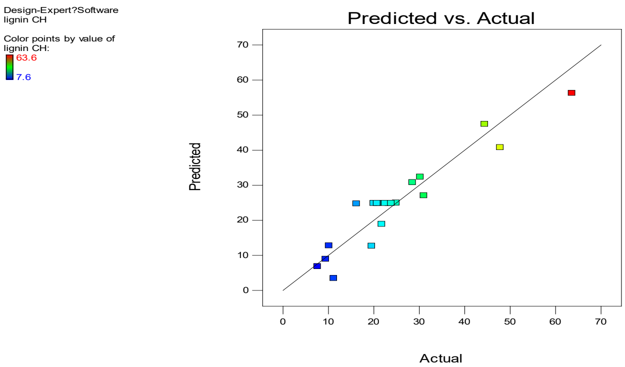

Figure 8: Diagnostic plot for predicted versus actual values of lignin removal of coconut hull.

3.4 Effect of NaOH concentration, and contact time and weight of sample on lignin removal for coconut hull

Figure 9 displays the red zone act as the optimum response value of 63.6%. The figures displayed the coconut hull lignin removal percentage increase gradually along the increase of contact time (hours) from 1 hour to 6.5 hours and NaOH concentration (M) remain constant at 1 M respectively. The maximum coconut hull lignin removal percentage for both NaOH concentration (M) and contact time (hours) interaction effect was 24.91% at the optimum condition of 1 M NaOH concentration and 6.5 hours contact time with 2.75 g constant weight of the sample.

Figure 10 displayed the coconut hull lignin removal percentage increase gradually along the increase of NaOH concentration (M) from 1 M to 5.5 M and sample weight remain constant at 0.5 g respectively. The maximum coconut hull lignin removal percentage for both NaOH concentration (M) and weight of sample (g) interaction effect was 47.80% at the optimum condition of

5.5 M NaOH concentration and 0.5 g weight of sample with constant 6.5 hours contact time.

Figure 11 displayed the coconut hull lignin removal percentage increase gradually along the increase of contact time from 1 hour to 6.5 hours and sample weight remain constant as 0.5 g respectively. The maximum rice hull lignin removal percentage for both contact time (hours) and weight of sample (g) interaction effect was 47.80% at the optimum condition of 6.5 hours contact time and 0.5 g weight of sample with constant 0.5 M NaOH concentration.

3.5 Optimisation of Coconut Hull using the Desirability Function

Experimentally, the best optimum values were 63.6% coconut hull lignin removal percentage while the Design Expert Software Version 10 predicted coconut hull lignin removal percentage which is 59.47% with the 0.926 desirability near to 1. This indicates NaOH concentration is a significant variable in this coconut lignin removal. Figure 12 demonstrated the interactive response of the NaOH concentration (M) and contact time (hours) on lignin removal (%) with 0.5 g weight of sample remain constant. The highest value of desirability represented by red colour is more than 0.9 desirability is approximate to 1.

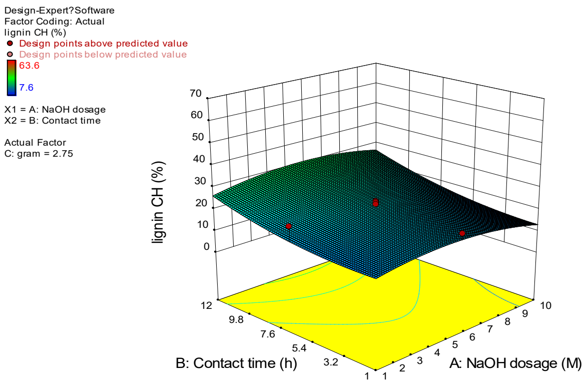

Figure 9: 3D response surface graph of the interaction effect of NaOH concentration (M) and contact time (hours) on lignin removal (%).

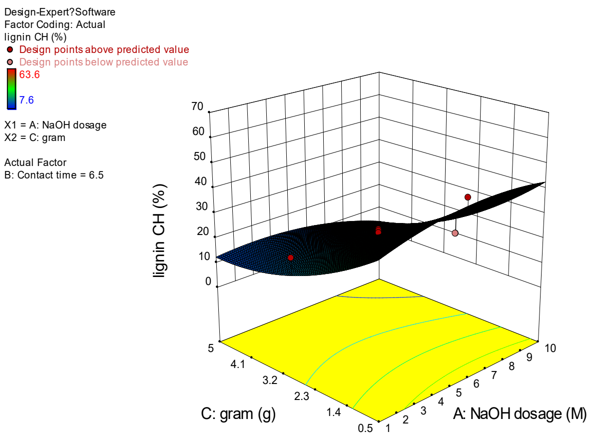

Figure 10: 3D response surface graph of the interaction effect of NaOH concentration (M) and sample weight (g) on lignin removal (%).

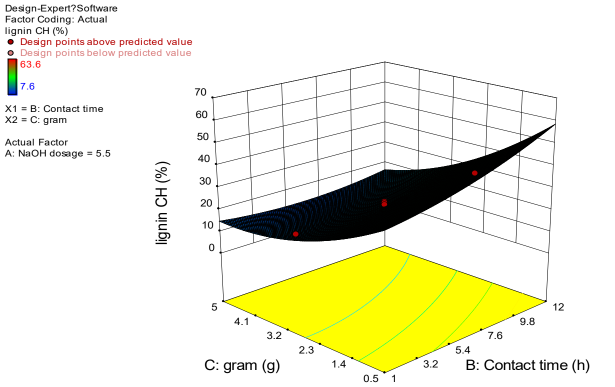

Figure 11: 3D response surface graph of the interaction effect of contact time (hours) and sample weight (g) on lignin removal (%).

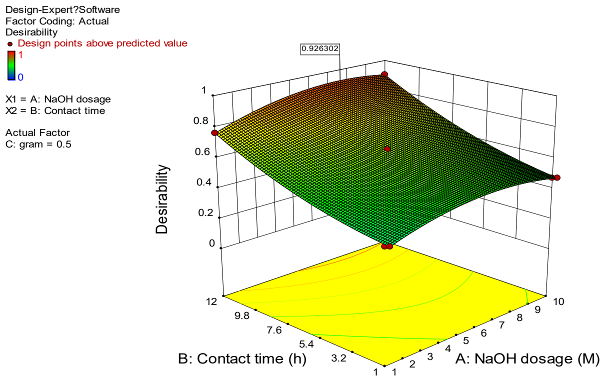

Figure 12: 3D response surface graph of the interaction effect of NaOH concentration (M) and contact time (hours) on lignin removal (%).

3.6 FTIR Spectra Analysis

Among the functional group, the significance chemical functional group to determine the accuracy of the lignin existence were hydroxyl (-OH), methoxyl ($O\text{-}CH_3$), carboxyl (-COOH), and carbonyl (C=O) groups (Shamsuri & Abdullah, 2010). Figure 13 showed the absorption peak of pre-treated rice hull decreased after pre-treatment process from standard untreated rice hull, 3403.31 cm^{-1} to around 3334.06 cm^{-1} indicated the presence of stretched -OH group and the amine group in pre-treated rice hull. The stretching of C=C vibration of 1632.49 cm^{-1} indicates alkenes and aromatic functional groups. The peak in 475.10 cm^{-1} showed the presence of -Si-H group. The peak in 1026.49 cm^{-1} indicates the presence of C=O ester group, a C-O ether group, and an alkyl halide group. The peak around 456.48 cm^{-1}, 447.76 cm^{-1}, 438.38 cm^{-1}, 429.08 cm^{-1}, 418.10 cm^{-1}, and 409.75 cm^{-1} showed the presence of $-OCH_3$ which have lower wavelength than untreated rice hull (580 cm^{-1}).

In Figure 14, the peak around 3630 cm^{-1} to 2980 cm^{-1} for untreated coconut hull were reduced to 3288.61 cm^{-1} in treated coconut hull indicates the presence of alcohol OH stretching. The C-H stretching vibration peak at 2900 cm^{-1} for untreated coconut hull to around 2922.47 cm^{-1} for treated coconut hull confirmed the alkane (cellulose and lignin) functional group emerged. The untreated coconut hull peak at 1700 cm^{-1} reduced to the treated coconut hull of C=O stretching vibration around 1605.74 cm^{-1} indicate the presence of carbonyl group with stretching of an ester linkage between carboxylic groups of lignin and hemicellulose. The peak around 1412.70 cm^{-1}

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indicates the presence of C=C stretching aromatic functional group and a -C-H bending bond. The peak at 1015.19 cm^{-1} C-F indicates the presence of alkyl halide group and C-O ether group. The peak around 525.74 cm^{-1} and 479.85cm^{-1} indicates the presence of Si-H group.

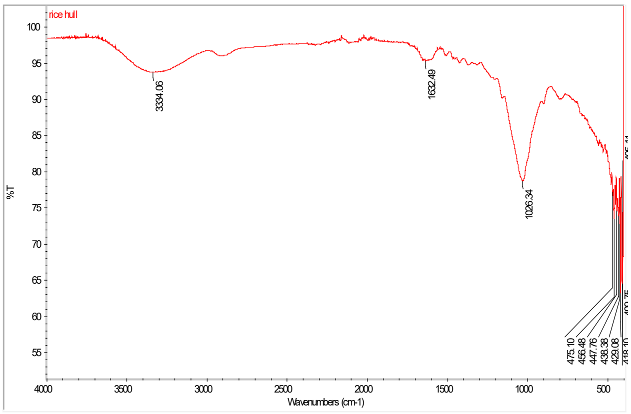

Figure 13: FTIR spectra identification of the treated rice hull.

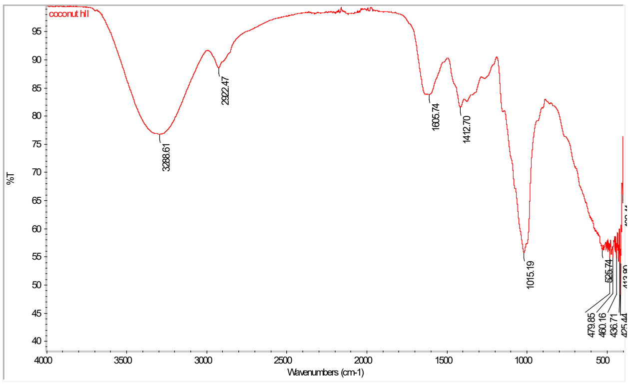

Figure 14: FTIR spectra identification of the treated coconut hull.

3.7 Comparison of Different Types of Alkaline Solution

Table 5 and 6 showed results for lignin removal in different alkaline solution for rice hull and coconut hull respectively. Both experiments showed that NaOH solution was superior compared to KOH and Ca $(OH)_2$ solutions.

4. CONCLUSION

The removal of lignin from rice hull and coconut hull by using alkaline pre-treatment designed via Response Surface Methodology (RSM) have been demonstrated and the results proved that this pre-treatment method using 10 M NaOH was appreciable and very efficient to remove high percentage of lignin within 12 hours for 0.5-gram samples.

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