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# Optimization of Biodiesel (Rubber Seed Oil) Process Parameters using Taguchi Method Based on Experimental Procedure in A Batch Stirred Tank Reactor

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***Abstract:** Due to the increase in the demand for an alternate fuel the research studies towards searching for a low cost source and optimum conditions with new reactors were gaining momentum. Hence the present investigation is focused on the design of a batch stirred tank reactor with two different impellers to produce biodiesel with the use of rubber seed oil. Further an experiment was conducted in optimizing the process parameters using Taguchi methodology. The Taguchi methodology is mainly focused on the enhancement of the product produced and this is the most precise method used. The best combinations were obtained through the L9 orthogonal array. The reactor specifications were fixed to be 7.5cm. height, 7.5cm diameter, 2.5 cm agitator diameter, 0.625cm baffle thickness with 2.5cm as distance between bottom of the tank and impeller. Two impellers with 0.5cm. Width, 0.625cm length and others with 1.1cm length were used to test the individual performance towards yield. The optimum conditions obtained were found to be Impeller type-Straight blade impeller, Temperature- 65ºC, Time - 45min, Reactant ratio - 1:6, Catalyst amount- 22.46% (mol% of Oil), and Impeller Speed- 500rpm with the yield of 88 % . Hence BSTR reactors could be scaled up for translational research studies too.*

***Keywords:** Baffle, Batch Stirred Tank Reactor, Biodiesel, Impeller.*

## I. INTRODUCTION

Biodiesel is an alternative energy source having substantial role in environment considering its Eco friendliness and is capable of restoring the world energy demand. However, at the same time, it also achieves environmental sustainability. Biodiesel is significant as it does not require any other special equipment and which can also be utilized in all forms of diesel engines when compared with other alternative sources. Overall, biodiesel is harmless and renewable; therefore, its production is increasing fast, notably in countries like Europe, United States, and Asia [1][2].

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Through transesterification process vegetable oil can also be subjected in producing biodiesel. Biodiesel can be produced by using plant or crop materials such as Jatropha curcas, Pungamia pinnata, soya bean, and sunflower as the major feedstocks. The extravagant edible oil cost constraints their use in biodiesel preparation, whereas, non-edible oils are mostly preferred for biodiesel production. It is also approximated that India has the capability to produce one million ton of biodiesel per year. Over 200 districts in India are inaugurated for biofuel project by the Government of India's planning commission. Jatropha (Jatrophacurcas) and Karanja (Pungamia pinnata), were considered efficient enough by the commission for the biodiesel production [3][4][5][6][7][8][9]. Moreover, the policy of auto fuel document clearly affirms that it is the most efficient, eco-friendly, and renewable alternate to gasoline fuels [10] Transesterification is a feasible method widely used for biodiesel production from lipids that react chemically like the animal fat and vegetable oils (associated with alcohol-producing fatty acid esters). Trading the alkoxy group of an ester compound with another form of alcohol are all the methods of transesterification. Alkoxy groups in general are the combination of alkyl (C and H) group linked with oxygen R–O. An acid or base act as catalyst for these reactions. The rate of reactivity is enhanced when a proton is donated from acids to the carbonyl group. On the other hand, bases catalyse the reaction of the compound by which the alcohols are more reactive as a proton is removed from it. By the way, the optimization of biodiesel production parameters is mandatory to achieve maximum yield. The consolidation of the transesterification process and for the production of biodiesel from Jatropha–alga oil [11] Response Surface Methodology (RSM) technique was performed. The Mahua and Karanja which are unpalatable food has served the purpose of bringing out the biodiesel with 50:50 by v/v mixing of both. Alkaline transesterification and acid esterification with a double-pass reaction was carried out where the former of free fatty acid (FFA) quantity was decreased to a necessitate limit and the latter converted the oils to fatty acids of methyl esters. By making use of catalyst the sulphuric acid was taken for the process to take place. Transesterification needs the mixture of KOH and methanol. Besides being cheap, methanol with the functional OH group in it depletes the reaction time. To extract biodiesel from jatropha both the non-catalytic and catalytic techniques were utilized.

The production of biodiesel could be considered perfect if the content of free fatty acid (FFA) of the Jatropha oil for an alkaline catalyst was less than 1% and a two-step transesterification process was greater than 1%.

Biodiesel was obtained employing transesterification technique for Jatropha Curcas using a single-pass alkaline catalyst and it was inspected [12]. To optimize the variables like reactant proportion, reaction temperature and methods of purification, Taguchi's methodology was preferred. Gemma Vicente [14] studied biodiesel production from sunflower oil and came up with an optimization procedure. They described that purity of biodiesel was regulated by the catalyst concentration [13]. They also reported the most favourable values of catalyst concentrations, reaction temperature and molar ratio. These working conditions forecasted maximum values of biodiesel purity (100% weight), maximum yield (98.4% weight), and its profitability. Gemma Vicente [14] precisely studied the material balance of the fatty acid methyl esters synthesis from sunflower oil utilizing potassium hydroxide (KOH) as a catalyst. In their research, they examined the effect of operating conditions of material balance by utilizing a central composite design process. The Rubber seed oil-based biodiesel offers an assuring substitute to diesel. The acid value was found to be high for Rubber Seed oil.

## II. MATERIALS AND METHODS

### A. Materials and Chemicals

Firstly, Rubber seed oil was obtained from nearby dealers.

These dealers are globally involved in producing the required raw materials in large quantities. 99% pure chemicals of methanol and KOH are of analytical grade reagents which were utilized. As base catalyst potassium hydroxide was used in granule shapes.

### B. Design of Reactor and Impellers

A batch stirred tank reactor was designed and fabricated for the production of biodiesel and the byproduct glycerol. The following standard protocol was followed for the design.

### C. Design of Reactor (5)

$$\frac{D_a}{D_t} = \frac{1}{3};\ \frac{H}{D_t} = 1;\ \frac{J}{D_t} = \frac{1}{12}$$

$$\frac{E}{D_a} = \frac{1}{3};\ \frac{W}{D_t} = \frac{1}{5};\ \frac{L}{D_a} = \frac{1}{4}$$

Where,

J - Baffles thickness, E - Distance between Tank bottom & Stirrer, Da - Diameter of Impeller (Agitator), Dt - Diameter ofthe Tank, H - Height of the Tank, L - Length of the Blade, W - Width of the Blade.

### D. Procedure for calculating the total volume of reactant:

Amount of oil taken = 238gm
Specific gravity of oil = 0.898
Volume of oil = 238/0.898
= 265ml

Volume of Methanol needed:
Molecular weight of oil ≈ 900 kg/kmole
Molecular weight of Methanol = 32 kg/kmole

Maximum moles ratio between oil and Methanol = 1:6
Moles of Oil taken = 238/900
= 0.264 moles
6 moles of methanol = (6x0.264)
=1.587moles

Therefore, amount of methanol required
= moles x weight
=1.587 x 32
= 50.77 gm.

Volume of methanol ($CH_3OH$) taken
V = (specific gravity x amount of $CH_3OH$)

Specific gravity of methanol = 0.791
Volume of methanol needed = 50.77/0.791
V = 64.189ml

Total volume reactant taken
= (265ml Oil + 64.189ml $CH_3OH$)
= 330ml.

From above reactor design ratio, the height of the reactor (H) and the H and diameter of the reactor ($D_t$) is same. Because ratio between H and $D_t$ is unity.

So volume of reactor = $\frac{\pi}{4} \times D_t^2 \times H$

[From design ratio: $(H/D_t) = 1$

$= \frac{\pi}{4} \times D_t^3$

= 330ml

$D_t = \{(330 \times 4)/\Pi\}^{(1/3)}$
=7.5cm

### E. Rushton Impeller Design

The Rushton Impeller is one which enable to create good turbulence and suitable for liquid-liquid agitation. It consists of 4-8 blades. A four blade Rushton Impeller was used for the process of Biodiesel production.

### F. Straight blade Impeller Design

Another type of impeller namely Straight blade Impeller was also used to compare the efficiency of the impeller. The design dimensions were been calculated using the following procedure.

Width of the impeller blade (W) = (1/2) x $D_a$
= (1/5) x 2.5
= 0.5cm

Length of the impeller blade (L) = (1/2) x ($D_a$ - $D_s$)
(Where $D_s$ – Shaft diameter)
= (1/2) x (2.5-0.3)
= 1.1cm

No. of blades = 4

### G. Rubber Seeds:

The Rubber seeds are produced by nearby dealers. Some amounts of the seed were bought from the dealers during their maturation period. These Rubber seeds were completely cleaned and scrubbed to remove unwanted substances and were kept at 4°C up to the time of the seeds being de-shelled first and after that they were dried for 4 hours at 60°C. The dried seeds were squashed finely and after that they were brought to oven and kept overnight at 45°C. Furthermore, 100g of the seeds that were ground were used for an extraction process.

It was kept for 4 hours at 60°C and as a solvent, 250 ml of n-hexane was utilized in this process. After the extraction process, the oil solvent mixture was completely put through the evaporation process which was performed using vacuum Rotavapor (R-200) at a temperature of 60°C. The evaporation process was performed to recover the extracted oil as the solvent evaporates.

### H. Biodiesel Production and Process Optimization:

The parameters that were used for optimization includes the temperature of the reaction, the catalyst used, the proportion of alcohol and oil, Impeller speed, Types of Impeller and Time of the reaction were the parameters used for the optimization. The temperature of the reactions firmly influences the reaction rate. In spite of that the reaction is held near to the boiling point of the methanol (60-70.8C) at atmospheric pressure for the required time. Pre esterification is done in order to remove the free fatty acids from the oil for these mild reaction conditions. The molar ratio of alcohol and oil is into important consideration. 3 moles of fatty acidsand 1 mol of glycerol are obtained by the Trans esterification reaction of 3 mol of alcohol per mole triglyceride. of glycerol. If the reaction has to be shifted to the right, it is essential to separate one of the products from the reaction mixture or utilize the excess alcohol. For the reaction to progress completion a second option is needed. In industrial process, a molar ratio of about 6:1 is usually required to acquire methyl ester yields more than 98% (w/w). Taguchi methodology is the recent statistical tool applied to perform the process of optimization. Taguchi's design of experiment using especially constructed tables known as "orthogonal arrays" was used. Standard analysis approach was selected. The Batch Stirred Tank Reactor BSTR is first topped up with an ideal amount of oil, and after that it is been positioned with stirrer. Once it attains the oil phase at the specific temperature, the reaction vessel is filled with methanol and the catalyst. In a report, the eggshells' Calcium oxide (CaO) has been utilized as a heterogeneous catalyst for the creation of biodiesel from extremely viscous rubber seed oil [15].

The Trans esterification response was completed under the state of the required reaction of time 25-55 minutes, Reaction ratio 1:3 to1:6, catalyst amount 0.75% (w/v) to 1.5%, Impeller speed 300 to 900 rpm temperature (35−55°C, amount of oil (2%), time for the reaction to complete (60−180 min), and temperature at which the reaction take place (35−55 °C). The mixture is separated from glycerol using a separating funnel in a day. As soon as the glycerol is removed, the methyl ester layer is been washed three times, with twice the volumes of hot distilled water that is used in the elimination of the catalyst and the glycerol deposits. Table- I shows the factors with the level used and the experiments were performed according to the inner orthogonal array derived using Taguchi's analysis software in Table- II.

**Table- I: Control Factors**

| Factors | Level- 1 | Level- 2 | Level- 3 | Level- 4 |
|---|---|---|---|---|
| **Types of impellers** | I1 | I2 | - | - |
| **Temperature** | 55 | 60 | 65 | 70 |
| **Time** | 25 | 35 | 45 | 55 |
| **Reaction ratio** | 1:03 | 1:04 | 1:05 | 1:06 |
| **Catalyst amount** | 0.75% | 1% | 1.25% | 1.50% |
| **Impeller speed** | 300 | 500 | 700 | 900 |

**Table- II: Inner Orthogonal Array**

| Trial No. | Impeller Type | Temperature | Time | Reactant ratio | Catalyst amount | Impeller speed |
|---|---|---|---|---|---|---|
| **1** | 1 | 1 | 1 | 1 | 1 | 1 |
| **2** | 1 | 1 | 2 | 2 | 2 | 2 |
| **3** | 1 | 1 | 3 | 3 | 3 | 3 |
| **4** | 1 | 1 | 4 | 4 | 4 | 4 |
| **5** | 1 | 2 | 1 | 1 | 2 | 2 |
| **6** | 1 | 2 | 2 | 2 | 1 | 1 |
| **7** | 1 | 2 | 3 | 3 | 4 | 4 |
| **8** | 1 | 2 | 4 | 4 | 3 | 3 |
| **9** | 1 | 3 | 1 | 2 | 3 | 4 |
| **10** | 1 | 3 | 2 | 1 | 4 | 3 |
| **11** | 1 | 3 | 3 | 4 | 1 | 2 |
| **12** | 1 | 3 | 4 | 3 | 2 | 1 |
| **13** | 1 | 4 | 1 | 2 | 4 | 3 |
| **14** | 1 | 4 | 2 | 1 | 3 | 4 |
| **15** | 1 | 4 | 3 | 4 | 2 | 1 |
| **16** | 1 | 4 | 4 | 3 | 1 | 2 |
| **17** | 2 | 1 | 1 | 4 | 1 | 4 |
| **18** | 2 | 1 | 2 | 3 | 2 | 3 |
| **19** | 2 | 1 | 3 | 2 | 3 | 2 |
| **20** | 2 | 1 | 4 | 1 | 4 | 1 |
| **21** | 2 | 2 | 1 | 4 | 2 | 3 |
| **22** | 2 | 2 | 2 | 3 | 1 | 4 |
| **23** | 2 | 2 | 3 | 2 | 4 | 1 |
| **24** | 2 | 2 | 4 | 1 | 3 | 2 |
| **25** | 2 | 3 | 1 | 3 | 3 | 1 |
| **26** | 2 | 3 | 2 | 4 | 4 | 2 |
| **27** | 2 | 3 | 3 | 1 | 1 | 3 |
| **28** | 2 | 3 | 4 | 2 | 2 | 4 |
| **29** | 2 | 4 | 1 | 3 | 4 | 2 |
| **30** | 2 | 4 | 2 | 4 | 3 | 1 |
| **31** | 2 | 4 | 3 | 1 | 2 | 4 |
| **32** | 2 | 4 | 4 | 2 | 1 | 3 |

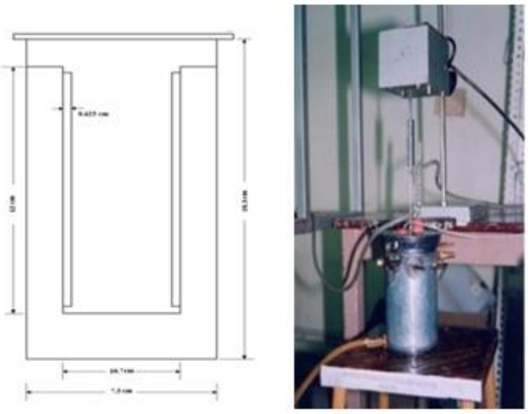
## III. RESULTS AND DISCUSSION

### A. Design of Batch Stirred Tank Reactor

A Batch Stirred Tank Reactor was designed and fabricated for the production of biodiesel Fig. 1(a) and Fig. 1(b). The reactor vessel was surrounded with a jacketed vessel used for heating the reactor and also maintaining the reaction temperature. The steam required for maintaining the temperature was supplied through a steam boiler attached to the reactor and also a cooling coil inserted into the reactor. The reactor was fitted with two baffles, a stirrer and a motor. The rpm of the impeller was monitored and controlled with a speed regulator attached to the motor. Top views of the BSTR reactor with different impeller were shown in the Fig. 2. Table- III shows the dimensions of the designed reactor. It was observed that the reactor is 7.5cm. Height, 7.5cm diameter and attached baffles will be suitable for the testing the optimization process of biodiesel production. Table- III reveals the design date of the reactor estimated using the standard design ratio.

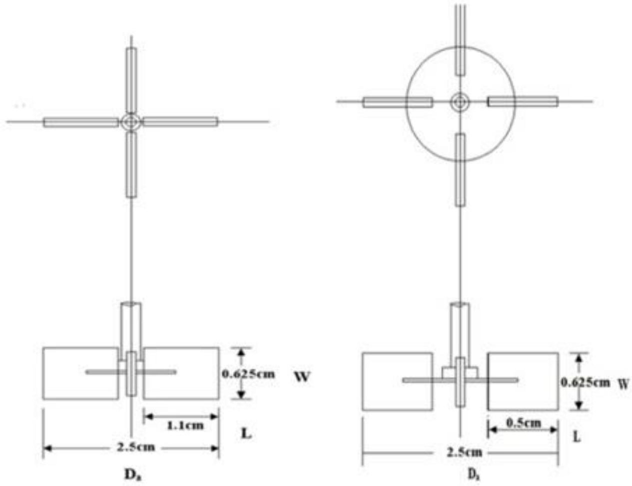
**Table- III: Design Data of Batch Stirred Tank Reactor**

| S. No. | Design of the batch stirred tank reactor vessel Design dimension of the reactor vessel | Dimensions |
|---|---|---|
| 1 | Diameter of Reactor **($D_t$)** | 7.5cm. |
| 2 | Height of the tank **(H)** | 7.5cm. |
| 3 | Original height of reactor needs some clearance from calculated height. Because the calculated height of reactor is obtain from total volume of reactant taken. | 12cm |
| 4 | Diameter of agitator **($D_a$)** | 2.5cm. |
| 5 | Baffle thickness **(J)** | 0.625cm. |
| 6 | Distance between bottom of the tank and impeller (E) | 2.5cm |
| **Rushton Impeller Design** | | |
| 7 | Width of the impeller blade **(W)** | 0.5cm. |
| 8 | Length of the impeller blade **(L)** | 0.625cm. |
| 9 | No. of blades | 04 blades |
| **Straight blade Impeller Design** | | |
| 10 | Width of the impeller blade **(W)** | 0.5cm |
| 11 | Length of the impeller blade **(L)** | 1.1cm |
| 12 | No. of blades | 04 blades |



**(a) (b)**

**Fig. 1. Batch Stirred Tank Reactor and their design for the production of biodiesel (a) – Dimensions, (b) – Fabrication view**




**(a) (b)**

**Fig. 2. Top view of batch stirred tank reactor(BSTR) with (a) - Straight blade Rushton, (b) - Impeller setup and dimensions**

### B. Process Optimization and Analysis of Variance

In order to change every independent variable and to observe ideal values for the reaction targets, Taguchi methodology is been carried out (Table- IV). Analysis of Variance was observed with high f value for the process variables and reveals the performance of the method adopted along with the optimum value. It was found to have no distinction in the mean from the study of variance (ANOVA) in the statistical overview of the experimental facts. From the above experimental analysis, apparently, more prominent the estimation of sum of square of an independent factors, the more it impacts the production variable. On enumerating the proportion of discrete sum of square of a particular independent factor to the entire sum of squares of all the factors, the percent improvement of the separate variable on the performance specification is found out. Hence the P value is high for reactant ratio which is 63.64 followed by11.69 of the catalyst amount. The F- test values are evaluated at 95% of confidence level to determine the mainfactors that affects the other factors. The result implies that the 'F' test values calculated at 95 % confidence is satisfied for classification of the impeller. The variance analysis (ANOVA) is used to estimate the speed of the stirrer based on the yield and the effects caused by KOH and methanol. Based on the outcome from the ANOVA table, it revealed that the effect of reactant ratio, catalyst amount and impeller were obtained as 63.4%, 11.69% and 52.5% respectively. Similar results were obtained when Pungamia oil was used with a proportion of methanol, KOH, and stirrer speed and the outcomes were 70.10%, 20.35%, and 5.78%, respectively [16]. In Bangladesh, biodiesel is produced by employing Rubber seed oil (RSO) as a significant source. Biodiesel has been produced by employing a three phased methodology which consists of alkaline hydrolysis of oil, acidulation of the soap and esterification of Free Fatty Acids. The overall outcome of FFA from RSO appears to be almost86% [17].

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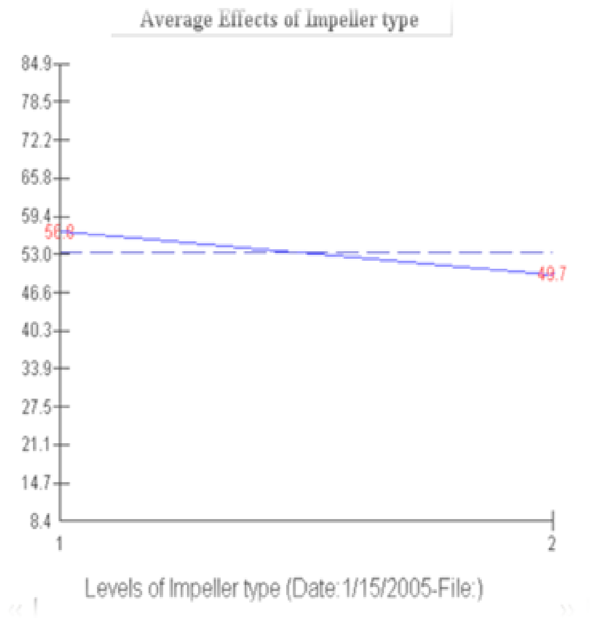
**Table- IV: Analysis of Variance (Anova)**

| Column#/Factors | DOF (f) | Sum of Sqrs. (S) | Variance (V) | f-Ratio (F) | Pure sum (S[1]) | Percent P(%) |
|---|---|---|---|---|---|---|
| **Types of impellers** | 1 | 395.078 | 395.078 | 1.995 | 197.097 | 0.793 |
| **Temperature** | 3 | 419.128 | 139.709 | 0.705 | 0 | 0 |
| **Time** | 3 | 431.258 | 143.752 | 0.726 | 0 | 0 |
| **Reactant ratio** | 3 | 1605.666 | 5468.555 | 27.621 | 15811.723 | 63.643 |
| **Catalyst amount** | 3 | 3499.519 | 1166.506 | 5.892 | 2905.576 | 11.695 |
| **Impeller speed** | 3 | 723.95 | 241.316 | 1.218 | 130.006 | 52.3 |
| **Other/Error** | 15 | 2969.719 | 197.981 | - | - | 23.35% |
| **Total** | 31 | 24844.319 | 23.346 | - | - | 100.00% |

### C. Effect of process variables

Impeller I showed a prominent result with 56.0 as level of index and the second impeller with 49.7 Fig. 3(a). Hence the Rushton turbine impeller was found to be better than the straight blade impeller due to the flow pattern prevailed with optimum speed inside the reactor. The effect of temperature with the level of index Fig. 3(b) reveals that maximum effect of 61.9 was observed in the level 3 which was 65 temperatures. Fig. 3 (c). the time reaction with the measure of index shows that maximum effect was observed in the level 3 with 65.2 followed by level 2with 52.9, level 1 with 49.2, level 4 with 51.0. Hence the optimum time required for the best Trans esterification was found to be 45 minutes. Fig. 3 (d). The effect of reaction ratio of the level of index shows that maximum effect was observed in the level 4 with74.0 followed by level 3 with 73.6, level 2 with 48.2, level 1 with 19.3. Hence the optimum reactant ratio required for thebest Trans esterification was found to be 1:6 of level 4. Fig. 3 (e). the effect of catalyst amount with the level of index shows that maximum effect was observed in the level 2 with68.0followed by level 3with 59.3, level 1 with 50.2, level 4 with 37.0. Hence the optimum amount of catalyst required for the best Trans esterification was found to be 22.46% (w/v) of level 2. Fig. 3 (f). the effect of impeller speed with the level of index shows that maximum effect was observed for level 4 with 59.4 followed by level 1 with 56.3, level 2 with 55.2 and level 3 with 45.0 and maximum was observed for level 4 with optimum speed of 500 rpm. There was a rise in the level of the rate of conversion of methanol/oil having the molar ratio that ranges from 2:1 to 4:1. Later the rate of conversion decreased where the ratio that ranges from 5:1 to 6:1 was noted. The finest molar ratio of methanol/oil was detected at4:1[18].



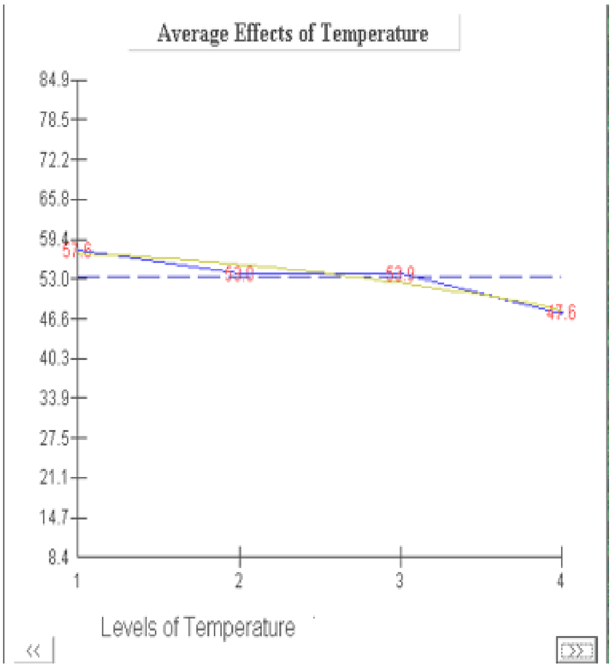




**Fig. 3(a). Average effects of Impeller type Fig. 3(b). Average effects of Temperature**

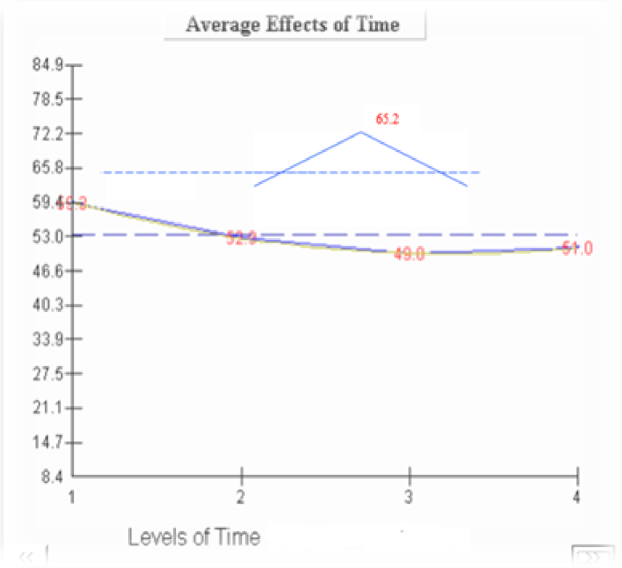



**Fig. 3(c). Average effects of Time**

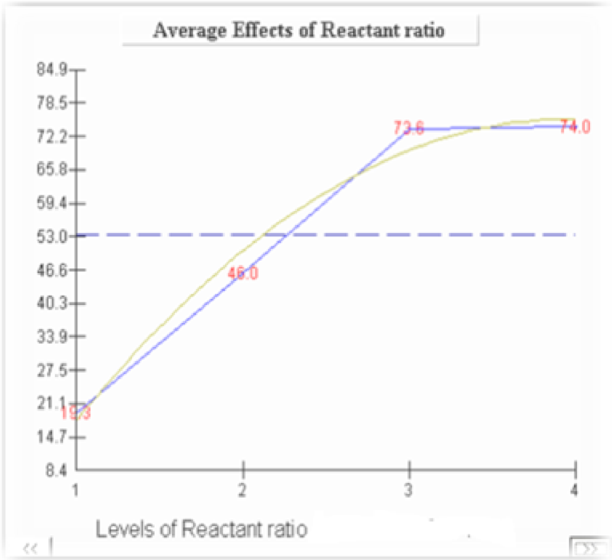



**Fig. 3(d). Average effects of Reaction ratio**

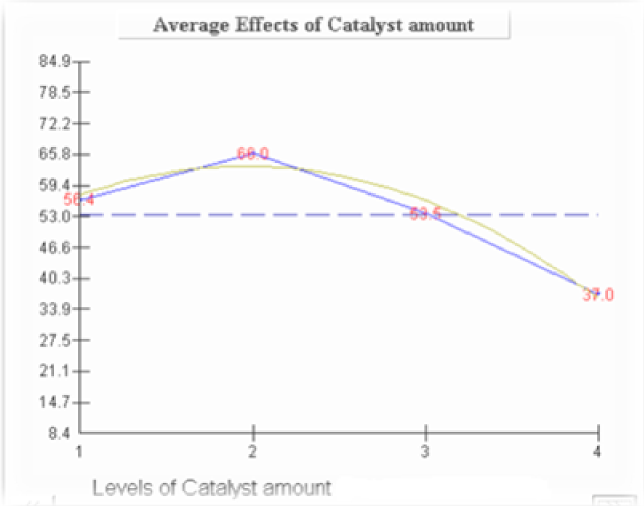



**Fig. 3(e). Average effects of Catalyst Amount**

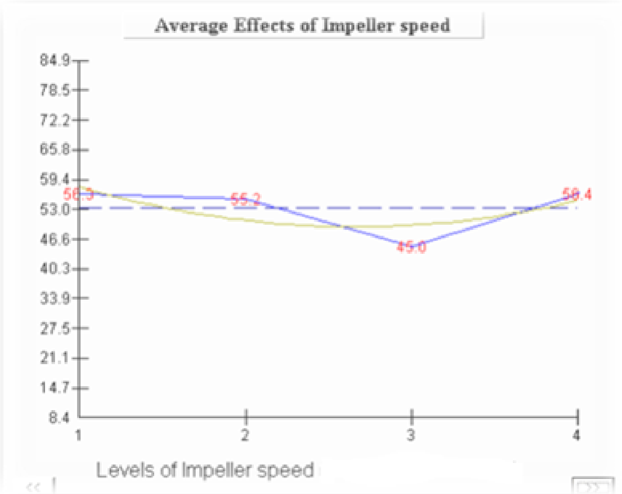



**Fig. 3(f). Average effects of Impeller Speed**

**Table- V: Optimum Condition and Performance**

| Factors | Level description | Level |
|---|---|---|
| **Types of Impeller** | I1 | 1 |
| **Temperature** | 65 | 3 |
| **Time** | 45 | 3 |
| **Reactant ratio** | 1:06 | 4 |
| **Catalyst amount** | 22.46% | 2 |
| **Impeller Speed** | 500 | 1 |

## IV. CONCLUSION

This project work has brought a concern to us to verify that using transesterification process, biodiesel is obtained with the use of rubber seed oil in a batch stirred tank reactor. The optimization of few important parameters like Impeller type-Straight blade impeller, Temperature-65ºC, Time - 45min, Reactant ratio - 1:6, Catalyst amount-22.46% (mol% of Oil), and Impeller Speed-500rpm produced a maximum output of biodiesel with the yield of 88 % . As an outcome of this project, the product can be used as fuel in diesel engines with mixing ratios B20 without using any other additional equipment's.

## V. SCOPE OF FUTURE WORK

The designed and fabricated BSTR reactor used for biodiesel production could be scaled up to 50 liter large scale reactor and can be used for batch operation. The biodiesel can also be utilized in diesel engines to trial the performance.

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