

# Exploring the Effects of the Design of Experiments Technique on the Biodiesel Production from Tung Oil

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**ABSTRACT**

**This study deploys the Design of Experiments (DoE) techniques to improve the biodiesel production process from tung oil. Biodiesel from vegetable oils, animal fat, and algae are promising for sustainable energy production, depending on various factors, such as Molar Ratio (MR), Catalyst Concentration (CC), Reaction Time (RT), and Reaction Temperature (RTemp). In order to obtain the best process conditions, this study used the Taguchi model of L16 orthogonal array combined with Signal-to-Noise Ratio (SNR) and Analysis of Variance (ANOVA). The results were analyzed utilizing MINITAB-21 statistical software to predict optimal process parameters.**

**Keywords-***transesterification; tung oil; Design of Experiments (DoE); optimization; Taguchi technique; energy efficiency*

**I. INTRODUCTION**

The increasing energy usage in transportation and industry depending on diesel, has led researchers to explore alternatives to petroleum products. The International Energy Agency (IEA) reports that energy demand in 2025 is expected to be 40% to 50% higher than in 2024, making the usage of biofuels, such as biodiesel, more necessary than before. Biodiesel from animal fats, vegetable oils, and Water-based Cooking Oils (WCO), is a substantial substitute for diesel engines due to its technical and environmental benefits [1, 2]. It is preferred over petroleum because of its ability to biodegrade and contains a high cetane number, low levels of carbon monoxide, unburned hydrocarbons, and sulfur, exhibiting high viscosity, oxidation, and poor fuel atomization. Due to the increasing demand for biodiesel, other ways of production are examined, such as the usage of non-edible oils; jatropha, castor and jojoba, which are efficient, but tung oil seems to be the best option [3]. Tung oil is extracted from the seeds of tung tree (*Vernicia fordii*), is classified as a dry oil, and it is adaptable to various soil conditions, from dry and rocky environments to wetlands. Tung oil is mainly produced in China from the tung tree after the third year of planting and can become economically viable in the fourth or fifth year with intensive cultivation. The peak production is reached in the 10th to 12th year, and the trees have a lifespan of about 30 years. The global productivity of tung oil is 1.3 Mt per year [4]. The main objective of the present research is to improve the efficiency of the transesterification process for tung biodiesel by optimizing various key factors. Since tung oil utilization for biodiesel production has not been thoroughly investigated [5], it is essential to study the properties of tung oil and Tung Oil Methyl Ester (TOME) and compare them with the ASTM D7652 biodiesel standard. By using the DOE approach,

specifically the Taguchi orthogonal array technique, the most favorable permutation of experimental factors was systematically determined based on the results of 16 experimental trials [5].

**II. MATERIALS AND METHODS**

The research approach was designed to ensure the accuracy of the results and to provide credibility through the careful selection and handling of all materials in a controlled environment [6].

**A. Materials**

Unrefined tung oil from local oil producers in Pune was used in its natural state without any further treatment, along with high purity chemicals including 99% methanol and potassium hydroxide (KOH) catalyst.

**B. Transesterification of Tung Oil**

In the transesterification process, triglycerides react with alcohol in the presence of a catalyst, which can be an acid, base, or lipase. This reaction results in the formation of monoalkyl esters, identified as biodiesel, and glycerin. In order to prepare tung oil for biodiesel production it is essential to filter it with paper [7]. Figure 1 shows the flowchart of the process, while Figure 2 depicts the tung tree, its seeds, and the extracted oil. In this experiment, 150 grams of tung oil were preheated to a temperature range of 100 °C - 110 °C to remove moisture before cooling to 55 °C to begin the transesterification process. The preheated oil was then combined with methyl alcohol and KOH catalyst in varying ratios to evaluate their influence on biodiesel. The reaction mixture was subjected to controlled heating and continuous stirring for different periods of time to optimize the conversion process. After the reaction, the products were allowed to settle in a separation funnel,

resulting in two distinct layers: glycerol as a by-product at the bottom and biodiesel, or ester layer, at the top. The biodiesel was then separated and purified using the bubble washing method, which effectively removed residual catalyst [8]. Throughout the process, key parameters, such as MR, CC, RT, and RTemp were systematically varied to analyze their effects on biodiesel production and quality. The final purified biodiesel was evaluated for purity and quality based on standards [9].

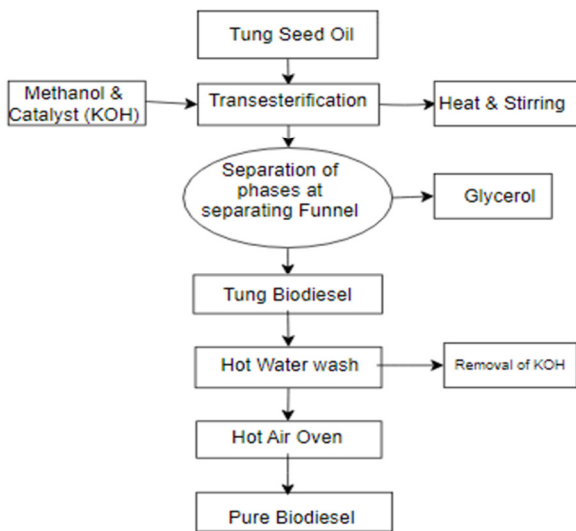


Fig. 1. Flowchart of biodiesel production process.

### C. Characterization

The physical and chemical properties of the obtained TOME were evaluated using internationally accepted standard methods to ensure reliability and comparability. Table I provides a detailed list of the main parameters evaluated for diesel, crude tung oil, and TOME along with the measured values. The analysis focuses on critical fuel properties, such as specific gravity, viscosity, fire point, calorific value, flash point, and cetane number, to determine the suitability of TOME as a biodiesel fuel [10]. The results show the following:

- **Specific gravity:** The specific gravity of TOME (0.88) is closer to diesel (0.83) than crude tung oil (0.90), indicating its improved compatibility with diesel engines after transesterification.
- **Kinematic viscosity:** The viscosity of TOME (4.60 cSt) is within the acceptable range for biodiesel and shows a significant reduction compared to crude tung oil (121.74 cSt). This makes TOME suitable for diesel engines without clogging problems, unlike untreated tung oil [11].
- **Fire Point:** TOME has a fire point of 180°C, which is much higher than diesel (64°C), but lower than crude tung oil (360°C) ensuring more safety.
- **Calorific Value:** The energy content of TOME (39,000 kJ/kg) is slightly lower than diesel (42,000 kJ/kg) but significantly higher than crude tung oil (35,810 kJ/kg). This indicates efficient energy release during combustion.

- **Flash Point:** With a flash point of 194°C, TOME is safer to store and transport than diesel (62°C), reducing the risk of fire.
- **Cetane Number:** TOME has a cetane number of 42, slightly lower than diesel (48). While this can affect ignition quality, it remains within acceptable limits for biodiesel use [12].

### III. DESIGN AND ANALYSIS OF EXPERIMENT

Designing and analyzing experiments is a critical process to understand and refine many engineering and scientific methods. It allows for a thorough examination of how various input factors relate to each other and affect outcomes. By using well-organized experimental designs, researchers can achieve better efficiency, less variability, and higher product quality [13]. The present study uses an experimental framework to determine the variables that affect biodiesel production and to optimize the associated process parameters. A well-structured experimental design not only helps to identify primary factors, but also conserves resources while improving result accuracy. Various statistical methods, such as regression analysis and ANOVA, are typically used to estimate the experimental data and confirm the results [14].

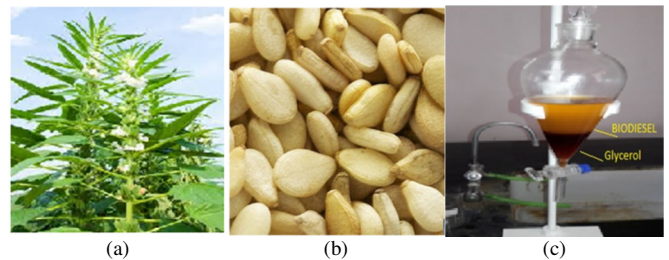


Fig. 2. (a) Tung tree, (b) tung seed, (c) tung biodiesel and glycerol.

TABLE I. COMPARATIVE ASSESSMENT OF DIESEL, TUNG OIL, AND TOME PROPERTIES

Property	Diesel	Tung oil	TOME
Specific gravity	0.83	0.90	0.88
Kinematic viscosity at 40°C (cSt)	3.72	121.74	4.60
Fire point (°C)	64.00	360	180
Calorific value (kJ/kg)	42,000	35,810	39,000
Flash point (°C)	62	322	194
Cetane no	48	--	42

### A. Design of Experiment

DoE, according to Taguchi, goes through a systematic process that seeks to gain a better understanding of the behavior of a system, allowing researchers to save costs and minimize the number of the conducted experiments. In this study, an experimental design based on the orthogonal arrays was used to determine the high accumulation factors of biodiesel production. An L16 orthogonal array was employed to improve the production of tung biodiesel, effectively optimizing the results with a minimum number of experiments. In this case, the number of experiments according to the selected levels and factors is specified in (1) and the response parameters are determined [15]:

$$N = (L - 1) F + 1 \tag{1}$$

where  $L$  is the selected level and  $F$  the number of factors.

**B. Optimization**

The data from the experiments were loaded into Minitab software for analysis, focusing on maximizing the desired criteria, as shown in (4). In Table II, the different parameters were examined at three different levels [15], while Table III portrays the tung biodiesel yield and SNR for the 16 experiments:

$$SNR_i = 10 \log \left( \frac{\bar{y}_i^2}{s_i^2} \right) \tag{2}$$

$$SNR_i = -10 \log \left( \sum_{j=1}^n \frac{y_j^2}{n} \right) \tag{3}$$

$$SNR_i = -10 \log \frac{1}{n} \left( \sum_{j=1}^n \frac{1}{y_j^2} \right) \tag{4}$$

where  $y_i = \frac{1}{n} \left( \sum_{j=1}^n y_{ij} \right)$  is the mean value of response and  $s_i^2 = \frac{1}{n-1} \left( \sum_{j=1}^n y_{ij} - \bar{y}_i \right)$  is the variance.

TABLE II. FACTORS AT VARIOUS LEVELS

Factors	Level 1	Level 2	Level 3	Level 4
MR	4.5	5	6	7
RTemp	50	55	60	65
RT	30	45	60	75
CC	0.5	1	1.5	2

TABLE III. ORTHOGONAL ARRAY UTILIZED FOR DOE INVOLVING DIVERSE PARAMETERS

MR	CC (wt %)	RT(min)	RTemp (°C)	Yield (wt %)	SN-RL
4.5:1	0.5	50	30	83.88	38.4732
4.5:1	1	55	45	84.45	38.5320
4.5:1	1.5	60	60	86.12	38.7021
4.5:1	2	65	75	89.95	39.0800
5:1	0.5	55	60	80.54	38.1202
5:1	1	50	75	82.11	38.2879
5:1	1.5	65	30	88.34	38.9231
5:1	2	60	45	86.65	38.7554
6:1	0.5	60	75	86.64	38.7544
6:1	1	65	60	83.62	38.4462
6:1	1.5	50	45	84.26	38.5124
6:1	2	55	30	91.38	39.2170
7:1	0.5	65	45	83.36	38.4192
7:1	1	60	30	87.1	38.8004
7:1	1.5	55	75	89.18	39.0053
7:1	2	50	60	90.22	39.1061

TABLE IV. RESPONSE TABLE FOR SN RATIOS (LARGER IS BETTER)

Level	MR	CC (wt %)	RTemp. (°C)	RT(min)
1	38.70	38.44	38.59	38.85
2	38.52	38.52	38.72	38.55
3	38.73	38.79	38.75	38.59
4	38.83	39.04	38.72	38.78
Delta	0.31	0.60	0.16	0.30
Rank	2	1	4	3

Table IV illustrates the different levels of CC, MR, RT, and RTemp used in the experimental design. The Signal-to-Noise Ratio with Lombard (SNRL) indicates the average value of each parameter at its respective level and provides insight into its influence on biodiesel yield. The delta value, which indicates the range between the highest and lowest SNRL for each parameter, is used to rank their relative importance. A higher delta value indicates a greater effect on the response variable. CC (rank 1) is the first parameter that determines the yield of tung biodiesel in a dominant manner. This means that CC should be optimized to achieve the maximum efficiency of biodiesel production.

**C. Analysis of Variance**

The SNR serves as an indicator of the average yield of biodiesel in relation to its standard deviation and is useful in determining the optimal parameter levels and conditions to maximize biodiesel production. However, it cannot accurately predict the specific parameters that influence biodiesel yield in a given situation. Therefore, an ANOVA is used to evaluate the effect of various factors on tung biodiesel yield [16]. Table V presents various process variables along with their  $F$  and  $P$  values. Equation (5) was utilized to determine the percentage contribution of each variable. The contribution of important parameters in the preparation of tung biodiesel and its yield can be determined by higher  $F$  values. The  $F_{max}$  value (9.48) and the resulting  $P_{low}$  value (0.049) are of particular significance [17]:

$$\% \text{ Contribution factor} = \frac{SS_f}{SS_T} \times 100 \tag{5}$$

where  $SS_f = \sum_{j=1}^3 n[(SNR_L)_{fj} - SNR_T]^2$  is the sum of squares for the  $f$ -th parameter,  $SS_T = \sum_{i=1}^9 [SNR_i - SNR_T]^2$  is the total sum of squares for all parameters, and  $N$  refers to the number of experiments conducted at level  $J$  of factor  $f$  [18].

TABLE V. ANOVA FOR VARIOUS PROCESS PARAMETERS

Parameter	DoF	Adj sum of sq.	Adj mean sq.	F value	P value
MR	3	19.437	6.479	2.09	0.280
CC (wt %)	3	88.244	29.415	9.48	0.049
RTemp (°C)	3	5.492	1.831	0.59	0.662
RT(min)	3	24.816	8.272	2.67	0.221
Residual error	3	9.311	3.104		
Total	15	147.299			

**D. Signal-to-Noise Ratio**

Figures 3 and 4 present the variations in CC, MR, RT, and RTemp. The highest value for each factor is the optimal level for achieving maximum tung biodiesel yield. Therefore, an MR of 7:1, CC of 2.0 gm, RT of 30 min, and RTemp of 60°C result in the highest biodiesel yield for tung oil [19].

**E. Regression Equation**

The regression equation is an algebraic model that defines the relationship between input and output parameters. It provides results with minimal error and effectively captures the effects of variables. The regression equation is used to determine the optimum yield of tung biodiesel. By extracting

the optimum values from the SNR graphs for maximum yield and entering them into the regression equation, the optimum yield value is calculated to be 90.56% [20]:

$$\% \text{ Yield} = 72.47 + 0.834 \text{ Molar} + 4.098 \text{ Catalyst} + 0.0768 \text{ Temp} - 0.0111 \text{ Time} = 90.56 \quad (6)$$

The experimental conditions were determined based on the optimal values obtained from the SNR graphs of MR (6:1), CC, RT (60 min), and RTemp (60 °C), with the procedure being repeated three times to ensure accuracy. The average yield of the three experiments was found to be 91.59%, as shown in Table VI [21].

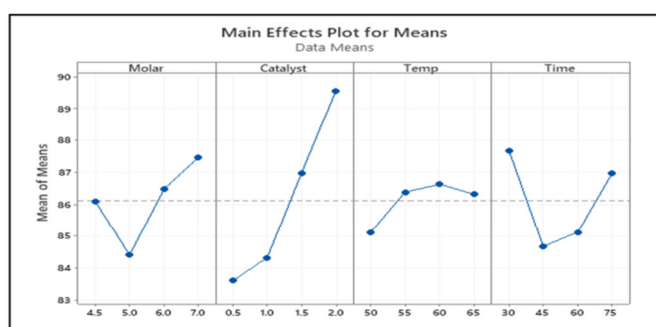


Fig. 3. Main effect plot for data means.

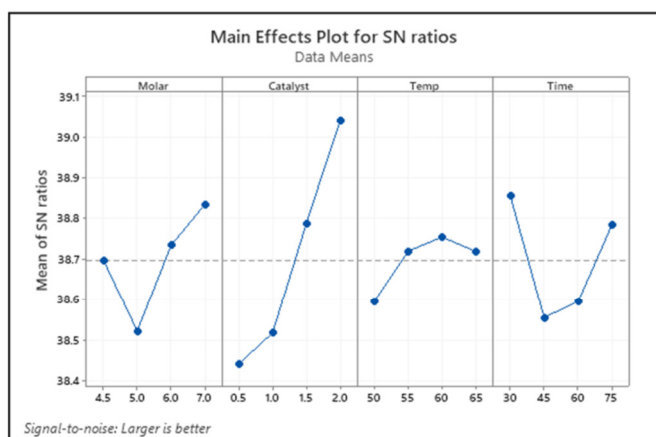


Fig. 4. Main effect plot for SNR.

TABLE VI. YIELD PERCENTAGE DERIVED FROM EXPERIMENTAL ANALYSIS

Trails	Yield (%)
1	91.15
2	92.26
3	91.38
Average	91.59

#### IV. CONCLUSIONS

The Taguchi optimization Design of Experiments (DOE) technique was used to improve the transesterification process for the production of biodiesel from tung oil. Four key process parameters - Molar Ratio (MR), Catalyst Concentration (CC), Reaction Time (RT), and Reaction Temperature (RTemp) -

were analyzed to maximize the yield of Tung biodiesel with minimal experimentation. The L16 orthogonal array and Analysis of Variance (ANOVA) were used to optimize these variables, resulting in proposed values of 6:1 MR, 2 gm CC, 30 min RT, and 60°C RTemp. The maximum yield obtained by the regression analysis was 90.59%, which closely matched the experimental yield of 91.38%, indicating an acceptable margin of error of 0.8%. The high R-squared value of 93.68% indicates a good fit of the model to the real data. The physicochemical properties of tung biodiesel were evaluated against standard diesel fuel, demonstrating similarity. The successful application of Taguchi optimization analysis with appropriate process variables underscores the potential of tung biodiesel as a viable alternative fuel for diesel engine applications, with an optimized parameter combination for maximum ester conversion rate identified by Signal-to-Noise (SN) analysis.

#### REFERENCES

- [1] A. A. Khaskheli, G. D. Walasai, A. S. Jamali, Q. B. Jamali, Z. A. Siyal, and A. Mengal, "Performance Evaluation of Locally-Produced Waste Cooking Oil Biodiesel with Conventional Diesel Fuel," *Engineering, Technology & Applied Science Research*, vol. 8, no. 6, pp. 3521–3524, Dec. 2018, <https://doi.org/10.48084/etasr.2333>.
- [2] M. Elkelay et al., "Study of diesel-biodiesel blends combustion and emission characteristics in a CI engine by adding nanoparticles of Mn (II) supramolecular complex," *Atmospheric Pollution Research*, vol. 11, no. 1, pp. 117–128, Jan. 2020, <https://doi.org/10.1016/j.apr.2019.09.021>.
- [3] V. Kumbhar, A. Pandey, C. R. Sonawane, A. S. El-Shafay, H. Panchal, and A. J. Chamkha, "Statistical analysis on prediction of biodiesel properties from its fatty acid composition," *Case Studies in Thermal Engineering*, vol. 30, Feb. 2022, Art. no. 101775, <https://doi.org/10.1016/j.csite.2022.101775>.
- [4] S. L. Gadhave and S. S. and Ragit, "Process optimization of Tung oil methyl ester (Vernicia fordii) using the Taguchi approach, and its fuel characterization," *Biofuels*, vol. 11, no. 1, pp. 49–55, Jan. 2020, <https://doi.org/10.1080/17597269.2017.1334441>.
- [5] D.-V. Manh, Y.-H. Chen, C.-C. Chang, C.-Y. Chang, C.-V. Minh, and H.-D. Hanh, "Parameter evaluation of biodiesel production from unblended and blended Tung oils via ultrasound-assisted process," *Journal of the Taiwan Institute of Chemical Engineers*, vol. 43, no. 3, pp. 368–373, May 2012, <https://doi.org/10.1016/j.jtice.2011.12.008>.
- [6] K. Y. Wong, J.-H. Ng, C. T. Chong, S. S. Lam, and W. T. Chong, "Pareto-hierarchical clustering framework for biodiesel transesterification," *Sustainable Energy Technologies and Assessments*, vol. 45, Jun. 2021, Art. no. 101160, <https://doi.org/10.1016/j.seta.2021.101160>.
- [7] A. Singh, S. Sinha, A. K. Choudhary, D. Sharma, H. Panchal, and K. K. Sadasivuni, "An experimental investigation of emission performance of heterogenous catalyst jatropa biodiesel using RSM," *Case Studies in Thermal Engineering*, vol. 25, Jun. 2021, Art. no. 100876, <https://doi.org/10.1016/j.csite.2021.100876>.
- [8] A. Thangthong et al., "Physicochemical properties of lard oil and rubber seed oil blends and their comprehensive characterization," *Chinese Journal of Chemical Engineering*, vol. 75, pp. 1–13, Nov. 2024, <https://doi.org/10.1016/j.cjche.2024.07.010>.
- [9] K. C. Goh et al., "Innovative circular bioeconomy and decarbonization approaches in palm oil waste management: A review," *Process Safety and Environmental Protection*, vol. 195, p. 106746, Mar. 2025, <https://doi.org/10.1016/j.psep.2024.12.127>.
- [10] S. Brahma et al., "Biodiesel production from mixed oils: A sustainable approach towards industrial biofuel production," *Chemical Engineering Journal Advances*, vol. 10, May 2022, Art. no. 100284, <https://doi.org/10.1016/j.cej.2022.100284>.
- [11] A. R. Patil and A. D. Desai, "Optimization of application of 2-ethyl-hexyl-nitrate on partial substitution of ethanol in CI engine for fuel economy and emission control using MADM method," *SN Applied*

- Sciences, vol. 1, no. 2, Jan. 2019, Art. no. 166, <https://doi.org/10.1007/s42452-019-0163-7>.
- [12] I. U. Khan, H. Chen, Z. Yan, and J. Chen, "Extraction and Quality Evaluation of Biodiesel from Six Familiar Non-Edible Plants Seeds," *Processes*, vol. 9, no. 5, May 2021, Art. no. 840, <https://doi.org/10.3390/pr9050840>.
- [13] A. Jankovic, G. Chaudhary, and F. Goia, "Designing the design of experiments (DOE) – An investigation on the influence of different factorial designs on the characterization of complex systems," *Energy and Buildings*, vol. 250, Nov. 2021, Art. no. 111298, <https://doi.org/10.1016/j.enbuild.2021.111298>.
- [14] G. K. B. S. Kumar, K. Rajesh, A. H. K. Sharma, S. Balachandran, and P. Gopinath, "Optimization of Biodiesel Production from Pongamia Oil using Taguchi Method," *International Journal of Engineering Research & Technology*, vol. 6, no. 4, Apr. 2018, <https://doi.org/10.17577/IJERTCONV6IS04025>.
- [15] A. Kolakoti, "Optimization of biodiesel production from waste cooking sunflower oil by taguchi and ann techniques," *Journal of Thermal Engineering*, vol. 6, no. 5, pp. 712–723, Oct. 2020, <https://doi.org/10.18186/thermal.796761>.
- [16] P. T. Kale and S. S. Ragit, "Optimization of Babassu (*Orbignya sp*) biodiesel Production from babassu oil by Taguchi Technique and Fuel Characterization," *International Journal of Petroleum Science and Technology*, vol. 11, no. 1, pp. 35–50, 2017.
- [17] D. V. Kadam and S. D. Jadhav, "Optimization of Vibration, Performance and Emission of C.I. Engine Operated on Simarouba Biodiesel Using Taguchi and Multiple Regression Analysis," SAE International, Warrendale, SAE Technical Paper, Sep. 2017. <https://doi.org/10.4271/2017-01-2137>.
- [18] S. Satya, A. Kolakoti, N. R. B., S. S. R., and R. Rao, "Optimization of palm methyl ester and its effect on fatty acid compositions and cetane number," *Mathematical Models in Engineering*, vol. 5, no. 1, pp. 25–34, Mar. 2019, <https://doi.org/10.21595/mme.2019.20469>.
- [19] G. Singh, Mohapatra ,Saroj Kumar, S. Ragit ,Satischandra, and K. Kundu, "Optimization of biodiesel production from grape seed oil using Taguchi's orthogonal array," *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, vol. 40, no. 18, pp. 2144–2153, Sep. 2018, <https://doi.org/10.1080/15567036.2018.1495778>.
- [20] N. F. Khanyi and F. Inambao, "Performance, Design and Operational Parameters of Variable Compression Ratio Diesel Engine," *Tuijin Jishu/Journal of Propulsion Technology*, vol. 45, no. 02, pp. 2943–2965, May 2024, <https://doi.org/10.52783/tjpt.v45.i02.6385>.
- [21] N. Sh. El-Gendy, S. F. Deriase, A. Hamdy, and R. I. Abdallah, "Statistical optimization of biodiesel production from sunflower waste cooking oil using basic heterogeneous biocatalyst prepared from eggshells," *Egyptian Journal of Petroleum*, vol. 24, no. 1, pp. 37–48, Mar. 2015, <https://doi.org/10.1016/j.ejpe.2015.02.004>.