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PROCESS PARAMETER OPTIMIZATION OF LOW TEMPERATURE TRANSESTERIFICATION OF ALGAE-JATROPHA OIL BLEND

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Abstract- The use of energy sources has reached at the level that whole world is relying on it. Being the major source of energy, fuels are considered the most important. The fear of diminishing the available sources thirst towards biofuel production has increased during last decades. Considering the food problems, algae and jatropha gain the most attention to be used as biofuel producers. The use of crop and food-producing plants will never be a best fit into the priorities for biofuel production as they will disturb the food needs. We can find the ways to achieve the required biofuel amounts easily and continuously to overcome the fuels deficiency. A model was developed to correlate the biodiesel yield with process parameters viz methanol/oil volumetric ratio, Catalyst concentration and reaction time. A biodiesel yield of 87.59% was achieved with methanol/oil volumetric ratio (3:5) using H2SO4 as catalyst (0.9% w/w) in 180 min time at 50°C temperature. It was observed that catalyst concentration, reaction time and methanol/oil volumetric ratio had a significant effect on blend yield.

Keywords: Jatropha, Algae, Biodiesel, RSM, Optimization

I. INTRODUCTION

Energy crisis is among the biggest problems, leading the world to be unsafe and non-peaceful. The demand is increasing day by day. The available resources are rapidly decreasing and indication is, soon will be vanished. In such situations, more attention is needed to be given towards renewable energy sources. Fossil fuels are used on a large scale in the world, but unsustainable because they increase CO2 level and accumulate greenhouse gases which make the environment unhealthy. To keep the environment clean and maintain sustainability, renewable and environmentally friendly fuels are needed to be produced [1]. Biofuels are defined as the liquid fuels produced from the biomass of different agricultural and forest products and biodegradable portion of industrial waste. Biodiesel is extracted from vegetable oils, biobutanol, Jatrophacurcas and algae. Brazil, the United States and the European Union are the world's largest biodiesel producers. Biofuel production has been estimated to be 35 billion litres [2].The algae are now becoming the main source of biofuel production

in the world. They are considered as the safer, non-competitive and rapidly growing organism among those could be used for biodiesel production. They have the abilities to grow without much care on waste nutrients, and are considered the better source of biodiesel production as other sources can cause food problems as they are mainly including those plants which are used for food [3]. Algae based biofuels requires a lot of energy and water and may lead to more GHG emissions than crop based biofuels over its complete life-cycle. On the other hand, the preparation of biodiesel from JatrophaCurcas oil is a promising alternative in the present situation due to its higher oil content and non-edible nature [4]. Indian government has emphasized on JatrophaCurcas oil production under national biodiesel mission since 11th five year plan. The oil can be converted to biodiesel using transesterification, but the type of transesterification to be adopted depends on the free fatty acid (FFA) content of the oil. For the conversion of high FFA JCO, two-step acid-base catalyzed method has been-developed which consists of acid-catalyzedpretreatment/esterification step to reduce the FFA to less than 1% using H2SO4 as acid catalyst and transesterification of pretreated oil to biodiesel using alkali catalyst. This process consume extra time also [5]. Above problems of pretreatment of high FFA oil can be reducedby making a blend of both algae oil and Jatrophacarcus oil, since it will not only reduce the amount of algae oil used but also the acid catalyzed esterification of Jatrophacurcas oil is not required Transesterification of the blend involveslarge number of parameters affecting the reaction, therefore optimization of these reaction factors needs a large number of experiments which is time consuming and uneconomical.

Researchers have applied various experimental techniques to optimize the process variables for biodiesel production. Gaurav et al. [6] implemented the BoxeBehnken response surface methodology (RSM) for maximizing the biodiesel yield to 98.4% from Pongamia oil by optimizing the four process variables. Siddiqua et al. [7] used BoxeBehnken response surface methodology to optimize three variables for predicting the best performing conditions (calorific value and yield) of algae biodiesel. Bandhu et al. [8] used RSM to Statistical design and optimize the single cell oil production from sugarcane bagasse hydrolysate by an oleaginous yeast Rhodotorula sp. IIP-33. Dasgupta et al. [9] used RSM to design and optimize the ethanol production from bagasse pith hydrolysate by thermotolerant yeast Kluyveromycessp. IIPE453. RSM was also used by Sukjit and Punsuvon[10] for maximizing the production of the Jatropha biodiesel to 93.55% using CaOeMgO mixed oxide catalyst in transesterification process. Lee et al. [11] used RSM for maximizing the production of the Jatropha biodiesel to 96.57%. Vicente et al. [12] applied RSM to optimize the biodiesel production from refined sunflower oil. Carvalho et al. [13]
Optimized the process variables using RSM for maximizing the production of biodiesel from cotton seed oil to 96.79%. Renita et al. [14] produced biodiesel from the macroalgaeCaulerpapeltata using central composite design to optimize the parameters like oil: alcohol ratio, catalyst amount, time and temperature. Xinyu et al.[15] used RSM and solid catalyst Cs2O/ceAl2O3 for production of biodiesel from animal fat, the biodiesel yield was found to be 95.5%. Betiku and Adepoju[16] used RSM to investigate the biodiesel production from sesame oil with, highest conversion yield of 99.71% obtained under the optimized conditions. Aworanti et al. [17] investigated the effects of methanol-to-oil molar ratio, catalyst amount and reaction time on the transesterification of waste cooking oil (WCO) to biodiesel using RSM. Orthai et al. [18] used box Behnken design for RSM for optimizing the operating condition to reduce COD, O&G, and SS by 55.43%, 98.42%, and 96.59%, respectively. Junhua Zhang et al. [19] used box Behnken design for RSM to optimize the conditions

for ZSO biodiesel production using CaO as a catalyst at this optimum condition, the conversion to biodiesel reached above 96%.

The aim of the present paper, therefore, is to analyse the effect of process parameters on transesterification process and yield of biodiesel. A model was formulated and validated for predicting the response. It is found out that this model can be used in the industry to improve the efficiency of biodiesel production from the blend thereby, saving time and cost of the process in optimizing the process parameters.

II. MATERIAL AND METHOD

The paper reports the results of the optimization of three process variables viz. catalyst (H2SO4) concentration (0-2% w/w), reaction time (60-180 min) and methanol/oil ratio (v/v) (20-60%) for the transesterification process of blend of algae oil and Jatrophacarcus oil at reaction temperature of 50°C using RSM based Box-Behnken Design in 17 experimental runs with the help of Design Expert 11 software. JCO was procured from JatrophaVikasSansthan, New Delhi, India. All chemicals like H2SO4 and methanol were of analytical reagent grade and 99%pure. H2SO4 was used as a acid catalyst. The fuel properties of JatrophaCurcas Oil and algae oil after refining were determined as per standard methods. FFA content Jatrophacurcas oil was calculated very high as 22% while that of algae oil was 0.5% therefore these two oils were blended (1:42 (v/v)) to get the final FFA of 1%. Table 1 shows that a FFA content of the blended oil is as 1.0%. Owing to low FFA content, here we have used base catalyzedtransesterification processes.

III. EXPERIMENTAL DESIGN

A Box-Behnken experimental design, with three variables, was used to study the response pattern and to determine the optimum combination of variables. The effect of the C (ratio of methanol to oil, (v/v)) A (reaction time (min)), and B (catalyst amount (w/w %), at three variables levels in the reaction process is shown in Table 2. A total of 17 experiments were conducted separately for getting the experimental response of yield. The methanol/oil ratio (v/v) (C), reaction time (A) (min), and catalyst concentration (B) (%) were the independent variables selected for optimization. The coded and uncoded levels of the independent variables used for the transesterification of JatrophaCurcas Oil are given in Table 2.

3.1. Statistical Analysis

The Design Expert 11 software is used for the regression and graphical analysis of the data. The maximum values of blend (JatrophaCurcas þ algae) yield were taken as the response of the design experiment for transesterification process. The experimental data obtained by the above procedure was analyzed by the response surface regression using the polynomial Equation.
where Y is the response, i and j are the linear and quadratic coefficients respectively, xi and xj are the uncoded independent variables, is the regression coefficient, k is the number of factors studied and optimized in the experiment. Equation was also validated by carrying out confirmatory experiments.

3.2. Transesterification

Blend of JatrophaCurcas oil and algae oils was transesterified by using methanol and H2SO4 as base catalyst for the production of biodiesel. The methyl ester layer was separated and processed according to Dwivedi and Sharma [5]. Transesterification of oil blend has been optimized using RSM for the maximization of biodiesel yield.

3.3. GC analysis

The optimized sample was analyzed for fatty acid composition by Gas Chromatograph (Netal make) equipped with a flame ionization detector and a capillary column for injecting the sample. The GC oven was kept at 230°C (5°C/min) and a total analytical time was 30 min. Nitrogen was used as carrier gas. Quantitative analysis of % ME was done using European standard EN 14103:2003 [19]. Free fatty acids in the samples were determined using stock solution (Methyl heptadecanoate and n-heptane).

TABLE 1. THE PROPERTIES OF JATROPHA CURCAS OIL AND ALGAE OIL

S.No	Properties	JatrophaCurcas oil	Algae oil	Blend of JatrophaCurcas oil and algae oil
1	Flash point (°C)	272	102	-
2	FFA (%)	22%	0.5%	1%
3	Density(kg /m3)	0.924	0.86	-
4	Viscosity(cst) @40 °C	34	21	-

TABLE 2. INDEPENDENT VARIABLES USED FOR BOX-BEHNKEN IN TRANSESTERIFICATION OF BLEND OIL.

Variables	Symbol	Unit	Levels		
			-1	*0*	*1*
Reaction Time	A	Minutes	60	120	180
Catalyst amount	B	%	00	01	02
Methanol/oil Ratio	C	%	20	40	60

IV. RESULTS AND DISCUSSION

4.1. Transesterification Process

The Experimental and predicted values for blend (Jatrophacurcas oil + algae oil) yield responses at the design point and all the three variables in uncoded form are given in Table 3.

TABLE 3. RESPONSES FOR TRANSESTERIFICATION OF BLEND (JATROPHA CURCAS OIL + ALGAE OIL).

Run	A: Reaction time	B: Catalyst amount	C: Methanol/ oil ratio	Blend biodiesel yield (%)	
				Experimental	*Predicted*
1	60	0	40	42.5	45.6
2	60	1	20	68.83	70.14
3	120	1	40	46	45.71
4	120	1	40	44.8	45.71
5	120	0	20	2.5	0
6	180	0	40	23.44	18.85
7	60	2	40	3.9	7.54

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8	180	1	20	20.42	17.85
9	180	2	40	5.21	7.21
10	120	0	60	43.79	51.02
11	60	1	60	50.39	54.41
12	120	2	60	24.58	27.98
13	120	1	40	76.12	71.38
14	120	2	20	8.69	12.07
15	180	1	60	88.62	91.42
16	120	1	40	14.74	9.67
17	120	1	40	43.91	51.49

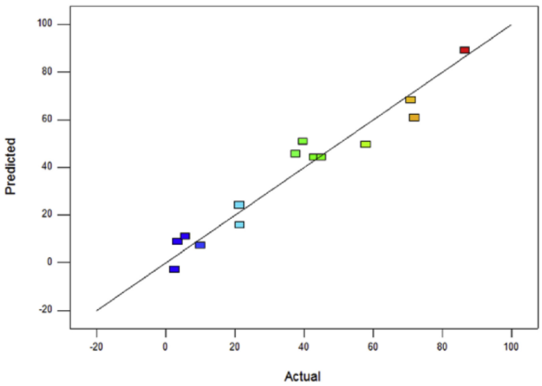

Fig. 1: Predicted versus actual JatrophaCurcas oil (%) yield values.

TABLE 4. ANOVA OF EXPERIMENTAL RESULTS.

S. No.	Parameters	Values
1	Model	Significant
2	Lack of Fit	Non-Significant
3	R2	0.92
4	Adjusted R2	0.84

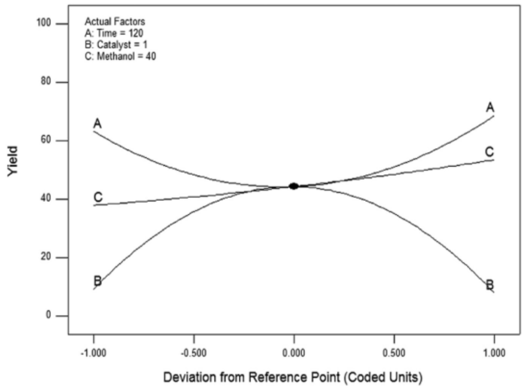

Fig. 2: Effect of catalyst concentration, time and methanol/oil ratio on blend (%) yield.

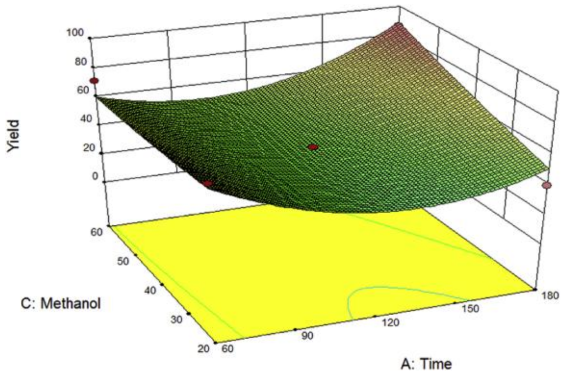

Fig. 3: Biodiesel yield (%) versus methanol to oil ratio (%) and reaction time (min)

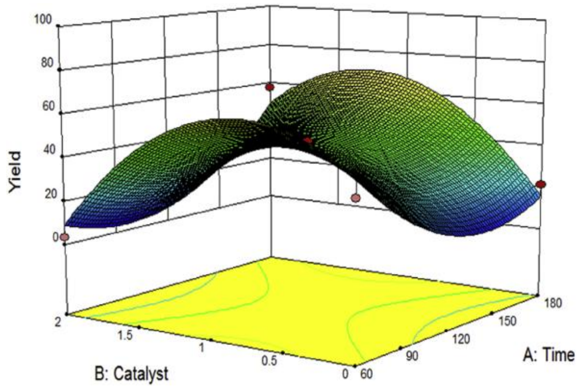

Fig. 4: Biodiesel yield (%) versus catalyst amount (%) and reaction time (min).

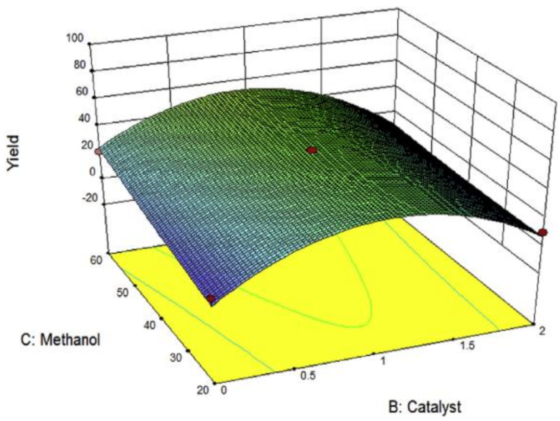

Fig. 5: Biodiesel yield (%) versus catalyst amount (%) and ratio of methanol to oil (%).

TABLE 5. OPTIMIZATION CRITERIA FOR MAXIMUM BIODIESEL YIELD

Parameter	Goal	Optimized value
Reaction Time	In range	179
Methanol Volumetric ratio	In range	58
Catalyst amount	In range	1.0
Biodiesel yield	Maximize	92

TABLE 6. RESULTS OF MODEL VALIDATION AT THE OPTIMUM CONDITION.

Number	Methanol/oil volumetric ratio	Reaction temperature (°C)	Catalyst loading (wt %)	Reaction time (min.)	Predicted biodiesel yield (%)	Experimental biodiesel yield (%)	% Error
1	60	50	1.0	180	92.64	87.59	5.05

TABLE 7. PHYSICOCHEMICAL PROPERTIES OF BIODIESEL.

S. No.	Property (Unit)	IS15607	IS 15607 limit	Biodiesel
1	Flash point (°C)	IS1448	-	115
2	Viscosity at 40°C (cSt)	IS1448	-	4.1
3	Water and sediment (vol%)	D2709	Max 0.05%	0.05

V. OPTIMIZATION OF RESPONSE PARAMETERS

The optimization of individual response was performed to achieve the maximization of blend yield based on the respective developed mathematical equations. The optimal value of inputprocess parameters is given in Table 5. For validation purpose the experiments were performed on optimized values of process parameters and it is found that predicted response is in good agreement with the experimental results with an error of 5.05% as given in Table 6. Blend biodiesel yield of 87.59% was achieved with methanol/oil volumetric ratio (3:5) using H_2SO_4 as catalyst (0.9% w/w) in 180 min at the temperature of 50°C. The biodiesel samples were tested for physicochemical properties as per ASTM D-6751 and Indian IS 15607 specification and the properties are given inTable 7.

VI. CONCLUSION

The present study deals with the optimization of biodiesel production from blend of algae and Jatrophacurcas oil using low temperature transesterification process. Three process variables viz. catalyst (H_2SO_4) concentration (0-2% w/w), reaction time (60-180 min) and methanol/oil ratio (v/v) (20-60%) for the transesterification process were optimized to get maximum yield using Box-Behnken Design with the help of Design Expert 11.0 software.
A biodiesel yield of 87.59% was achieved with methanol/oil volumetric ratio (3:5) using H_2SO_4 as catalyst (0.9% w/w) in 180 min time at 50°C temperature. It was observed that catalyst concentration, reaction time and methanol/oil volumetric ratiohad a significant effect on blend yield.It is found out that this model can further be tested at industrial level to use in the industry to improve the efficiencyefficiency of biodiesel production from blend of Jatropha and algae oil thereby, saving time and cost of the process in optimizing process parameter.

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