

# Catalytic Transesterification of Rubber Seed Oil to Biodiesel Using SrO/Al<sub>2</sub>O<sub>3</sub>: A Statistical Approach

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**Abstract.** This work is focused on studying biodiesel synthesis from Rubber Seed Oil (RSO) with SrO/Al<sub>2</sub>O<sub>3</sub> heterogeneous catalyst to optimise the parameters for improved biodiesel production. The transesterification reaction is carried out under different process variable conditions such as feed temperature, catalyst loading and molar ratio of methanol/oil. The Taguchi approach is applied to establish nine experimental runs in order to efficiently study the influence of these factors and also to reduce the experimentation work. The yield of biodiesel is calculated for each experiment and the results are analysed by statistical methods, such as S/N ratio and ANOVA to find out the most influential factor. According to the results, catalyst concentration and methanol-to-oil ratio are most crucial factors to improve biodiesel yield and ideal temperature plays an important role for effective reaction kinetics. Overall, This study suggests potential usage of non-edible rubber seed oil with solid base catalyst for the sustainable and low-cost production of biodiesel which in turn helps to the development of renewable energy technologies.

## 1 Introduction

Biodiesel plays an important role in the energy evolution in industrialized and developing countries. Transesterification Reaction is the dominant reaction for the production of biodiesel, which can be performed with either homogeneous or heterogeneous catalysts. The biodiesel production catalysts are mostly based on homogeneous alkaline catalysts, which include NaOH, KOH, CH<sub>3</sub>ONa and CH<sub>3</sub>OK. The selected catalysts possess increased reaction rates as the result of their selection approach. Researchers are focusing on the design of various heterogeneous catalysts because of the high feedstock price. The development of bio-nanocatalysts based on solid/enzyme hybrids has the potential to overcome some deficiencies associated with homogeneous catalysts [1][2]. Heterogeneous catalysis is preferable when considering the easy separation and reusability of catalysts for biodiesel production. Extensive utilization studies have shown that these systems suffer from major performance limitations, particularly in efficacy for some reactions and productivity. The use of a catalyst derived from biomass waste material makes the conversion to biodiesel cost-effective as they are inexpensive and easily available. The selectivity and moderate reaction conditions of biocatalysts are not fully exploited due to high costs. The application of nanocatalysts in heterogeneous catalysis and lipase immobilisation is crucial based on their excellent selectivity, improved reactivity, and advanced reaction rate for the high surface area [3].

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In another study, Optimization of the transesterification reaction was carried out through Central Composite Design involving four significant process parameters such as methanol to oil ratio, reaction time, reaction temperature and catalyst to oil ratio. Testing confirmed the fact that blast furnace slag converted to geopolymer as a result of chemical reaction without change in its composition, while for transesterification, it was found that there was a loss in strength and variation in flexural behavior up to 500°C. The maximum biodiesel yield of 97.436% was obtained for the RSM optimisation using a stirring speed of 450 RPM, reaction duration of 6.254 h and catalyst loading of 9.996 wt%. %, and methanol/animal fat ratio of 33.435 wt. % and a reaction temperature of 50.509°C as experimentally confirmed. The kinetics of the transesterification reaction followed pseudo-first order with an activation energy value of 43.76 kJ/mol[4]. The utilization of solid catalysts for the transformation of vegetable oils into biodiesel is an excellent method for biodiesel synthesis. To clarify the role of MgO in the promotion effect, a series of CaO-MgO catalysts with various Mg/Ca ratios was prepared by co-precipitation. The reaction efficiency of soybean oil and methanol was tested in a batch stirring reactor. Synthesized pure MgO had an inappropriate pore shape and difficulty in the existence of weak basic sites, which were not good for performance. The magnesium-containing CaO + MgO composites had added to them not only magnesium but also the magnesium that had been brought into the system[5]. In another paper, different types of heterogeneous catalysts are investigated for augmenting the transesterification reaction for biodiesel conversion. The work highlighted the utilization of waste materials and non-food feedstocks for saving production costs. The article evaluates the progress made by heterogeneous catalysts in biodiesel production and also reviews the characteristics of heterogeneous catalysts, focusing on solid acid catalysts, solid base catalysts, as well as acid-base and biocatalysts[6]. The incorporation of an acid-base bifunctional catalyst enables the production of different biofuel products from waste vegetable oil at mild reaction conditions. The scientists used Waste shell modified catalyst WSMCs samples in one-pot to implement the esterification and transesterification of unrefined palm-based waste oil (PDWO) for biodiesel obtainment. The maximised efficiency through the catalysed process recorded 94.1% of the highest biodiesel yield obtained in one hour under optimal conditions [7]. Some Researchers have also studied the operability of hetero-catalysts in the process of biodiesel production. Physical characteristics of biodiesel obtained with various nano catalysts and process flow diagram, were also examined. The production costs of biodiesel are minimized by the use of inexpensive nanocatalysts such as CaO, leading to the elimination of washing during the synthesis. The biodiesel production conversion of nano catalysts is more than 98% [8]. The carbonisation of glucose was effected directly in water using temperature control conditions, yielding an acid-rich carbon solid. The synthesised catalyst showed a relatively high acidity of 1.99 mmol g<sup>-1</sup> owing to the presence of numerous sulfonic (-SO<sub>3</sub>H), carboxylic (-COOH) and hydroxyl groups (-OH). Functional groups present on biochar surfaces enhance the biomass conversion activities, including esterification pathways in biodiesel production reaction [9].

The study employed Taguchi methodology to find the best biodiesel production parameters from Argemone Mexicana seed oil extraction. The three most influential elements for biodiesel production include molar ratio (A), catalyst concentration (B), and reaction or heating duration (C). The L9 orthogonal array determines three levels of evaluation for experimental trials in all three criteria. The biodiesel yield percentage was measured separately throughout all experimental runs. The statistical software Minitab calculated the signal-to-noise ratio based on the significant criterion of larger-is-better. Among the factors analyzed in the Taguchi response table, we determined the best operational levels for all parameters. ANOVA analysis determined the main influencing factors of biodiesel yield by evaluating each element's respective contribution level.

## 2 Materials and methods

The production of biodiesel from rubber seed oil (RSO) with an SrO/Al<sub>2</sub>O<sub>3</sub> catalyst starts with RSO preheating to temperatures of 70-90°C. The reaction mixture receives 2-6 wt% SrO/Al<sub>2</sub>O<sub>3</sub> catalyst, then methanol with 3:1- 9:1 molar ratio is added to RSO (Table.1). Generally, SrO contributes with good basic sites that speed up the transesterification process, leading to increased biodiesel production efficiency, whereas Al<sub>2</sub>O<sub>3</sub> serves as the support medium, increasing the surface area, improving the dispersion of SrO, and avoiding any losses due to particle detachment. The reactor maintains a set range of 600–800 rpm stirring speed during 2–4 hours of reflux conditions to drive the transesterification process. The reaction mixture needs time for settling before a separating funnel separates the biodiesel product from its glycerol by-product. The biodiesel obtains its purification by rigorous water washing procedures using 50–60 degrees Celsius deionized water to eliminate reaction catalyst and contaminants. Fig.1 shows the schematic diagram for the production of Bio-diesel. The biodiesel production process reaches its completion when the solution is heated at 110°C for 30–60 minutes to dry the product until purified biodiesel achieves readiness for analysis and further applications.

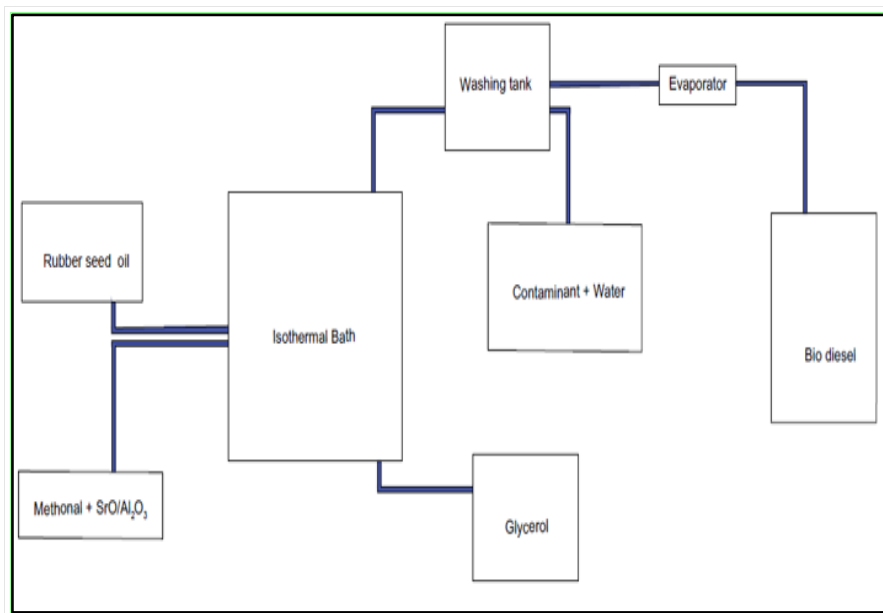


Fig. 1. Production of Bio-diesel process

Table.1.properties of Rubber seed oil

S.no	parameters	values
1	pH	5-6
2	viscosity	10.5 poise
3	Specific gravity	0.9
4	Melting point	40-45°C
5	Moisture content	7.8-8.5%
6	Acid value	1.5

### 3 Design of Experiment

According to the L9 orthogonal array in the Taguchi method, nine different experiments were conducted, where each trial indicated distinct parameter combinations at specific levels (Table.2). The statistical design structure helps to identify key process variables and their best values while offering substantial time and cost benefits over complete factorial exploration methods. Biodiesel synthesis through base-catalyzed transesterification of refined Rubber Seed oil with methanol and SrO /Al<sub>2</sub>O<sub>3</sub> catalyst led to the production of biodiesel, as measured by using this Equation (1) [11][12].

$$\text{Biodiesel yield (BD)} = (\text{mass of biodiesel production}) / (\text{mass of Rubber Seed Oil used}) \times 100$$

Table.2. input parameters with levels

Parameters	Symbols	Levels		
		1	2	3
Temperature (°C)	T	70	80	90
Catalyst concentration (wt.%)	C	2	4	6
Methanol to oil ratio	M	3	6	9

## 4 Result and Discussion

Fig. 2 shows that the optimum biodiesel yield is obtained at temperature of 90 °C and catalyst loading of 4 wt%. The 4 wt% of catalyst loading provides adequate number of active sites to improve triglyceride conversion. Low catalyst loadings lead to incomplete conversion and high concentrations increase slurry viscosity, retard mass transfer and promote soap formation resulting in decreased yields for the biodiesel and complicates downstream purification. The reaction temperature of 90 °C significantly promoted molecular collision, reduced oil viscosity, and improved the mixed reaction between substrates by which to boost the rate and efficiency of the transformation of triglycerides into biodiesel. While this high temperature results in overheating of methanol, still it can be used at 90 °C for safe and efficient conversion. This low catalyst loading and high temperature combination reduces byproduct formation and facilitates phase separation. As illustrated in Fig. 3, increasing the methanol-to-oil molar ratio (3:1), and temperature 90 °C, which improves biodiesel yield percentage due to enhanced reaction kinetics and improved mass transfer. However, excessive methanol can dilute reactants, reduce effective collisions, and complicate glycerol–biodiesel separation, ultimately lowering yield. The 3:1 methanol-to-oil ratio provides a more concentrated reaction medium that promotes effective interaction between methanol and triglyceride molecules. Under optimized reaction conditions (90 °C, a 3:1 methanol-to-oil molar ratio, and 4 wt% catalyst) ,a maximum biodiesel yield of approximately 96% was obtained (Fig. 4). This carefully balanced combination of operating parameters establishes a highly favourable reaction environment by accelerating transesterification kinetics, maximizing conversion efficiency.

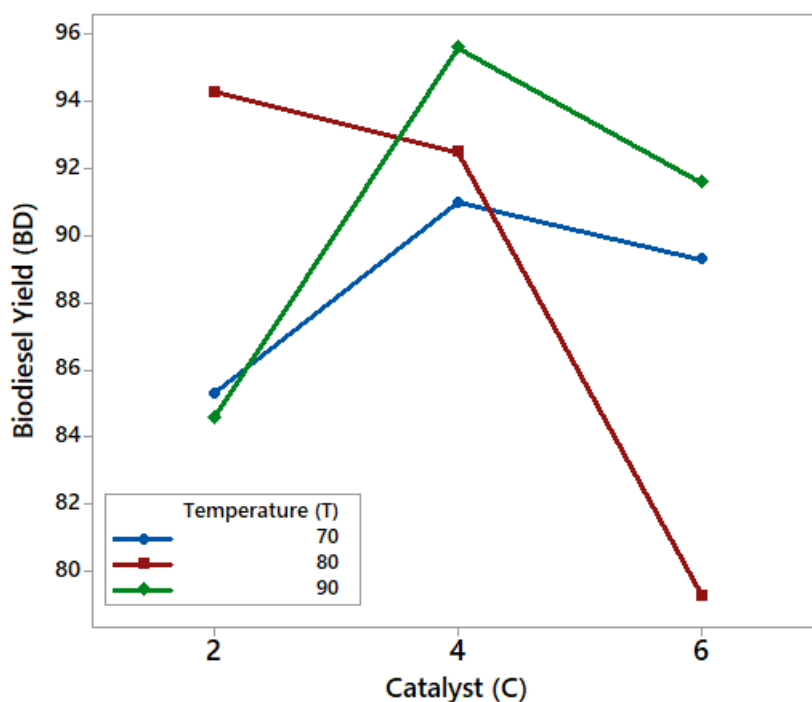


Fig.2. Catalyst versus Temperature on BD

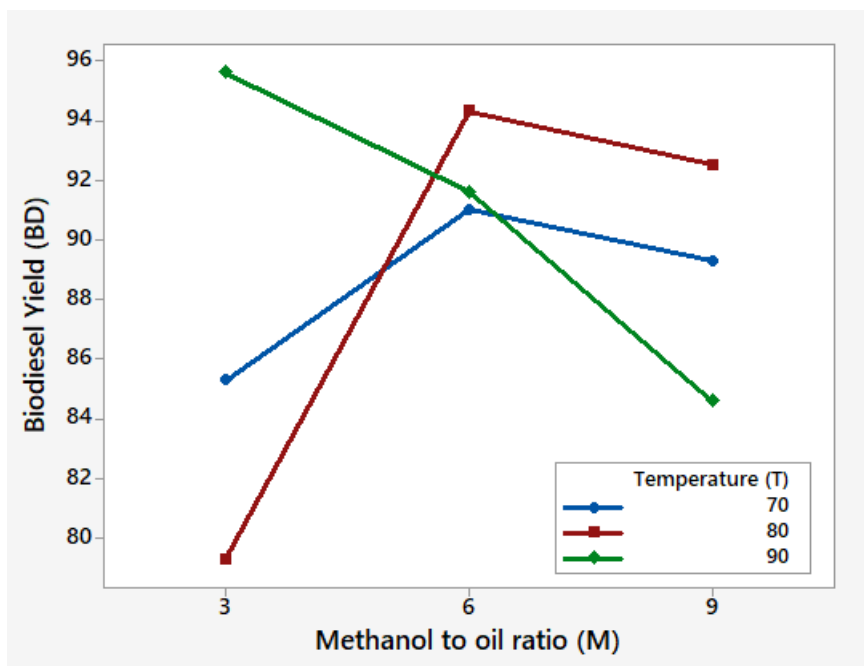


Fig.3. Methanol to oil ratio versus Temperature on BD

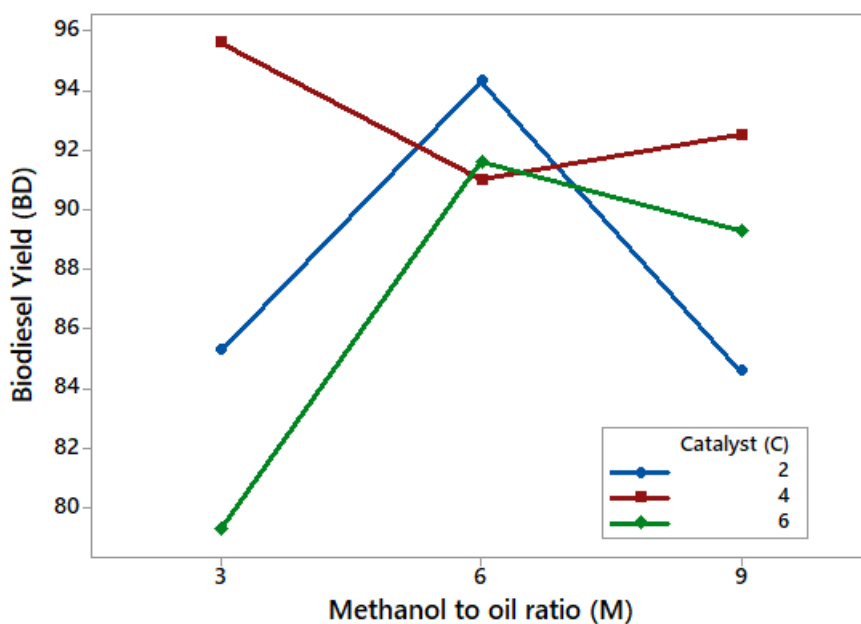


Fig.4. Methanol to oil ratio versus Catalyst on BD

### 5 Probability plot

Probability plots play an important role for assessing the distribution, variability and statistical nature of biodiesel production. These plots allow us to determine whether the experimental data comes from particular probability distribution. The biodiesel yield results are shown in Fig. 5 which has an average of 89.28, standard deviation 5.258 with probability p-value from AD test =0.512 and a statistic of Anderson-Darling 0.298. The p-value, much greater than 0.05, shows that the data is significantly distribution. The confidence level allows for their identification as robust process parameters which offer predictable and consistent biodiesel production.

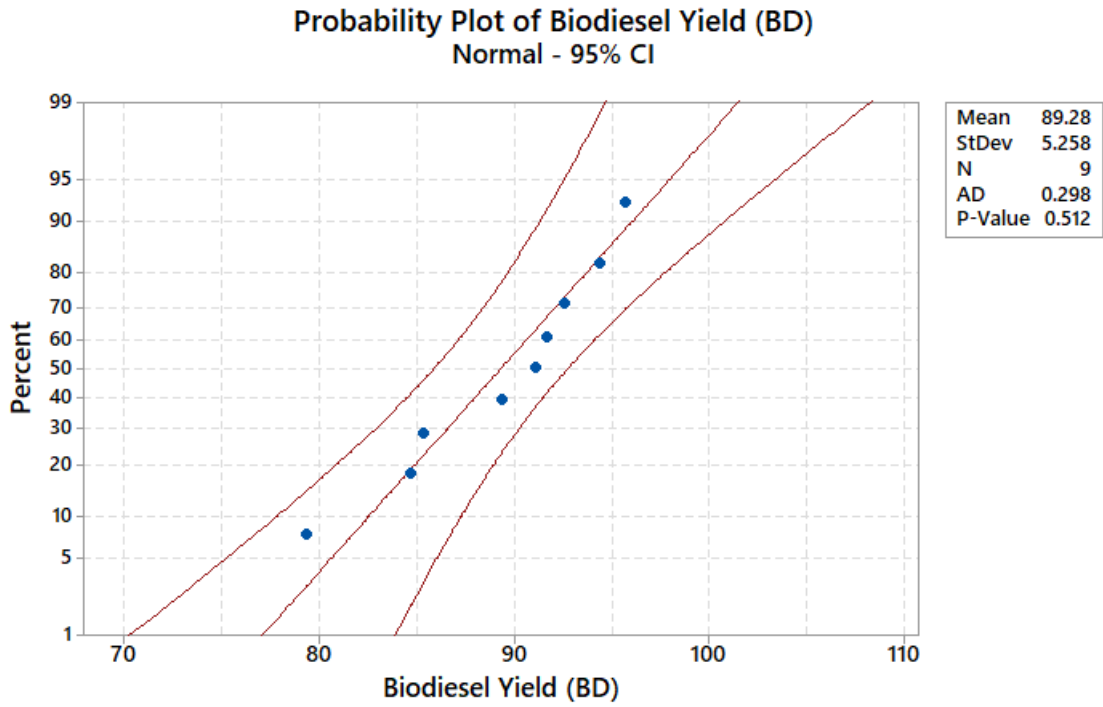


Fig.5. Probability plot for biodiesel production

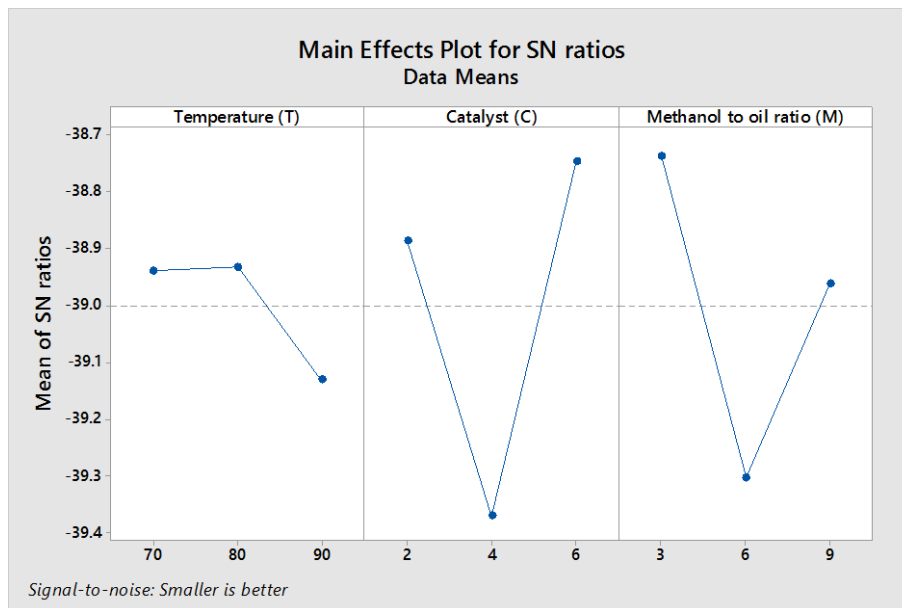


Fig.6. Main effects plot of SN ratios for BD

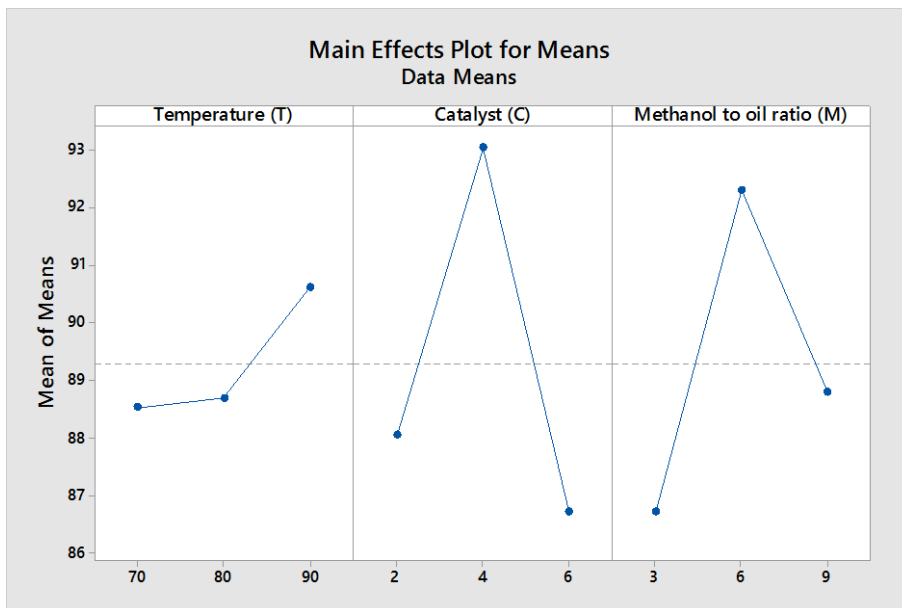


Fig.7. Main effects plot for BD

Table.3. Signal to Noise Ratios result

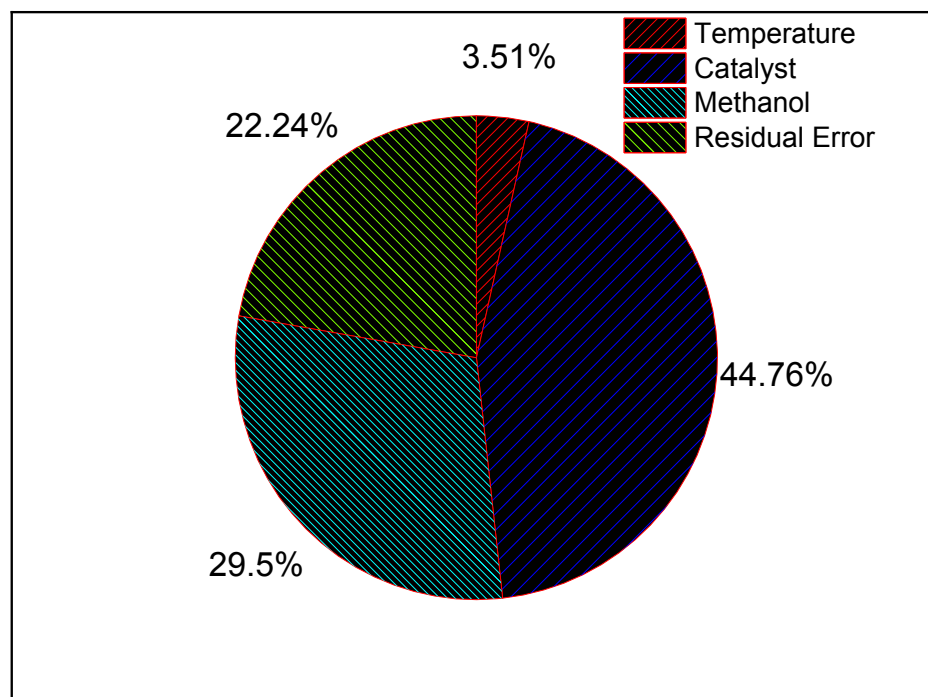
Level	Temperature (T)	Catalyst (C)	Methanol to oil ratio (M)
1	-38.94	-38.89	-38.74
2	-38.93	-39.37	-39.30
3	-39.13	-38.75	-38.96
Delta	0.20	0.62	0.57
Rank	3	1	2

The efficiency of biodiesel production was systematically determined by S/N ratio analysis and ANOVA test, which were performed with MINITAB statistical software to investigate the effect of significant process factors [13][14][15]. SN ratio for the biodiesel yield at different levels of the independent input variables temperature, catalyst concentration and methanol/oil ratio along with its ranking were shown (Table 4).

Table.4 ANOVA for BD

Source	DF	Seq SS	Adj MS	F	P	percentage
Temperature (T)	2	0.07657	0.03829	0.45	0.0727	3.51
Catalyst (C)	2	0.97774	0.48887	5.6	0.0023	44.76
Methanol to oil ratio (M)	2	0.64441	0.32221	3.3	0.0403	29.5
Residual Error	2	0.48578	0.24289			22.24
Total	8	2.18450				

The formulation significance index indicates that the catalyst is the most influential factor affecting biodiesel yield (Rank = 1), and the optimal process parameter combination for maximum biodiesel production is T3–C2–M2, corresponding to 90 °C, 4 wt% catalyst, and a 6:1 methanol-to-oil ratio.



**Fig.8.** Contribution percentage of parameters on BD

Table 4 and Fig. 8 confirm the significance of each factor based on their F-value and P-value, where Catalyst concentration had most important factor which has a contribution of 44.76%, followed by Methanol-to-Oil ratio of 29.5%, Temperature of 3.51%. This full statistical study clearly shows the relevance of parameter optimisation in biodiesel production and validates a process for an efficient scaling-up at industrial level.

## 6 Conclusion

This work clearly demonstrates that Rubber Seed Oil (RSO) is a promising green material for the preparation of biodiesel using SrO/Al<sub>2</sub>O<sub>3</sub> catalyst. The effects of process parameters i.e., reaction temperature, catalyst concentration, and methanol-to-oil molar ratio were systematically analysed using the Taguchi L16 orthogonal array. The probability plot analysis results make the experimental data statistically viable, as well as relatively agreeing with each other (89.28% for biodiesel) and possessing a p-value greater than 0.05 to prove the adequacy of model used. The interaction plots showed that the optimal yield of 96% can be obtained at a methanol/oil ratio of 3:1, catalyst concentration of 4 wt.% and temperature of 90 °C. The S/N analysis further confirmed the most significant process parameters to be T5-C4-M5 (i.e., temperature: 90°C, Catalyst 4wt%, 6:1 methanol-to-oil ratio). ANOVA showed that catalyst concentration (44.76%) is the most significant variable, followed by molar composition of methanol to oil (29.5%) and temperature (3.51%). The results reveal the extremely important role of catalyst loading and methanol ratio in enhancement of biodiesel production. The present extensive study provides some valuable information of the efficiency of parameter optimization in improving biodiesel yield for industrial-scale production of biofuel from non-edible oil sources and sustainable heterogeneous catalyst.

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