Optimization of Pretreatment For Wood Waste By Design Expert

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Abstract- *Biofuel has attained more focus in the recent decade. Gaseous biofuels are derived from many ways and it is working well within certain limit. Main concern now is on liquid bio fuel. Which is the alternative for the liquid fossil fuels. Biodiesel is normally produced from Edible oil and in recent days from non edible oil. Concentrations were shifted to produce biodiesel by biotechnological applications by using glucose present in the plant cells. Preparation of raw plants play vital role for lipid production. In this study Pretreatment was carried out by using alkali aided with ultra-sonication. Also it the process was optimized by design expert software. Based on the study highest C/N ratio was obtained is380 mg/l. The Optimum organic loading was found out as 7.5 gram and the optimum ultrasonication time was found out as 25 min using design expert software.*

Keywords- Waste, Pretreatment, Hydrolysis, Ultrasonication, Design expert

I. INTRODUCTION

The increasing scarcity of conventional fossil fuels, growing emissions of combustion-generated pollutants and their increasing costs have made biofuel sources more attractive in the past few decades. The current alternative fuels, the only liquid fuel can be termed as biodiesel. These biofuels are extracted from the various types of biomass including waste wood, organic waste biomass available around the world. These biomasses are produced from the lipids that were present in the organic waste material. Production of biofuel from the organic waste by the method of trans-esterification depends on the availability of lipid and biomass. Increasing the production of lipids is based on effective pretreatment methodology of conversion of complex carbohydrates. Pretreatment is the process of breakdown the complex substrate into simple substrate. Pretreatment process is effectively breakdown of the molecular bonds was increasing the carbon / nitrogen percentage. So it is directly increasing the lipid production for producing higher yield of biomass.

This paper focussed on effect of various pretreatment processes for wood organic waste that was collected from the government college of technology, Coimbatore. In this study, wood waste which was containing higher amount of cellulose, hemi cellulose and lignin was chosen for pre-treatment studies. So thermal, alkaline, acid and alkaline ultrasonication pretreatments were conducted for wood waste. Thermal pretreatment is the one of the physical pretreatment methodology. By the applying of heat energy which inducted collusion in between the particle so the breakdown process was done. Chemical pretreatment that are acid and alkaline were performed using sulfuric acid and sodium hydroxide respectively. Alkaline ultra-sonications was found to be the appropriate physico-chemical method where dual effect accounted for the breakdown the complex substrates.

II. METHODS AND MATERIALS

2.1Material Collection

The wood waste was collected from Government College of Technology, Coimbatore. Wood waster were contains higher amount of cellulose, hemi cellulose and lignin. Normally wood waste contains cellulose was 38.14, hemicellulose 28.50 and lignin 34.The collected wood waste were broken into the small pieces and dried in sun light for a period of one day and then oven dried at 110°C for four hours. The wood waste was grinded and then the sample was sieved by using 75µm as wood waste contains higher amount of fibers. These wood waste fibers cannot be broken easily by the normal pretreatment methods.

2.2Ball Milling

Sieved sample were ball milled because of size of the sample particle need to be reduced for effective breakdown of cell wall. The samples were Ball milled using planetary ball mill with titanium carbide balls at the 120 RPM for one hour at the atmospheric temperature.

2.3Pretreatment

2.3.1Alkaline- Ultrasonication Treatment

1, 2 and 3 grams of organic loads were taken in a 500ml of conical flasks and suspended with the 1% NaOH concentration. The content were mixed well and sonicated for 30min at 25 kHz frequency with an effective ultrasonic power of 150 W using a bath ultrasonicator. The resultant liquid after ultrasonication was analyzed for C/N ratio, using TOC Analyzer.

2.3.2 Batch study for alkaline ultrasonication

From result of C/N ratio for various pretreatment methods analyzed above. It was concluded that the effective pretreatment method for wood waste biomass was Alkaline-ultrasonication.

For the process of optimization of various parameters for enhanced lipid production, batch study conduted for this particular effective pretreatment method of Alkaline-ultrasonication.

2.3.3Batch study

Hence the two parameters that are organic loading and ultrasonication time were taken as variables. The other parameters like frequency (25 kHz) were maintained as constant. Alkaline concentration was kept as 1% v/v of 1N NaOH.

Organic loadings were taken as 1, 5 and 10 grams and ultrasonication times were varied as 10, 20 and 30 minutes. The various experiments were done for these combinations are shown in table.3.1.

2.4 Optimization

The optimization of important variables was carried out using Response Surface - Central Composite Experimental Design. The method used in response surface for optimization was steepest accent. In central composite design, each variable was varied into 5 levels and coded –α, -1, 0, 1, and α which represent minimum, low, center, high, and maximum level, respectively. This low and high level for each variable were determined using experimental data from batch study, in which the organic loading range between 5 to 10 grams and ultrasonication time in the range between 20 to 30 minutes. Experimental design points were arranged with the assistance of Design Expert software version 7.00.

2.4.1 Response Surface Analysis Study

Design set of combination were arrived at from the design expert 7.0 software, after feeding the limits that was obtained from batch study. For design purpose the limits were taken in the range -α to + α. Hence the range analyzed to the software was 4 to 11 grams and 18 to 32 min of organic loading and ultrasonication time respectively.

These various set of samples were studied in the given various factors conditions. That are shown in the figure 3.8.After the various factors condition were studied and analyzed the values of TOC and TN are obtained from TOC analyzer. The C/N ratio was found for all set of design combinations.

S.No	Organic loading (grams)	Ultrasonication time (minutes)	Frequency (kHz)	Alkaline concentration (ml)1% NaOH
1	1	10	25	100
2	1	20	25	100
3	1	30	25	100
4	5	10	25	100
5	5	20	25	100
6	5	30	25	100
7	10	10	25	100
8	10	20	25	100
9	10	30	25	100

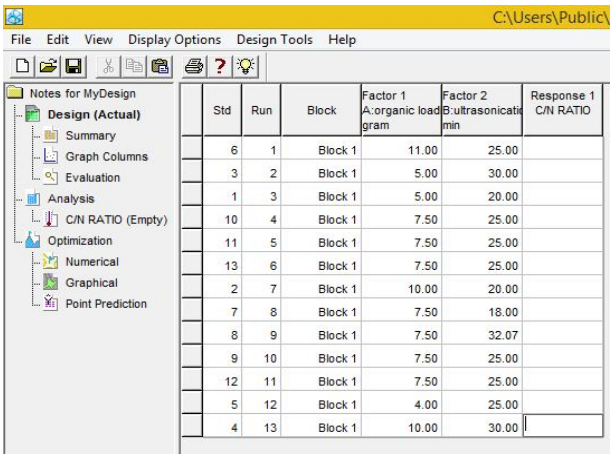


Fig 2.1Various set of response analysis study

III. RESULTS AND DISCUSSIONS

3.1Alkaline – Ultrasonication Treatment

The sample before sonication was suspended with the 1% (v/v) 1N NaOH solution and ultrasonication were conducted. Then the samples were analyzed, TOC and TN values were obtained from the TOC analyzer, that was shown in the Graph.4

Graph.4 ALKALINE – ULTRASONICATION TREATMENT

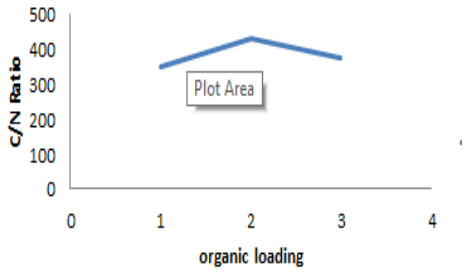


Fig. 3.1 Alkaline pretreatment

From the experimental results it can be inferred that the maximum value of C/N ratio for alkaline ultrasonication was 428. Due to the dual effect of alkaline and ultrasonication.

3.2 Batch Study Result

Batch study was conduted on alkaline ultrasonication method and the samples of TOC and TN were analyzed by using TOC analyzer, which was shown in the batch study table.

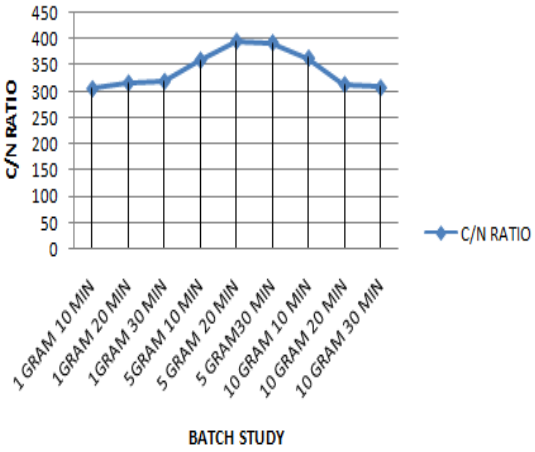


Fig. 3.2 Batch Study

3.3 C/N Ratio for Batch Study

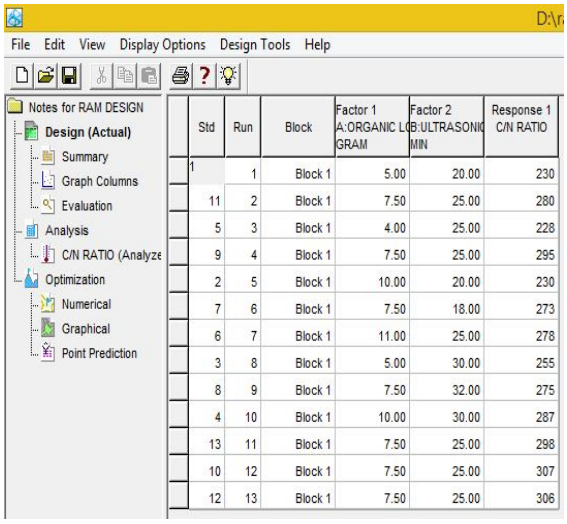
From the batch study analysis the optimum contions lies between the 5 to 10 gram of organic load and the ultrasonication time in between 20 to 30 min. so the graph, shown that 5 gram of 20 min, 5 gram of 30 min and 10 gram of 10 min gave the high C/N ratio.

Hence for optimization purpose the parameter of organic load and ultrasonication time were taken in the range between 5 gram to 10 gram and 20 min to 30 min respectively.

3.4 Optimization Study

In this optimization process the variable of factors maximum and minimum limit were found from the batch study analyzis. Hence the organic load and ultrasonication time were taken as the variable factors. The limits in the range between 5 to 10 gram and 20 to 30 min for organic loading and ultrasonication time respectively. Hence this limits were feed into the Design expert 7.0 software.

Then the sofware gave the various set of combinasion of given factors. That was analyzed for C/N ration by used TOC analyzer. Hence the C/N ratio of the samples were taken as a response. Where optimization were obtained based by the these response (C/N ratio). That found response values were feed in to the response column in the software.



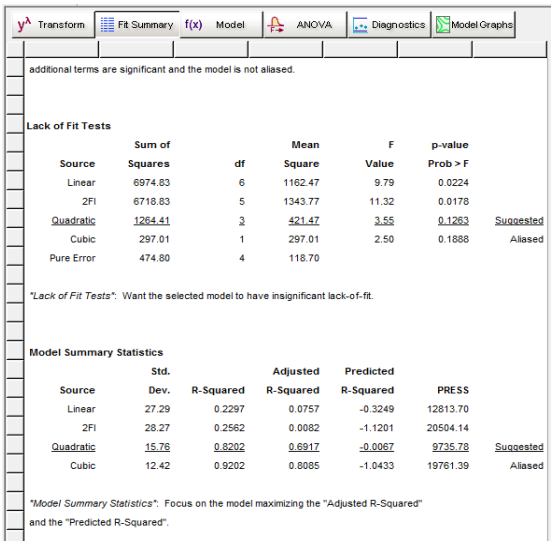
	Std	Run	Block	Factor 1 A:ORGANIC LOAD GRAM	Factor 2 B:ULTRASONIC MIN	Response 1 C/N RATIO
1	1	1	Block 1	5.00	20.00	230
11	2	1	Block 1	7.50	25.00	280
5	3	1	Block 1	4.00	25.00	228
9	4	1	Block 1	7.50	25.00	295
2	5	1	Block 1	10.00	20.00	230
7	6	1	Block 1	7.50	18.00	273
6	7	1	Block 1	11.00	25.00	278
3	8	1	Block 1	5.00	30.00	255
8	9	1	Block 1	7.50	32.00	275
4	10	1	Block 1	10.00	30.00	287
13	11	1	Block 1	7.50	25.00	298
10	12	1	Block 1	7.50	25.00	307
12	13	1	Block 1	7.50	25.00	306

Fig. 3.3 various set of response feeded combinations

RESPONSE SURFACE ANALYSIS

Various factors were studied and the respective response values were obtained after analyzed the the sample in the TOC anlayzer that was feeded into the software for response surface analysis.

The response surface analysis gave the result of fit to the parameters for optimization. The model of the given sample was QUADRATIC because the R- SQUARE value were 0.8202. it was shown that the given sample fit was good but the R- SQUARE value was less than the 90% so in the sample fit was good. And the adjusted R- SQUARE value was 0.6917, so the model was lies in the QUADRATIC Fit.



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additional terms are significant and the model is not aliased.

Lack of Fit Tests

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Linear	6974.83	6	1162.47	9.79	0.0224	
2FI	6718.83	5	1343.77	11.32	0.0178	
Quadratic	1264.41	3	421.47	3.55	0.1263	Suggested
Cubic	297.01	1	297.01	2.50	0.1888	Aliased
Pure Error	474.80	4	118.70			

*"Lack of Fit Tests": Want the selected model to have insignificant lack-of-fit.

Model Summary Statistics

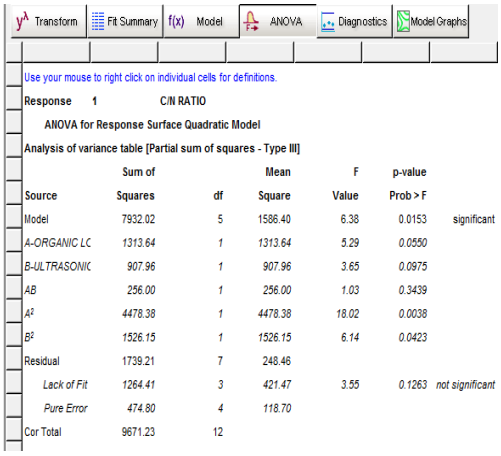
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	27.29	0.2297	0.0757	-0.3249	12813.70	
2FI	28.27	0.2562	0.0082	-1.1201	20504.14	
Quadratic	15.76	0.8202	0.6917	-0.0067	9735.78	Suggested
Cubic	12.42	0.9202	0.8085	-1.0433	19761.39	Aliased

*"Model Summary Statistics": Focus on the model maximizing the "Adjusted R-Squared" and the "Predicted R-Squared".

3.5 Response Surface Analysis
anova result

From the analysis the model was significant, the prob > f value was 0.0153, it was less than the 0.0500.so the model was fit for the optimization.

The lack of fit for the model was not significant , the value of 0.126 shown that the value of lack of fit were 3.55.



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Response 1 C/N RATIO

ANOVA for Response Surface Quadratic Model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	7932.02	5	1586.40	6.38	0.0153	significant
A-ORGANIC LC	*1313.64*	*1*	*1313.64*	*5.29*	*0.0550*	
B-ULTRASONIC	*907.96*	*1*	*907.96*	*3.65*	*0.0975*	
AB	*256.00*	*1*	*256.00*	*1.03*	*0.3439*	
A^2	*4478.38*	*1*	*4478.38*	*18.02*	*0.0038*	
B^2	*1526.15*	*1*	*1526.15*	*6.14*	*0.0423*	
Residual	1739.21	7	248.46			
Lack of Fit	*1264.41*	*3*	*421.47*	*3.55*	*0.1263*	*not significant*
Pure Error	*474.80*	*4*	*118.70*			
Cor Total	9671.23	12				

Fig. 3.4 Anova model

3.6 Optimization Result:

The result of optimization gave the value of 7.50 gram and 25 min of organic load and ultrasonication were obtained ad optimum value for given modal.That was gave the predicted value of C/N was 297.298. The various 30 set of combination that were run by the design expert software finally gave the value of maximum probability of optimum value as organic load of 7.50 grams and 25 min.

It was conformed that the optimum value of two factors in the peak of the contour.

Then the analysis gave the equation of optimum yield of C/N ratio.

Factor	Name	Level	Low Level	High Level	Std. Dev.	Coding
A	ORGANIC LOA	7.50	5.00	10.00	0.000	Actual
B	ULTRASONICA	25.00	20.00	30.00	0.000	Actual

Response	Prediction	SE Mean	95% CI low	95% CI high	SE Pred	95% PI low	95% PI high
C/N RATIO	297.298	7.05	280.63	313.97	17.27	256.47	338.13

Fig. 3.5 Predicted optimum factors

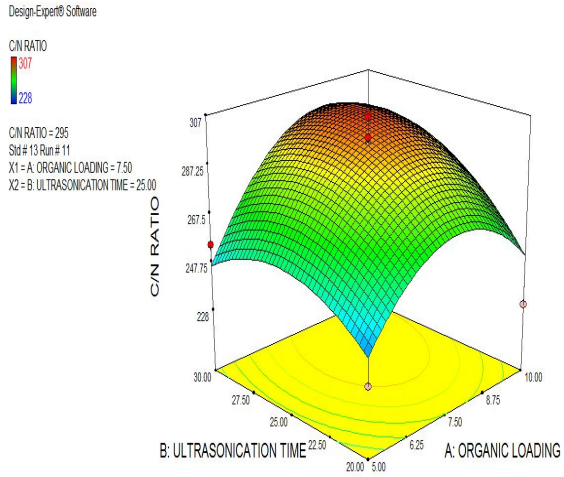

Fig. 3.6 3D view of optimized factors

The model obtained basen batch study results is given as

C/N RATIO =
-282.20948
+50.92987 * ORGANIC LOADING
+27.39481 * ULTRASONICATION TIME
+0.64000 * ORGANIC LOADING * ULTRASONICATION TIME
-4.11856 * ORGANIC LOADING^2
-0.60107 * ULTRASONICATION TIME^2

IV. CONCLUSION

From the study and the results obtained, Alkali pretreatment aided with ultrasoination gives good amount of cell disruption and gave maximum C/N ratios 380mg/l. the optimum organic loading was found out as 7.5 mg/l and the optimum ultrasonication time was determined as 25 min. Also

the design expert softawres's optimized conditions are matching with experimental results. Hence the model obtained by design expert software for the alkali pretreatment aided with ultrasonication is more reliable for further study.

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