Optimization of Process Parameters for Biodiesel Production from Waste Cooking Oil using DOE Techniques

Sunil S, Shrishail Kakkeri

Abstract*: *In the current study the process parameters for used cooking oil biodiesel production are optimized by using Design of Experiments (Taguchi technique) and validation experiments are carried out at the optimized parameters to cross verify the results obtained. A specially designed equipment which can maintain the exact conditions required for optimization is used. This equipment is microcontroller operated and does not require any human intervention to maintain the accurate temperature, time and other parameters. By Taguchi strategy best yield acquired is by Molar ratio of A_2 (1:9), Catalyst concentration of B_3 (1.00%), Reaction temperature of C_2 (60ºC), Reaction time of D3 (120 min). The approval trial was completed for the results and the yield is observed to be 93.25%, which resulted in improvement of up to 15% yield as compared to that of crude method. The properties of acquired biodiesel are found out and it very well may be inferred that the properties of got biodiesel are within ASTM norms.

Keywords* : *Biodiesel, Transesterification, Waste Cooking oil (WCO), Optimization, Taguchi Technique.

I. INTRODUCTION

The world stores of primary energy materials are limited. As per the reports of International Energy Agency, worldwide oil demand of 106.4 million barrels every day in 2040, up from 96.9 million of every 2019 [1]. The gigantic development of total population, expanded technical advancement and way of life in the developed countries has prompted this energy demand. The existing fuel reservoirs are depleting day by day. This requires creating and commercializing petroleum product substitutes from bio-based sources. This likely could be the major cause of developing awareness for unconventional bioenergy sources for fuels. Squander items like utilized cooking oil, spent coffee powder and so on., have numerous disposal issues like water and soil contamination, human health concern [2]. Also reusing the cooking oil multiple times increases the FFA of the oil which causes harmful health effects. Instead of disposing or reusing it, it tends to be utilized as feedstock for biodiesel production. Thusly transformation of low-quality lipid-rich sources in to business grade biodiesel is an ideal system for limiting ecological damage while it can help meeting the vitality challenge.

According to the survey India is perhaps the most noteworthy consumer of edible oil and in the year 2017, 22.75 million tons of edible oil was expended [3]. When the oil is warmed past its smoke point, it gets unfit for human consumption. Further on the off chance that it is reused it prompts a few medical problems. In the event that the oil which is utilized once for cooking is collected and changed over to Biodiesel, it can give biodiesel yield up to 80% (Roughly 17 million tons according to the information given above), which turns into a significant source of fuel.

Biodiesel can be produced by the standard transesterification process, in which vegetable oil is responded with alcohol in the presence of an impetus [4]. The key parameters affecting the transesterification process are alcohol to oil molar ratio, reaction time, catalyst concentration and reaction temperature [5]. In order to obtain highest yield these key parameters are to be optimized and from the literature survey, it is understood that the yield can be improved by 10 to 12% by optimizing these parameters which makes the process more economical [6] [7]. In the current study a specially designed equipment which can maintain the exact conditions required for optimization is used. This equipment is microcontroller operated and does not require any human intervention to maintain the accurate temperature, time and other parameters. The process parameters for used cooking oil biodiesel production are optimized by using Design of experiments (Taguchi technique) and validation experiments are carried out at the optimized parameters to cross verify the results obtained.

II. MATERIALS AND METHODS

A. Materials

Used cooking oil is collected from restaurants near International Airport, Bengaluru. The collected oil is filtered to remove the unwanted particles left in the oil. All the chemicals methanol, sulphuric acid and sodium hydroxide used were of analytical reagent grade and bought locally.

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B. Experimental Set up

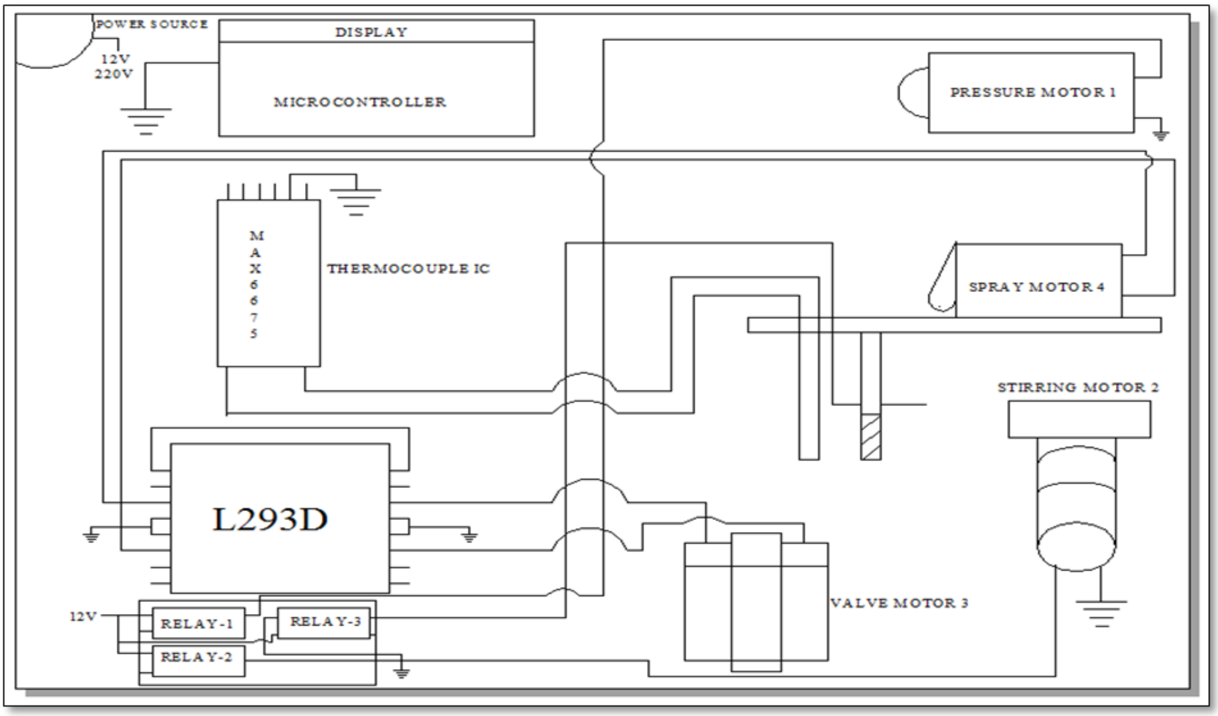


Fig 1: Circuit diagram of Reactor used

The circuit diagram of Microcontroller based Transesterification test rig used in the experimentation is shown in the figure 1. The test rig comprises of a reactor tank, which is utilized for transesterification reaction dependent on Free Fatty Acid (FFA) content. The reactor is tall and restricted with a tapered base to ensure gravity separation. The reactor comprises of Inlet, Outlet, Connections to condenser and a blending system. This reactor is a fully automated mechanism wherein accurate experimental conditions are maintained to carry out the experiments.

C. Transesterification

Oils are made out of triglycerides and free unsaturated fats, FFA assumes a fundamental job during the generation of biodiesel. The FFA over 1% diminishes the yield of the biodiesel [9]. In case of waste cooking oil, the FFA increases when the oil is reused a greater number of times. The FFA of oil collected was found to be in the range of 4 to 5%. The FFA of the oil is reduced by esterification; it decreases the FFA substance in the oil within the sight of concentrated hydrochloric acid as an impetus and methyl alcohol as a reactant.

The product of esterification is fed into the reactor and mixed methoxide is added to the reactor. The reactor is set for temperature and time. The temperature is maintained constantly by the rector. After the fulfilment of pre-set time, reactor power supply is cut of automatically so that isolating of glycerol begins. After 20 mins of isolating an alert is given by the equipment, so that the operator can isolate the triglycerides and glycerol, the isolated Triglycerides are fed back to the reactor for methanol recovery.

D. Design of Experiments (Taguchi Technique)

The design of experiments is an efficient technique to decide the association between factors affecting a process and the yield of that process. It is utilized to find cause and effect connections. This data is expected to coordinate input parameters to optimize the output.

To improve the yield of biodiesel Taguchi technique is utilized to optimize the response parameters like methanol to oil proportion, Catalyst concentration, Reaction temperature and Reaction time. Based on orthogonal array this procedure is completed and it gives a lot of diminished variance for the tests with ideal setting of controlled parameters. A set of well-balanced tests and signals to noise ratios (SNR) are acquired by orthogonal array, which are log elements of wanted yield.

There are three SNR for optimization of static problems.

i) Smaller the better

   $n = -10 \log_{10}$ [mean of sum of squares of measured data]

   This is generally the picked SNR for every single unwanted characteristic like imperfections for which the best value is zero.

ii) Larger the better

   n = - 10 log10 [mean of sum of squares of reciprocal of measured data]

   This is suitable when the output of the process is to be maximized.

iii) Nominal the best

   n = 10 log10 [square of mean / variance]

   This case is suitable when the output and imperfections are to be balanced.

In the current work larger the better case is chosen as the yield is to be maximized.

Four predominant factors affecting the yield at three different levels are chosen based on the preliminary experimentation. The selected parameters and levels are shown in Table 1. Table 2 shows the Taguchi L9 orthogonal array used for the experiment purpose.

Four predominant factors affecting the yield at three different levels are chosen based on the preliminary experimentation. The selected parameters and levels are shown in Table 1. Table 2 shows the Taguchi L9 orthogonal array used for the experiment purpose.

Parameters/ Levels	Symbol	1	2	3
Molar ratio	A	1:6	1:9	1:12
Catalyst concentration (%)	B	0.50	0.75	1.00
Reaction temperature (°C)	C	45	60	75
Reaction time (min)	D	40	80	120

Table 1: Selected parameters and their levels

EXPT. NO	PARAMETERS			
	A	B	C	D
E1	1	1	1	1
E2	1	2	2	2
E3	1	3	3	3
E4	2	1	2	3
E5	2	2	3	1
E6	2	3	1	2
E7	3	1	3	2
E8	3	2	1	3
E9	3	3	2	1

Table 2: L9 Orthogonal Array

III. RESULTS

A. Analysis of Signal to Noise Ratio (SNR)

Expt No.	PROCESS CONTROL FACTORS				S/N Ratio	Yield (%)
	Molar Ratio	Catalyst Conc.	Reaction Temperature	Reaction Time		
1	6	0.50	45	40	36.5215	67
2	6	0.75	60	80	37.0252	71
3	6	1.00	75	120	37.6163	76
4	9	0.50	60	120	37.3846	74
5	9	0.75	75	40	38.8897	88
6	9	1.00	45	80	39.2758	92
7	12	0.50	75	80	38.1697	81
8	12	0.75	45	120	38.3816	83
9	12	1.00	60	40	38.7904	87

Table 3: SNR and Yield

The SNR for different set of experiments are calculated and tabulated in Table 3. Results show that experiment number 6 (Molar Ratio 1:9, Catalyst concentration 1%, Reaction Temperature 45^{0}C and Reaction time of 80 minutes) gave highest yield of 92%, whereas experiment number 1 (Molar Ratio 1:6, Catalyst concentration 0.5 %, Reaction temperature 45^{0}C and Reaction time of 40 minutes) gave lowest yield of 67%. Even though in the manual experiments trial 6 gives the highest efficiency, this may not be the optimized value. The mean SNR for each parameter is calculated and tabulated in table 4. The Main effects plot for mean of SNR is plotted in fig 2. SNR for the parameter Molar ratio is calculated at all the three different levels and it is found to be 37.5, 38.52 and 38.45 at level 1, 2 and 3 (1:6, 1:9 and 1:12) respectively. This shows that highest yield is obtained at level 2 i.e., 1:9. Similarly the SNR for all parameters are calculated and shown in table 4. From the fig 2 it can be seen that highest yield is obtained at Molar ratio of 1:9, Catalyst concentration 1 %, Reaction temperature 75^{0}C and Reaction time of 80 minutes.

The value of delta is calculated as the difference between highest and lowest SNR for any parameter and based on delta the ranking is given for different parameters. It can be concluded that Molar ratio has the highest effect on yield whereas reaction time has relatively lowest effect. Validation experiment was carried out at the optimum conditions and the yield was found to be 94.6%.

Level	Molar Ratio	Catalyst Conc.	Reaction Temperature	Reaction Time
1	37.5	37.36	38.06	38.07
2	38.52	38.10	37.73	38.16
3	38.45	38.56	38.23	37.79
Delta	1.46	1.20	0.49	0.36
Rank	1	2	3	4

Table 4: Response Table for SNR

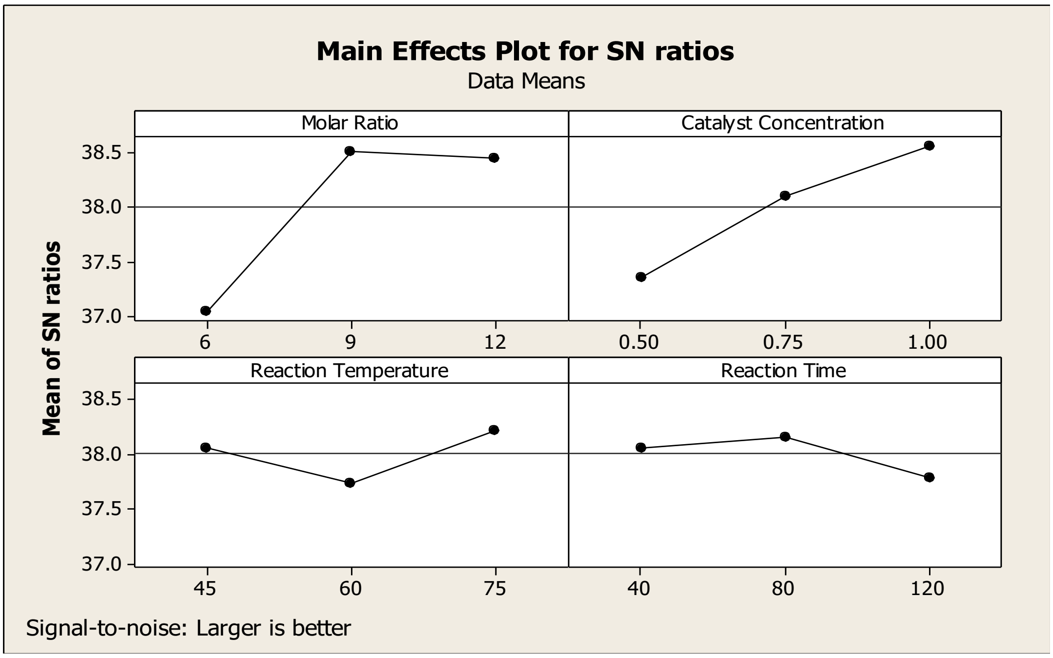
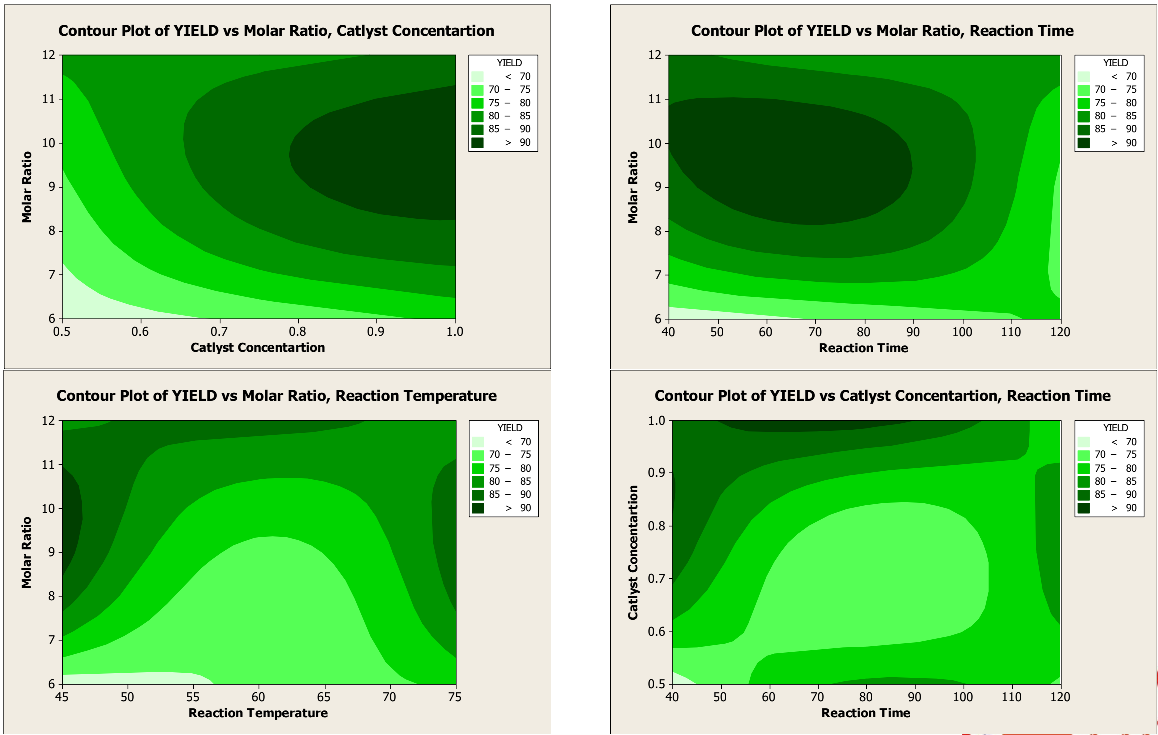


Fig 2: Main Effects plot for SNR

B. Effect of individual parameters on yield

Contour plots of the four key parameters affecting the yield are shown below (Fig 3)



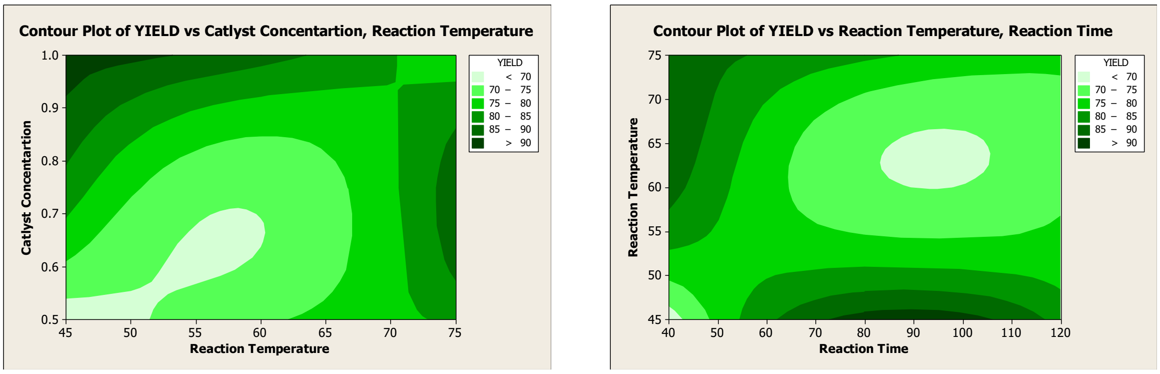


Fig 3: Contour plots for conversion of waste cooking oil to biodiesel.

C. Molar Ratio

Molar ratio of alcohol to oil is an important parameter in the transesterification process. In the earlier discussion of effect of SNR, it is concluded that molar ratio is the top priority factor which affects the yield. As per the stoichiometric calculations three moles of alcohol is required for every mole of oil to produce three moles of fatty acid ester and one mole of glycerol [8]. However, transesterification is a reversible reaction and excess alcohol is required to drive the reaction in the forward direction. In the current study, it can be observed from contour plot as well as main effects plot for SNR that 1:9 molar ratio gives minimum yield, further increasing the molar ratio has no significant effect.

D. Catalyst Concentration

Transesterification reaction is a very slow process, in order to speed up the process catalyst is used. In the current work sodium hydroxide is used as catalyst. Lower amount of catalyst leads to a slower rate of reaction whereas excess catalyst leads to soap formation which reduces the yield [10]. From the preliminary experiments it was observed that catalyst concentration above 1% w/v lead to excess soap formation and less than 0.5 % w/v lead to very low rate of reaction. In the current study, it can be observed from contour plot as well as main effects plot for SNR that 1% w/v catalyst concentration gave the highest yield.

E. Reaction Temperature

It very well may be contended that, the yield of biodiesel increments with response time. At the start, the response is delayed because of the blending and scattering of alcohol into the oil. Then the reaction progresses steadily [10]. it can be observed from contour plot as well as main effects plot for SNR that initially with respect to the reaction time the yield increased up to 80 minutes and beyond 80 minutes the yield curve had a negative slope. This may be due to the reverse rection that occurs beyond some time limit.

F. Reaction Time

Increased temperature will result in an increasing fraction of molecules which results in higher chemical kinetics and in turn higher yield [12]. It can be observed that highest yield is obtained at 75^0C. From the preliminary experiments it was observed that increasing temperature beyond 75^0C lead to decreased yield, this may be due to the higher evaporation rate of alcohol used and also at higher temperatures soap formation is higher. [11][12]

G. Fuel Properties

Various properties of waste cooking oil biodiesel were determined by the ASTM standards and are presented in Table 5. It will in general be seen from this table the fuel properties of biodiesel are practically comparable to that of Diesel and are well inside the ASTM benchmarks for biodiesel.

Table 5: Fuel Properties

Properties	Standard	Range	Diesel	WCO Biodiesel
Flash point (°C)	ASTM D93	>130 *	58	162
Kinematic Viscosity (Cst) at 40°C	ASTM D445	1.9-6.0	1.764	4.8
Specific gravity	ASTM D4052	0.87-0.90	0.82	0.87

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Calorific value (MJ/kg)	ASTM D240	--	43	37.85
Cloud point, °C	IS:1448 (P10)	-3 to 12	-9	10
Ash, %w/w	IS:1448 (P 4)	0.5max	NIL	NIL
Carbon residue, Rams bottom, %w/w	IS:1448 (P 8)	0.05max	NIL	0.012
Pour point, °C	IS:1448 (P10)	-15 to 10	-12	7

IV. CONCLUSIONS

In the current study the factors affecting waste cooking oil biodiesel production was optimized using Taguchi technique using a microcontroller-based reactor.

Outcomes of the work are as follows:

- The properties of made biodiesel blends were well within ASTM standards.
- By Taguchi strategy best yield acquired is by Molar ratio of A_2 (1:9), Catalyst concentration of B_3 (1.00%), Reaction temperature of C_2 (60ºC), Reaction time of D_3 (120 min). The approval trial was completed for the results and the yield is observed to be 93.25%, which resulted in improvement of up to 15% yield as compared to that of crude method.

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Sunil S: Currently working as Assistant Professor at Department of Mechanical Engineering, Sri Venkateshwara College of Engineering, Bengaluru. Taken the lead of setting up Biofuel Research and Development unit at Sri Venkateshwara College of Engineering. Worked in the field of alternate fuels for I C engines, LHR Engines. Worked on various oils like Spent Coffee Powder oil, Simarouba, Hongae, Jatropha, Neem, Published 10 Technical Papers in various National / International Conferences / Seminars / Journals. Responsible for preparing & getting sanctioned projects from KSCST and KSBDB.

- Obtained 7,20,000/- fund from Kuwait oil Company for waste to energy conversion project.
- Obtained best paper award for Paper titled "Production & Characterization of Biodiesel from Neem oil & Optimization of Reaction Parameters using Taguchi Method" in National Conference on Innovative trends in Mechanical engineering, SVCE, Bengaluru, April 21-22, 2016
- Obtained best poster award for paper titled "CHARECTERISATION OF BIODIESEL FROM WATERMELON SEED OIL AND OPTIMIZATION OF REACTION PARAMETERS FOR MAXIMISING THE YIELD" at 10th Science and Technology Academy Conference organized by KSTA in association with REVA University, 18th -19th Jan 2018, REVA University, Bengaluru.

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