OPTIMIZATION BIODIESEL PRODUCTION FROM HONGE OIL (PONGAMIA PINNATA)

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Resume
Rapid industrialization and increased diesel engine usage have driven up diesel consumption, burdening economies with heavy petroleum product imports. Diesel fuel emissions, including carbon dioxide, carbon monoxide, and smoke, pose environmental concerns. Biodiesel produced from non-edible vegetable oils offers a sustainable solution.
Therefore, a study was conducted on optimizing Honge oil biodiesel production using Taguchi optimization. The research aimed to maximize biodiesel yield from Honge oil, employing Taguchi's technique for enhanced output. Through systematic optimization, the study sought to achieve an efficient and improved process for generating biodiesel from Honge oil. During experimental work, reaction time, reaction temperature and molar ratio of methanol/oil were varied. Among all the experiments conducted during the optimization process, the highest yield of biodiesel was about 88.7 %, as compared to weight of oil. These findings contribute to improving biodiesel production efficiency and promoting its use as an eco-friendly alternative to fossil fuels.

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1 Introduction

The increasing use of vehicles and industrialization has led to a significant increase in energy consumption. Many countries rely on fossil fuels, especially petroleum products, which are concentrated in a few parts of the world. These reserves are limited, and with increasing consumption, it is possible that they will be depleted in the near future [1-4]. This would put a strain on oil-deficient countries, like India, which import most of their petroleum products. Diesel fuel is a prevalent energy source for compression ignition engines in various sectors, such as automotive, power generation, and agriculture. From the literature review it is found that fossil fuels are depleting at faster rate, which may cause the fuel crises in the near future. One of the alternatives for fossil fuels (diesel fuel) can be the biodiesel derived from native vegetable oils [5-11].

Using straight vegetable oil in compression ignition engines leads to reduced thermal efficiency and increased smoke emissions, primarily because of its high viscosity and low volatility. In addition, the clogging of fuel filters and injectors, carbon deposits, and engine damage can be observed. The vegetable oil can be made suitable for diesel engine applications by reducing the glycerol content in it to promote its volatility [12-17]. Biodiesel production involves the conversion of vegetable oil/animal fats into their esters transesterification [18-20]. The yield of biodiesel can be optimized by adjusting process parameters.

In this study, the Taguchi method was employed to optimize the conversion of Honge oil (Pongamia pinnata) into its methyl ester, commonly referred to as Honge biodiesel. The highest yield of biodiesel was obtained with an 8:1 molar ratio of methanol to oil, a reaction temperature of 60 °C, and a catalyst concentration of 0.6 %. This study suggests that the Honge oil can be used for biodiesel production. By optimizing the production process using the Taguchi method, it is possible to improve the biodiesel yield. This could lead to increased utilization of biodiesel as a renewable fuel, thereby reducing reliance on imported petroleum products.

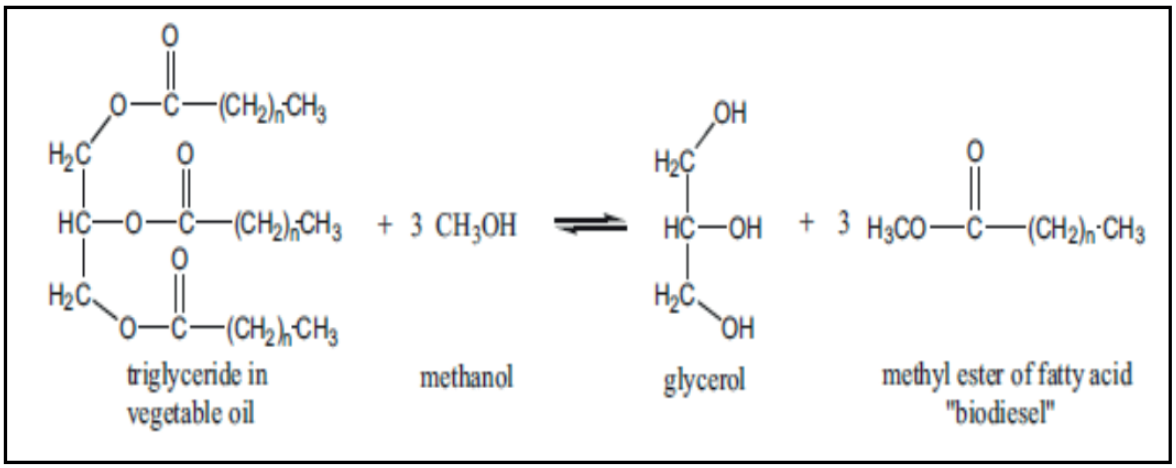

Figure 1 *Transesterification reaction*

2 Literature survey

Graboski et al. [21] studied the straight vegetable oils used in the engine, which leads to various problems like fuel filter clogging, poor atomization, and incomplete combustion because of its highly viscosity, high density, and poor non-volatility. To reduce the viscosity of the straight vegetable oil, the following four techniques were adopted: heating/pyrolysis, dilution/blending, micro-emulsion, and transesterification. Among all these techniques the transesterification is an extensive, convenient and the most promising method for the reduction of viscosity and density of the straight vegetable oils. However, this process introduces additional costs due to the transesterification reaction, which requires both chemical inputs and energy inputs in the form of the process heat.

Jana, et al. [22] focused on optimization of the transesterification of waste cooking oil under the CaO-based catalyst, derived from an ostrich eggshell by different types of machine learning approaches. They used various machine learning techniques like the fuzzy logic system (T1FLS), response surface methodology (RSM), adaptive neuro-fuzzy inference system (ANFIS), and type 2 fuzzy inference logic system (T2FLS). They studied the effect of various parameters on the biodiesel yield. They found the maximum yield of biodiesel at 93.3 % with fuzzy logic models.

Chintagunta et al. [23] studied production of biodiesel from waste cooking oil. They found that biodiesel is an eco-friendly, renewable, and potential liquid biofuel mitigating greenhouse gas emissions. They indicated that waste cooking oil can be a suitable feedstock for the biodiesel production.

Tamoradi et al. [24] goal was to optimize the production of Biodiesel from rapeseed oil and waste corn oil. They obtained the optimum yield of biodiesel at a molar ratio of 13:1 and a catalyst concentration of 8 %w/vol at 65 °C in 7 h reaction time. These results were validated over ANOVA and 3D response surface contour plots.

Degfie et al. [25] used a CaO nano-catalyst as a catalyst for biodiesel production from waste cooking oil. They achieved biodiesel yield of biodiesel at 96 % at waste cooking oil to methanol molar ratio of 1:8, 1 wt. % of CaO nano-catalyst, 50 °C reaction temperature and 90 minutes reaction time. The various properties of biodiesel, like the viscosity, specific gravity, water and sediment, total acidity, ash content and sulfur content, were tested according to the ASTM D 6571 and found in good agreement with the standard.

From the above literature survey, it can be found that combination of various factors effecting the biodiesel production varies with the type of feed stock.

3 The transesterification process

Transesterification process involves conversion of triglyceride from vegetable oil or animal fat into its esters and glycerol through a reaction with alcohol and a catalyst. This reaction is depicted in Figure 1. It shows that reaction between the triglyceride and alcohol (molar ratio of 1:3), results in three moles of fatty acid esters and one mole of glycerol. It is important to note that transesterification is a reversible reaction and tends to proceed at a relatively slow rate [19-20, 26-27].

4 Factors effecting the biodiesel production

4.1 Molar ratio of alcohol and oil

A molar ratio of alcohol to oil is a crucial factor in transesterification process, impacting both the reaction rate and cost. A higher molar ratio accelerates the transesterification reaction but reduces catalyst availability, potentially lowering the biodiesel yield. The recommended molar ratio typically ranges from 4:1 to 10:1, with methanol commonly used due to its affordability. Achieving the optimal molar ratio depends on the specific oil type and desired biodiesel

characteristics. By precisely managing the alcohol-to-oil ratio, it is feasible to produce the high-quality biodiesel with a favorable yield [28].

4.2 Reaction temperature.

Rate of any chemical reaction depends on reaction temperature. With increase in temperature, chemical reaction accelerates, which reduces the time for completing the reaction. In the case of the transesterification process, an increase in temperature results in a shorter reaction time due to the reduction in the viscosity of the oil. However, increasing the reaction temperature beyond a certain limit, speeds up the saponification of triglycerides resulting in lower biodiesel yield. Heating reactants beyond the vaporization temperature of alcohol, results in lower biodiesel yield due to decreased availability of reactants for reaction [29]. Hence, during the transesterification reaction, reactants are to a temperature, which accelerates the reactions and does not cause evaporation of alcohol.

4.3 Free fatty acid and water content in oil

The quantity of free fatty acid (FFA) and water within the oil plays a significant role in determining the biodiesel yield. Oils with elevated FFA levels necessitate a greater amount of alkali catalyst to counterbalance these acids, resulting in increased production expenses. Moreover, heightened FFA levels can impede the reaction process, slowing it down notably. Water present in the oil also contributes to this deceleration, creating hurdles in the separation of glycerol. Additionally, it has the potential to generate foam during the reaction, further complicating the process [30]. By carefully controlling the FFA and water content, it is possible to achieve optimal conditions for transesterification and produce the high-quality biodiesel with a satisfactory yield.

4.4 Catalyst

Choice of catalyst and its quantity in the transesterification process significantly impacts the yield and quality of biodiesel. Alkali catalysts like KOH and NaOH are commonly employed due to their effectiveness at lower temperatures and cost-effectiveness. However, their usage can result in soap formation when the oil's free fatty acid (FFA) content is high. Acid catalysts, such as H_2SO_4 and HCl, are less sensitive to FFA content but exhibit slower reaction rates and higher corrosiveness.

The quantity of catalyst employed also influences the biodiesel outcome. A higher catalyst amount accelerates the reaction, but it may lead to soap formation. Conversely, a lower catalyst amount prolongs the reaction time and can result in incomplete conversion [29]. The optimal catalyst type and quantity depend on the specific oil type and desired biodiesel properties. By carefully selecting and balancing these factors, it is possible to achieve high-quality biodiesel with a satisfactory yield.

4.5 Stirring rate

The transesterification process is a chemical conversion that transforms triglycerides into esters and glycerol, facilitated by an alkali, acid, or enzyme catalyst. Since the reactants, alcohol, and oil, have different densities and are immiscible, the reaction occurs solely at their interface. To ensure uniformity, continuous mixing of the reactants is employed using either a mechanical or magnetic stirrer. The mixing intensity must strike a balance between the thorough mixing and prevention of soap formation, which can occur if the reaction is reversed due to excessive agitation [31]. Achieving the optimal mixing intensity is crucial for producing the high-quality biodiesel with a favorable yield, contingent on the specific oil type and desired biodiesel properties.

4.6 Reaction period

Conversion of triglycerides to esters increases with reaction time, but it is crucial to find the optimal duration as the reaction is reversible and prolonged reaction times can result in the reversion of esters to triglycerides. The optimal reaction time depends on factors such as the type of oil, catalyst quantity, and desired biodiesel properties [17]. Typically, a reaction time of 1-2 hours is sufficient, but other considerations include reaction temperature, catalyst type, and FFA content. Experimentation is recommended to determine the ideal reaction time for achieving the high-quality biodiesel production with a desirable yield.

5 Methodology

5.1 Production of biodiesel

Honge seeds are collected from trees planted on roadsides, forests, and dry private land around Bidar city. The seeds are manually decorticated and dried for four days until they become completely dry. Using an oil extraction machine, the dried seeds are crushed to obtain the oil, which has a recovery rate of about 45% compared to the kernel weight. Chemicals, such as phenolphthalein indicator, anhydrous methanol, potassium hydroxide, sulphuric acid, and potassium hydroxide pellets, are purchased from the local market. A biodiesel preparation kit consisting of a 2-liter capacity three-neck round bottom flask, a magnetic stirrer with

(a) (b)

Figure 3 *a) Transesterification reactor b) settling of biodiesel and glycerol*

Figure 2 *Determination of FFA in oil*

speed control, and other glassware is used for the conversion process.

Initially the FFA content of Honge oil is determined using the titration method. During this method, Honge oil is titrated with phenolphthalein indicator until oil turns into pink color as shown in Figure 2 and accordingly FFA content is determined. The Honge oil is initially subjected to esterification to reduce the FFA below 4%. To achieve this, methanol and sulphuric acid are used in a molar ratio of 6:1, with the acid concentration of 0.5% of the oil. The esterification is carried out at 60 °C for 75 minutes, while stirring at 400 rpm. After the reaction, the mixture is left to settle for 5-8 hours, and the FFA content is determined, resulting in approximately 3.2% FFA. Subsequently, the transesterification is performed using methanol and potassium hydroxide. The oil-methoxide mixture is maintained at 60 °C for 1.5 hours during which it is stirred at 400 rpm in Figure 3(a). Once the trans-esterification is finished, the products of reaction are allowed to settle for 24 hours, leading to separation of biodiesel and glycerol into distinct layers, with the biodiesel as the upper layer, which can be seen in Figure 3(b). The biodiesel and the glycerol are separated in distinct layers (upper layer is the biodiesel and the lower layer is glycerol). It should be noted that the biodiesel might contain impurities like residual methanol and soap. Therefore, the raw biodiesel is washed using the distilled water to eliminate the soap content. The biodiesel is heated to 100 °C to remove traces of water if any present in it. Finally, the biodiesel is filtered to eliminate i mpurities.

5.1.1 Optimization of factors affecting the transesterification process

As previously mentioned, yield of biodiesel is influenced by various factors. To determine the optimal combination of these factors for achieving the highest biodiesel yield, it would be impractical and costly to systematically vary each factor individually. Instead of that, in this study was employed the Taguchi method, an approach to design of experiment, to efficiently identify the best combination of molar ratio, catalyst concentration, and reaction temperature that would

Table 1 *Factors affecting the biodiesel production*

Process parameters	Levels		
	1	2	3
Molar ratio of methanol to oil	4:01 (A1)	8:01 (A2)	10:01 (A3)
Catalyst concentration % (w/w of oil)	5 (B1)	0. 6 (B2)	0. 8 (B3)
Reaction temperature	50 (C1)	60 (C2)	65 (C3)

Table 2 *Orthogonal array*

Trial No.	Methanol-oil molar ratio (A)	Catalyst concentration (B)	Reaction temperature (C)
1	4:1	0.5	50
2	4:1	0.6	60
3	4:1	0.8	65
4	8:1	0.5	60
5	8:1	0.6	65
6	8:1	0.8	50
7	10:1	0.5	65
8	10:1	0.6	50
9	10:1	0.8	60

maximize the biodiesel yield, while minimizing the number of trials required.

The Taguchi method is a statistical technique used to optimize the process by identifying and minimizing the variability associated with influential factors. By utilizing this method, effort is made to determine the optimum combination of process parameters, which results in the highest yield of biodiesel. This study highlights the value of utilizing the Taguchi method as a robust tool for process optimization, as it allows for identification and mitigation of influential factors' variability.

In the study, the process parameters are carefully selected to determine their optimal levels. A literature review and equipment availability were considered in choosing the following parameters. In the present study, a molar ratio of methanol to oil levels of 4:1, 6:1, and 8:1; concentration of catalyst at levels at 0.5%, 1%, and 1.5% (w/w) as compared to oil and reaction temperature at levels from 50 °C to 70 °C, were considered. Those parameter levels were based on extensive research and practical considerations, forming the basis for subsequent experiments and analysis. Table 1 shows the parameters that are affecting the biodiesel production.

To analyze the effect of different levels of process parameters on the biodiesel yield, an orthogonal array (OA) was chosen. The choice of the OA is done based on the total number of degrees of freedom (DOF) of all the process parameters. The number of DOFs for each process parameter is calculated using the formula (L - 1) [27], where L is the number of levels of each process parameter. For example, the number of DOFs for the molar ratio is 2, as there are 3 levels (3-1 = 2). Similarly, the number of DOFs for catalyst concentration and reaction temperature is also 2. The total number of DOFs for all the process parameters is calculated by multiplying the numbers of DOFs of each parameter by the number of parameters, resulting in a total number of DOFs of 6. According to the OA selection criteria, the OA chosen for analysis should have the number of trials equal to N+1, where N represents the total number of DOFs of all the process parameters.

A standard L9 orthogonal array (3x3) was selected for the present investigation, which suggests that conducting 9 experiments would be sufficient to optimize the parameters. The details of the L9 orthogonal array used in the present work are shown in Table 2.

After conducting the experiments according to the selected orthogonal array, the signal-to-noise ratio was calculated for each trial. The signal-to-noise ratio is a logarithmic function, which measures the deviation of the quality characteristics from the optimum value. The objective in this study was to maximize the yield of biodiesel. Hence, the "higher the better" S/N ratio is used. The mean S/N ratio and the S/N ratio for each level of each process parameter were determined to predict the optimal results. Table 3 presents the biodiesel yield and S/N ratio for each trial.

After computing the signal-to-noise ratio for each trial, the average S/N ratio for each level process parameter was determined. These values are then plotted in Figures 4, 5, and 6, respectively. From these

Table 3 *Biodiesel yield and S/N ratio for each trial*

Trail No	Biodiesel yield (% of wt of oil)	S/N Ratio
1	83.3	38.413
2	86.5	38.74
3	85.7	38.66
4	87.5	38.84
5	87.3	38.82
6	87	38.79
7	83.7	38.455
8	85.2	38.609
9	85.7	38.66
Average	85.7	38.665

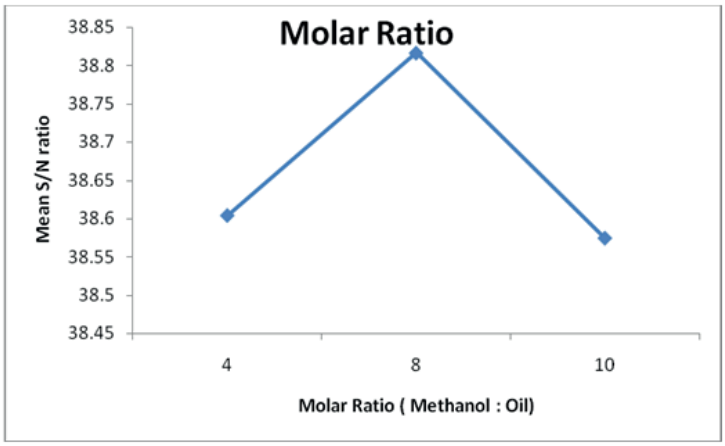

Figure 4 *Molar ratio*

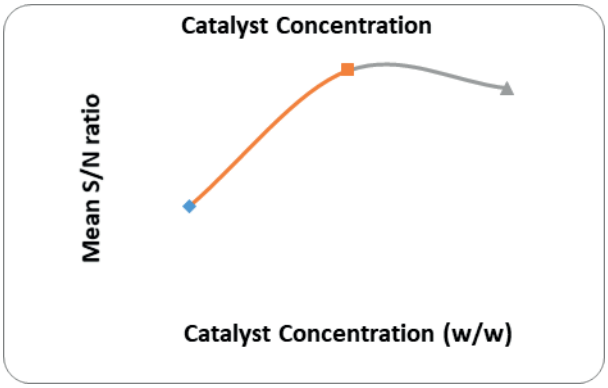

Figure 5 *Catalyst concentration*

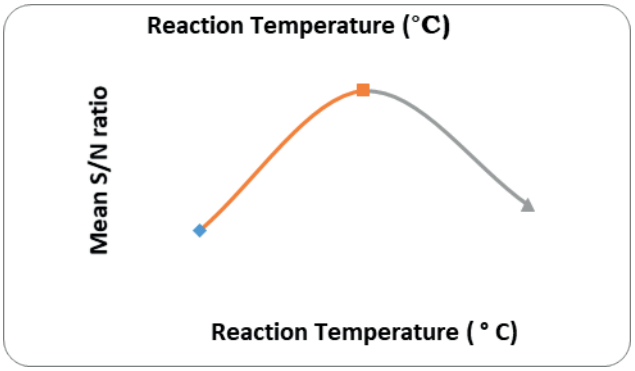

Figure 6 *Reaction temperature*

figures is found that the S/N ratio for all the three parameters is the highest at level 2, which shows that at those levels of process parameters the biodiesel yield can be the highest. The optimal combination of process parameters for achieving the highest biodiesel yield is identified as A2-B2-C2 (highest S/N ratio). In other words, according to the S/N ratio analysis, the optimal parameters are: A at level 2 (8:1), B at level 2 (0.6 wt. %), and C at level 2 (60° C).

The biodiesel yield, at optimal combination of process parameters (Yopt), is determined by

$$Yopt = M + \sum_{i=1}^{0} (X_i - M), \quad (1)$$

where M represents the overall mean of the biodiesel yield of all test runs, and X_i represents the mean biodiesel yield for the trials with i-th level of the given control parameter X. The biodiesel yield, at the optimal combination A2-B2-C2, was observed to be 88.7 %. To confirm the validity of these results, a confirmation test was conducted at the same combination of A2-B2-C2, and the biodiesel yield is found to be 87.43 % of the weight of Honge oil. This value is very close to the calculated value, which supports the accuracy of the optimization process.

6 Conclusions

This study employed the Taguchi approach for optimizing the biodiesel yield by determining the optimal combination of methanol to oil molar ratio, concentration of catalyst and reaction temperature. The Taguchi method is a statistical approach that identifies influential factors in a process. An orthogonal array (L-9) experimental design was used to evaluate all the possible combinations of the process parameters economically. The study revealed that at combination of a methanol to oil (molar ratio of 8:1), a catalyst concentration (0.6 wt. %), and a reaction temperature (60 °C) a biodiesel yield is approximately 88.7 %. The results of the Taguchi analysis are compared to those of the experimental confirmation test. The results of Taguchi prediction are close to results of the conducted confirmation test. At optimized combination of process parameters, the biodiesel yield is the highest of all the experiments conducted. In conclusion, this study demonstrates the efficacy of the Taguchi method in determining the optimum combination of process parameters for the maximum yield of biodiesel from the Honge oil.

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Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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