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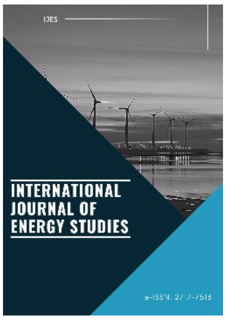

Research Article

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Optimization of ethyl ester production from linseed oil using the Taguchi method with an L16 orthogonal design matrix

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Highlights

- L16 Taguchi design: Novel approach for linseed oil ethyl ester.
- Optimal conditions: 1% NaOH, 10:1 ethanol:oil 75°C, and 60 minutes.
- Ethanol:oil ratio most influential (48.95%) among parameters.
- 95.20% actual yield, close to 95.98% theoretical yield.
- Promising eco-friendly alternative to conventional diesel.

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ABSTRACT

Renewable energy and its many forms, have been the focus of major interests because of their energy potential and environmental benefits, are now emerging as subjects that need more investigation, particularly in relation to biodiesel fuel. This is due to the uncertainty surrounding oil prices and emission regulations. The study included NaOH concentration (0.6, 0.8, 1, and 1.2 wt.%), ethanol:oil molar ratio (6:1, 8:1, 10:1, and 12:1), temperature of reaction (30, 45, 60, and 75 °C), and duration of reaction (30, 45, 60, and 75 min) as significant parameters influencing the yield of ethyl ester. For the first time, to the best of our knowledge, the L16 orthogonal design matrix of the Taguchi method approach was applied in the present research to optimize the transesterification step parameters from linseed oil. ANOVA validation studies determined the relative influence of the process parameters. A maximum biodiesel yield of 95.20% was obtained under optimum reaction conditions: 1 wt% NaOH, 10:1 ethanol:oil molar ratio, 75°C reaction temperature and 60 min reaction time. The highest contribution ranking of the four variables was 48.95% with the ethanol:oil molar ratio, 22.32% with NaOH loading, 18.24% with the temperature of the reaction, and 9.59% with the duration of the reaction. The fuel properties of synthesized linseed oil ethyl ester, at the specified ideal reaction conditions, were met the range of the standard EN14214.

Keywords: Linseed, Transesterification, L16 orthogonal design, Taguchi method, Ethyl ester, Fuel characteristics

1. INTRODUCTION

Efforts to transition the global energy landscape are growing, driven by concerns over reliance on fossil fuels. The use of these types of energy resources has critical effects on increasing air pollution, global warming, and greenhouse gas emissions. For this reason, a global effort has begun to emerge in recent years to research and create sustainable alternatives. Biodiesel, which is obtained from raw material sources such as algae oils, animal fats, waste oils, and vegetable oils and burns cleanly, has emerged as a solution in the quest for sustainable energy [1]. In this respect, it can be considered a viable and environmentally friendly option because it offers advantages over diesel fuel produced from petroleum. In addition to significantly reducing the release of pollutants such as particulate matter (PM), carbon monoxide (CO), and hydrocarbons (HC), it is biodegradable and non-toxic. It has a higher flash point, making it much safer during handling and transportation [2]. In addition, the carbon dioxide (CO_2) emitted during the combustion of biodiesel theoretically makes it a zero-carbon fuel, as it is fixed with the carbon dioxide absorbed as the plants used in the production process grow [3]. The presence of triglycerides in the oils or fats used as raw materials in their production can be converted into biodiesel [4]. To produce biodiesel, a process called transesterification is required, in which triglycerides in oils or fats are converted into methyl or ethyl esters by mixing them with alcohol such as methanol or ethanol and a catalyst. Although edible vegetable oils, including soybean, rapeseed, and palm oils, have been extensively used for biodiesel production, their use gives rise to concerns over food security and competition with food resources. Accordingly, there is an increasing interest in investigating non-edible oils, such as linseed oil obtained from the seeds of the lin plant, as alternative raw materials for producing biodiesel [1,5].

Linseed, scientifically classified as *Linum* L., is a type of herbaceous plant that grows annually and belongs to the Linaceae family [6]. With a rich history spanning centuries, this incredibly adaptable crop has made a profound impact on a wide range of industries, including textiles and pharmaceuticals [7-10]. In addition to its high oil content, the fact that it can be used for many purposes can be considered among the main reasons for growing linseed [11]. In the past, linseeds were grown extensively in the textile industry for the production of linen fibers, which were widely used in the production of clothing, home textiles, and canvas. However, in recent years, oil obtained from seeds has become more prominent, especially in the biofuel industry. It is known as a reservoir of abundant polyunsaturated fatty acids, especially alpha-linolenic acid [12,13].

The use of linseed oil as a feedstock for biodiesel production can differentiate it from its traditionally used rival in terms of providing various potential benefits [5]. First, linseed is widely farmed worldwide. This unlockable fact makes it easily accessible and plentiful [14]. Additionally, the relatively cheaper price of linseed oil compared to other types of vegetable oil makes it a financially viable choice over its peers for biodiesel production, thereby increasing the overall cost efficiency of the process [5]. Moreover, the distinctive fatty acid structure of linseed oil, characterized by a significant amount of alpha-linolenic acid, as well as its increased resistance to oxidation and improved performance at low temperatures, can give biodiesel produced from its advantageous properties [15-19]. It is also aligned with broader goals such as sustainability and efficient use of resources towards the use of linseed oil in biodiesel production. Using this inedible oil for biofuel production also contributes to reducing concerns about food safety, as food resources are not consumed. In addition, growing linseed can promote agricultural sustainability and improve soil health by helping diversify crops and rotate cropping [20]. Previous studies have demonstrated the viability of linseed oil for biodiesel production. Kumar et al. [21] achieved 88-96% triglyceride conversion through alkali-catalyzed transesterification, while Taherkhani et al. [2] obtained 82.15% biodiesel yield using KOH catalysis under optimized conditions. These studies established the potential of linseed oil while highlighting the need for process optimization to improve efficiency and economic viability. It can be noted that past studies have recognized the potential of linseed oil as feedstock for biodiesel production. The studies described examined different parts of this process, provided vital information, and also paved the way for future research. However, these studies also pointed out certain limitations and highlighted the need for optimization to increase the efficiency and economic viability of biodiesel produced using linseed oil. Also, an important detail emphasized in previous studies is some discrepancies between the yield and quality of biodiesel. This discrepancy may be related to several variables such as the catalyst and/or alcohol used, the conditions under which the reaction takes place (such as temperature, time, and molar ratios), and the presence of contaminants in the raw materials. Furthermore, aspects such as the energy consumed in certain steps required for production, such as the extraction and purification of the oil from the seed, have been pinpointed as areas that need to be improved to reduce production costs and thus the total environmental footprint. The use of novel approaches and experimental designs is necessary to improve the efficiency of biodiesel production from linseed oil by overcoming the negativity of these limitations.

Dr. Genichi Taguchi originated the Taguchi technique, a powerful statistical method that can optimize industrial processes [22]. With this technique, it is possible to identify the factors with the most important influence and the ideal parameters for them. This approach can be utilized especially when dealing with multiple factors and their interactions. It utilizes orthogonal arrays, which are specialized experimental designs that allow simultaneous evaluation of several factors with a minimum number of tests. It is centered on the concept of responsive design, which aims to reduce the impact of uncontrolled factors, known as noise factors, on the performance of a product or process. Through the use of orthogonal arrays, it can effectively reveal the most advantageous levels of factors and their interactions, achieving enhanced process performance, decreased variability, and cost savings. This methodology has been used effectively for a wide variety of purposes in sectors where process improvement is desired, such as machinery, chemistry, manufacturing, industry, engineering, etc., and has established its effectiveness as a valuable tool for process optimization [23,24]. Following in the footsteps of previous research and recognizing the need for further process optimization, the current study aims to make the most of the power of the Taguchi method to maximize the yield of biodiesel production from linseed oil. In particular, the goals are to determine the optimum combination of transesterification process parameters and factor levels that yield the highest yield of ethyl ester, as well as minimize the influence of uncontrollable factors to increase the efficiency and reduce the cost of the process from start to finish. It was chosen as the L16 orthogonal array of the Taguchi technique, an experimental design framework that allows many factors and their interactions to be studied simultaneously with fewer experimental runs. For the current study, the selection of factors and their respective levels was carried out carefully based on their importance in the transesterification reaction and their potential impact on the yield of biodiesel.

An important factor to consider is the molar ratio of ethanol to oil, which can vary from 3:1 to 15:1. [25]. The ratios mentioned here are extremely important in determining the progress of the reaction and realizing an efficient conversion of triglycerides to ethyl esters. Catalyst concentration (0.5–1.5% w/w) also significantly affects transesterification by influencing reaction kinetics and yield [26]. Reaction temperature (25–75°C) and time (30–180 minutes) directly impact the conversion rate, with higher temperatures and longer times generally increasing biodiesel yield, though improper adjustments can lead to side reactions or product degradation [27-30]. The rate of the transesterification process is directly influenced by these factors. In general, higher temperatures and longer reaction times tend to contribute to a greater increase in the conversion rate to biodiesel. However, if the necessary adjustments are not made properly, unwanted side reactions or degradation of the product may occur. This study employs an L16 orthogonal array to optimize reaction conditions for maximizing ethyl ester yield from linseed oil while minimizing uncontrollable factors like impurities. Previous studies highlight similar optimization efforts. Danish et al. [31] achieved a 98.6% biodiesel yield using FCCD with optimal methanol-to-oil ratio (5.9:1), catalyst (0.51%), temperature (59.2°C), and reaction time (33 minutes). Etim et al. [32] reported a 96.5% yield using bio-alkaline salt as a catalyst, with reaction time being the most critical factor. Ahmad et al. [33] used FCCD to predict a 99.5% yield, experimentally achieving 98% under optimized conditions. Mandal et al. [34] optimized methanolysis of low-FFA flaxseed oil, achieving a 94% yield at a 6:1 molar ratio, 1% KOH, and 45 minutes.

Optimizing the factors involved in the transesterification reaction plays a rather key role due to their talent to increase the yield return of the biodiesel production process. Furthermore, optimization is necessary both to increase the overall efficiency of the process and to reduce feedstock use and waste generation. It also leads to cost savings and promotes environmental sustainability. Furthermore, fine-tuning the reaction conditions as well as the alcohol and catalyst concentration often has a significant impact in ensuring that the resulting biodiesel meets the required criteria for fuel quality [30,33,35]. Viscosity, density, flash point, and oxidative stability are important factors that affect how well biodiesel performs and whether it is compatible with current engine technology [36,37]. By determining the most favorable configurations, the resultant biodiesel may meet the specified criteria, hence improving its commercial feasibility and acceptability in the market. It has the potential to boost the oxidative stability, low-temperature performance, and lubricity of the fuel, all of which are important qualities for a high-quality product. Furthermore, using ethanol as the alcohol in the transesterification procedure provides further advantages. Since ethanol is a renewable and more environmentally friendly alcohol made from agricultural feedstocks like corn, sugarcane, or cellulosic biomass, as opposed to methanol, which is usually derived from non-renewable fossil fuel sources, it can have a positive impact on the production of biodiesel [38-40]. Moreover, the use of sodium hydroxide (NaOH) as the catalyst is beneficial since it is readily accessible, inexpensive, and very efficient in promoting the transesterification process [41].

In this research, the Taguchi technique, especially using the L16 orthogonal design matrix, is used to optimize the key factors that impact the ethyl ester. These parameters include the concentration of the catalyst (NaOH), the molar ratio of ethanol to oil, the reaction temperature, and the reaction duration. The purpose of this systematic method is to determine the most effective combination of these characteristics, in order to obtain the maximum possible yield in ethyl ester production while reducing the need for extensive experimentation. Current research presents a novel approach to biodiesel production by utilizing linseed oil as a feedstock. It contributes to the ongoing pursuit of alternative and sustainable biofuel sources. Furthermore, this study is the first, to our knowledge, to employ a combined linseed oil and ethanol system for biodiesel production, utilizing the L16 orthogonal design matrix to optimize the process. This technique allows the concurrent assessment of various components and their interactions, leading to a thorough comprehension of the dynamics of the process and allowing the identification of the most relevant parameters. Furthermore, this research includes an assessment of the fuel characteristics of the generated biodiesel, offering significant knowledge on the excellence and appropriateness of linseed oil-based biodiesel for real-world uses. The analysis of fuel properties, such as density, viscosity, flash point, and cetane number, will contribute to the existing knowledge base and facilitate the assessment of linseed oil as a viable raw material for biodiesel production. The results of this research are anticipated to enhance the existing knowledge in the area of biodiesel production, particularly from non-edible oil sources. The study findings provide significant reference for future studies and have the potential to assist in the development of efficient and cost-effective techniques for biodiesel synthesis using linseed oil as a feedstock. Moreover, the procedure used in this work may be modified and utilized to enhance biodiesel synthesis from other non-edible oil sources, hence broadening the range of sustainable biofuel research.

2. MATERIALS AND METHODS

The present section provides an overview of the materials and experimental methods used in the current investigation. The transesterification method was enhanced for the generation of ethyl ester from linseed oil utilizing the Taguchi technique, a reliable approach. A comprehensive description is provided for the materials used, the experimental design used, the methodology used for the biodiesel generation technique, and the fuel property.

2.1. Materials

The priority in the selection of chemicals and other materials required in the biodiesel synthesis process and analysis is their easy availability. The research study took place utilizing substances of analytical-grade quality. Ethanol (C_2H_5OH, 99.9% purity) and sodium hydroxide (NaOH, 99.5% purity) were used in their native analytical-grade condition without further purification, as they met the required specifications for the transesterification process. Ethanol with a minimum purity of 99.9% was procured from Isolab Chemicals, Eschau, Germany. The feedstock for biodiesel manufacturing was produced by cold pressing linseed oil, which was acquired from a local market in Istanbul. The cold-pressing method was specifically chosen to preserve the oil's natural properties and prevent thermal degradation during extraction, ensuring optimal quality for biodiesel production. The transesterification process used sodium hydroxide (NaOH) pellets as the catalyst. NaOH pellets with a minimum purity of 99.5% were supplied by Chemsolute, a trademark of Th Geyer, Renningen, Germany. For the purification processes, 125-mm filter paper was supplied by S&H Labware (Türkiye). The acid value was determined using a 0.1 N potassium hydroxide (KOH) solution and a phenolphthalein indicator obtained from Norateks Chemical Company in Istanbul, Türkiye. Pure water, acquired from a Millipore Direct-Q 8 UV system, was used for the wash. Ancillary equipment, such as magnetic stirring bars, beakers, glasses, volumetric flasks, separating funnels, spoons, etc., required for the experiments, was purchased from a reliable domestic supplier with EN ISO 17025 certifications and ISO9001 Quality-Management, ensuring quality and reliability.

2.2. Experimental Design (Taguchi Method)

The use of design of experiments (DOE) has the benefit of minimizing the total number of experimental investigations that need to be optimized. In this scenario, it will not only reduce the use of raw materials and chemicals but also save energy, workforce, and time. Finding the optimal combination that we took into consideration in the experimental investigation and seeing its impacts on the performance variables using a statistical approach included in the DOE is another crucial step. The Taguchi approach focuses on analyzing a limited number of variable variations in a manufacturing process without requiring the evaluation of all performance metrics. The statistical analyses of the experiments undertaken using the Taguchi technique were performed using the Minitab 21 program. The Taguchi approach, a resilient statistical tool, was used to optimize the transesterification process parameters to maximize the ethyl ester output derived from linseed oil. This approach contributes to minimizing the number of experimental runs needed and has the key advantage of supporting the investigation of multiple factors simultaneously. The Taguchi technique studies the effects of several variables and their interactions on the response variable(s) by using orthogonal arrays, which are specifically constructed experimental matrices.

In this study, the “bigger-is-better” Signal-to-Noise (SNR) ratio was used as the optimization criterion within the Taguchi Method framework. This approach is preferred for studies where maximizing a response variable is desired; here, in a similar manner, the aim is to maximize the efficiency of the biodiesel production process. This SNR ratio was used to evaluate how different control factors such as NaOH concentration and ethanol-oil ratio affect the output. Focusing on maximizing this ratio, the experiments determined the most favorable conditions leading to the highest efficiency and productivity. Four important variables have been defined to have the potential to affect the ethyl ester output in the present study: sodium hydroxide (NaOH) concentration (A), ethanol: oil molar ratio (B), temperature of reaction (C), and reaction duration (D). The levels of these factors, shown in Table 1, were selected based on preliminary experiments and literature reviews. Initial tests were carried out varying the NaOH concentration from 0.5% to 2.0% to observe its effect on the reaction yield. It was noted that higher concentrations did not provide a proportional benefit and even increased costs. Based on these observed data, the range was limited from 0.8% to 1.5%, as these values provide an optimal balance between reaction yield and efficiency.

Table 1. Variables studied and selected levels of each

Variables	Levels			
	1	2	3	4
A	0.6	0.8	1.0	1.2
B	6	8	10	12
C	30	45	60	75
D	30	45	60	75

The L16 orthogonal array design, which requires only 16 experimental runs rather than 256 (4^4) runs, was chosen because it allows the evaluation of four variables with four levels each [42]. This design, presented in Table 2, not only minimizes the number of tests required, but also provides information on the effects of each variable on ethyl ester yield as a result of their interaction with other variables.

Table 2. L16 orthogonal array design

Run no.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
A	1	1	1	1	2	2	2	2	3	3	3	3	4	4	4	4
B	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
C	1	2	3	4	2	1	4	3	3	4	1	2	4	3	2	1
D	1	2	3	4	3	4	1	2	4	3	2	1	2	1	4	3

All test runs were randomized to mitigate the impact of extraneous variables and experimental inaccuracies. The ethyl ester yield that was selected as the response variable was measured for each test run. The data were analyzed utilizing the SNR ratio methodology of the Taguchi method. The SNR ratio quantifies the resilience of the process by taking into account both the average and the variability of the response. This investigation utilized the larger-the-better SNR ratio to top out the ethyl ester yield as shown in the equation below. In Equation SNR=-10 $\log_{10}\left(\sum\frac{1/y^2}{n}\right)$ (1), y is the biodiesel yield, which is the observed value of the quality characteristic and n is the number of experiments [43].

$$SNR = -10\ log_{10}\left(\sum\frac{1/y^2}{n}\right) \tag{1}$$

The analysis of variance (ANOVA) was conducted to assess the statistical significance and percentage contributions of the main effects of the variables and their interactions among themselves [44]. The optimal levels of the variables were determined by selecting the greatest SNR ratio values. Additionally, confirmatory experiments were performed to verify the optimized conditions. The outcomes of the Taguchi optimization were further examined and discussed in more detail under later headings.

2.3. Biodiesel Production Process

The current investigation included the implementation of a single-step transesterification procedure to generate biodiesel from linseed oil. To achieve this goal, a compact biodiesel reactor shown in Figure 1 was used. To carry out this process, a 250 ml three-necked flat-bottom (TNFB) bottle was placed on the magnetic stirrer with a heated bottom, as shown in the figure. A funnel was fixed to the left of the neck of the bottle, and a thermometer was attached to the right neck. To avoid the evaporation of alcohol, a reflux condenser that uses water for cooling was positioned on the middle neck.

Figure 1. A compact biodiesel reactor for the transesterification process

To achieve an effective transesterification process and enhanced separation of biodiesel and glycerol layers in the reactor in the figure above, it is important to keep the FFA concentration in crude oil below 2%, thereby minimizing soap production [45]. A process utilizing acid-base titration was applied to ascertain FFA content of the linseed oil that is being investigated. Since the FFA content of linseed oil was identified as 1.2%, a one-step transesterification process was preferred. The ranges of the chosen variables A, B, C, and D were defined according to the findings of previous research and the qualities of the oil. For example, the boiling point of ethanol limits the reaction temperature. The factors listed above were considered in the present research to optimize the procedure of transesterification for the production of linseed oil ethyl ester (LOEE) with the maximum yield percentage.

The following is a summary of the one-stage transesterification process, which is illustrated step by step in Figure 2: First, 100 g of linseed oil was added to the TNFB batch reactor, and the temperature was increased to the required level. The rotational speed of the stirrer was set at a constant value of 600 revolutions per minute (rpm) for all experimental trials. The recalibrated amount of NaOH base was dissolved in the right amount of alcohol to form the ethanol-NaOH mixture. The ethoxide solution that was created was introduced to the reactor in a progressive manner using a funnel, adding approximately 50 mL per minute to ensure uniform mixing and prevent localized reactions, after the linseed oil had achieved the required temperature. A digital timer was started immediately after the complete addition of the ethoxide solution to precisely monitor the reaction time. After the designated time of the reaction, the contents of the reactor were transferred into the funnel for separation. A valve was used to remove the glycerol that had collected on the bottom layer from the splitter hopper after it had settled for eight hours. Following settling, two separate layers were formed: a dark brown glycerol layer on the bottom and a lighter biodiesel layer on top. The bottom glycerol layer was carefully drained through the valve at a controlled flow rate of approximately 25 mL/min until the line between the layers was reached, ensuring complete separation of the glycerol from the biodiesel. The unrefined biodiesel was thereafter poured into a glass beaker made of high-temperature resistant material and subjected to a temperature of 85°C to remove any surplus ethanol present in the biodiesel. Prior to commencing the washing procedure, the temperature was reduced to 55°C. To avoid either the distilled water heating up suddenly or the crude biodiesel cooling down suddenly, the temperature was kept fixed. To eliminate any remaining catalyst, soap, and other contaminants, the product was subjected to a washing process using distilled water that constituted 20% of its volume, all while maintaining the same temperature. The purified biodiesel was raised to 120 °C to start the dewatering process after waste water was removed from the funnel with the help of the valve. Ultimately, the ethyl ester was stored in a glass jar in a darkened room to settle and lower its temperature. After reaching room temperature, measurement was carried out with a precision scale.

To increase the clarity of the above-mentioned ethyl ester production from linseed oil by transesterification process, the diagram is presented in Figure 2.

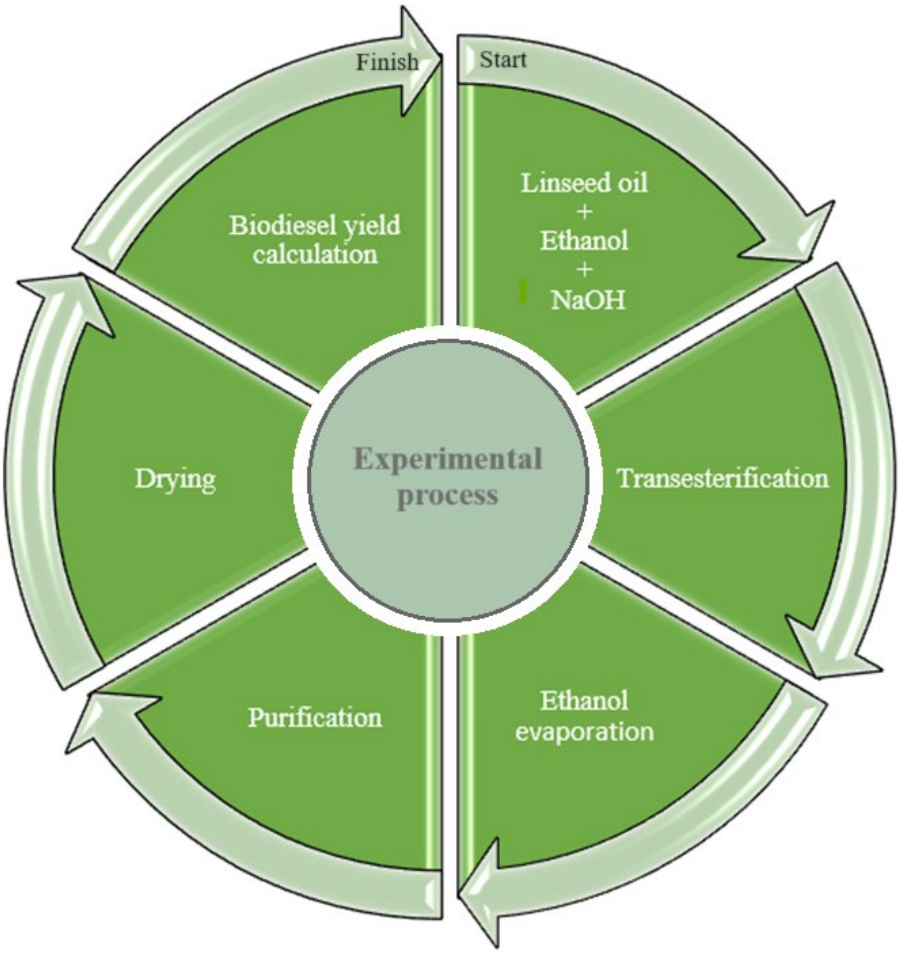


Figure 2. The experimental process utilized to acquire ethyl ester from linseed oil and to determine the yield

2.4. Fuel Property Analysis

Following the realization of the synthesis of ethyl ester from linseed oil under optimized reaction conditions of the transesterification procedure considering the results of the Taguchi method, many of the important physical and chemical properties were tested in line with the EN test methods. Subsequently, the results were benchmarked against the international biodiesel standard EN14214 to reach a conclusion. The tested properties include density, kinematic viscosity, flash point, cloud point, pour point, cold filter plugging point, acid value, iodine value, cetane number, ash content, water content, oxidation stability, and higher heating value, which are critical for assessing the suitability of linseed oil ethyl ester as a biodiesel fuel. Each property tested is directly linked to biodiesel quality. For example, kinematic viscosity affects fuel flow and atomization in engines, density influences combustion performance, acid value provides an indication of fuel stability and cetane number defines ignition quality, etc. Fuel properties were tested following standard methodologies such as the EN14214 protocol to ensure accurate and reliable measurements. For example, kinematic viscosity was determined at 40°C using a viscometer and density at 15°C using a hydrometer.

2.5. Fatty Acid Composition and Elemental Analysis

Elemental analysis and fatty acid composition tests were carried out in the Occupational and Environmental Toxicology Laboratory at the Science and Technology Application and Research Center of Yozgat Bozok University. An inductively coupled plasma-mass spectrometer (ICAPQC) from Thermo Scientific, USA, was utilized to measure trace elements and heavy metals in linseed oil biodiesel. The setting of the experimental conditions was as described below: spray chamber temperature 3.7°C, dwell time 0.01 ms, nebulizer pressure 3.01 bar, plasma gas 0.88 L/min, nebulizer gas 0.96 L/min, and radiofrequency power 1550 W. The steps of clearing the sampler probe between injections were as follows: (i) churned with ultrapure water for 30 seconds; (ii) decontamination with 2% HNO_3 for 50 seconds; and (iii) washed with ultrapure water for 50 seconds. Each sample was tested 3 times following the standards to ensure the validity of the outputs. Each repetition of all metal analyses was performed with the sample in a single run. A sample of 0.5 g. was digested in Teflon vessels with 10 mL suprapure HNO_3 in a microwave oven (Milestone D5, USA). After cooling, the clear supernatant was transferred to polypropylene tubes and diluted to 20 mL with deionized water.

3. RESULTS AND DISCUSSION

3.1. Optimization of Process Parameters

The yield and SNR results for each linseed oil ethyl ester obtained by running the sixteen experiments determined for the L16 experimental run are reported in Table 3. In order to increase the experimental validity of each experiment performed under the same conditions, the run was repeated three times and the average of these runs was accepted as the result. The results recorded that the highest LOEE yield and SNR were 93.22% and 39.39% in run 10 and the lowest LOEE yield and SNR were 85.20% and 38.61% in run 1.

Table 3. Results of LOEE yields and SNR ratios

Run no.	A	B	C	D	LOEE yield (%)	SNR
1	0.6	6	30	30	85.20	38.61
2	0.6	8	45	45	86.85	38.78
3	0.6	10	60	60	91.97	39.27
4	0.6	12	75	75	90.64	39.15
5	0.8	6	45	60	86.39	38.73
6	0.8	8	30	75	87.18	38.81
7	0.8	10	75	30	91.81	39.26
8	0.8	12	60	45	90.73	39.16
9	1.0	6	60	75	88.80	38.97
10	1.0	8	75	60	93.22	39.39
11	1.0	10	30	45	92.55	39.33
12	1.0	12	45	30	90.88	39.17
13	1.2	6	75	45	90.50	39.13
14	1.2	8	60	30	88.65	38.95
15	1.2	10	45	75	91.11	39.19
16	1.2	12	30	60	92.63	39.34
Overall mean					89.944	39.08

While the above Table indicates that the parameter set of run 10 corresponds to the highest yield of ethyl ester, it is not in fact the optimal choice. For this reason, the SNR level (SNR_L) was taken into pay regard to achieve the true optimal set of reaction parameters. SNR_L values corresponding to each level were calculated and recorded in Table 4. The calculation of SNR_L values, for example, for NaOH concentration (A), was executed as follows: To calculate the SNR_L value to be written in the Level 1 section of A in Table 4, the average of the SNR values corresponding to the value of 0.6 (wt.%) in Table 3 (38.61, 38.78, 39.27, and 39.15) was taken and the obtained value (38.95) was written in Table 4. Similarly, the same process was repeated for Levels 2, 3, and 4. Then, the SNR_L values corresponding to the Levels of the parameters B, C, and D were also created in this way. Afterwards, the mean values of each Level were computed and transferred to the Table. For example, the Means values for the temperature of the reaction (C) were derived as below: To generate the Means value for Level 3 of C in Table 4, the Means values at 60 °C in

Table 3 (91.97, 90.73 88.80, and 88.65) were averaged and the resulting value (90.04) was added to Table 4. The same procedure was applied for Levels 2, 3, and 4. Means values for Levels B, C, and D parameters were then generated in the same method.

For the Delta line in Table 4, the difference between the highest SNR_L value and the lowest SNR_L value of each parameter is evaluated. The method applied is as follows; for example, the highest SNR_L value of 39.21 in section A is subtracted from the lowest SNR_L value of 38.95 to get a Delta value of 0.26. After that, the Rank values were passed to Table 4 in descending order, with the highest Delta value being Rank 1. The ranking line was created by determining the variables with the most and least significant effect on the efficiency of LOEE. In this context, when Table 4 is analyzed, it can be observed that the molar ratio of ethanol to oil (1st rank) and reaction time (4th rank) are the highest and lowest effective factors on the ethyl ester production efficiency. In addition, reaction temperature and NaOH concentration were the second (2nd rank) and third (3rd rank) effective parameters.

Table 4. Main effects for Means and SNR_L

	A		B		C		D	
Level	SNR_L	Means	SNR_L	Means	SNR_L	Means	SNR_L	Means
1	38.95	88.66	38.86	87.72	39.02	89.39	39.00	89.13
2	38.99	89.03	38.98	88.97	38.97	88.81	39.10	90.16
3	39.21	91.36	39.26	91.86	39.09	90.04	39.18	91.05
4	39.15	90.72	39.20	91.22	39.23	91.54	39.03	89.43
Delta	0.26	2.70	0.40	4.14	0.27	2.73	0.18	1.92
Rank	3		1		2		4	

Figure 3 provides insight into the order of influence on the yield of LOEE of SNR_L variations at 4 different levels of each of the 4 selected factors in the transesterification reaction. For the peak SNRL value visible in each of the 4 frames in the figure, it can be said to have the most significant effect on the LOEE yield. Also, the peak SNRL value in each frame corresponds to the optimal operating conditions on LOEE for that particular factor. Therefore, the factors that maximize the percentage transformation yield of LOEE can be listed as follows: 1% (wt.) at level 3 for NaOH concentration, 10 mol/mol at level 3 for ethanol: oil molar ratio, 75 °C at level 4 for the temperature of reaction, and 60 minutes at level 3 for the duration of the reaction.

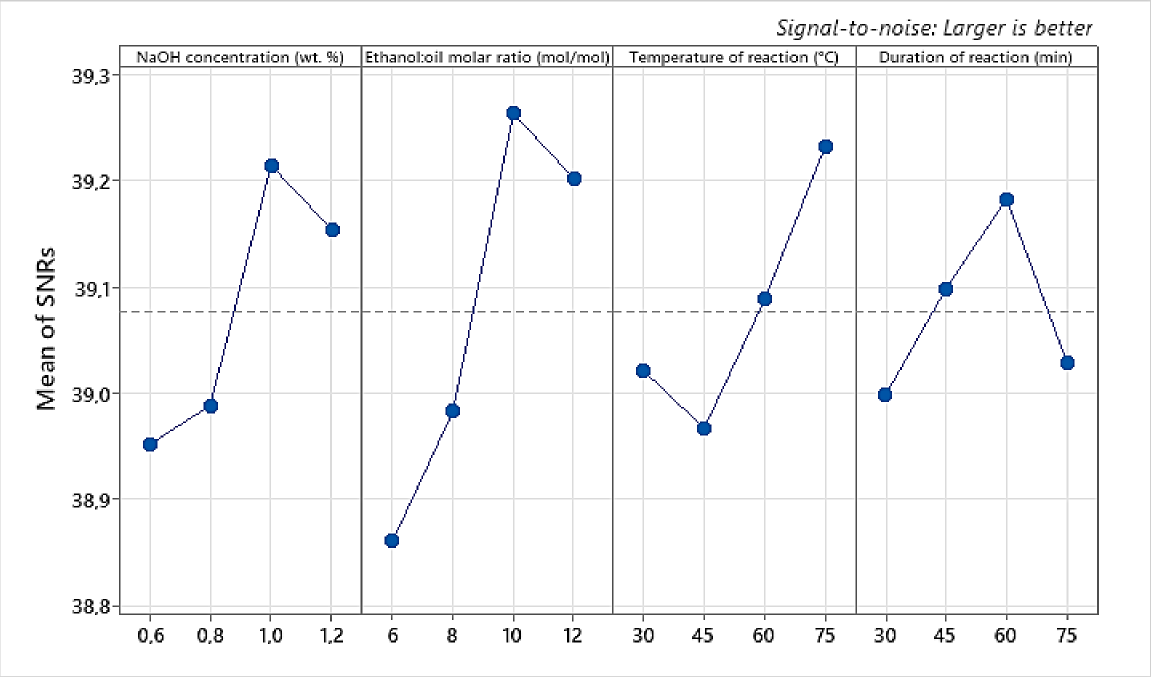
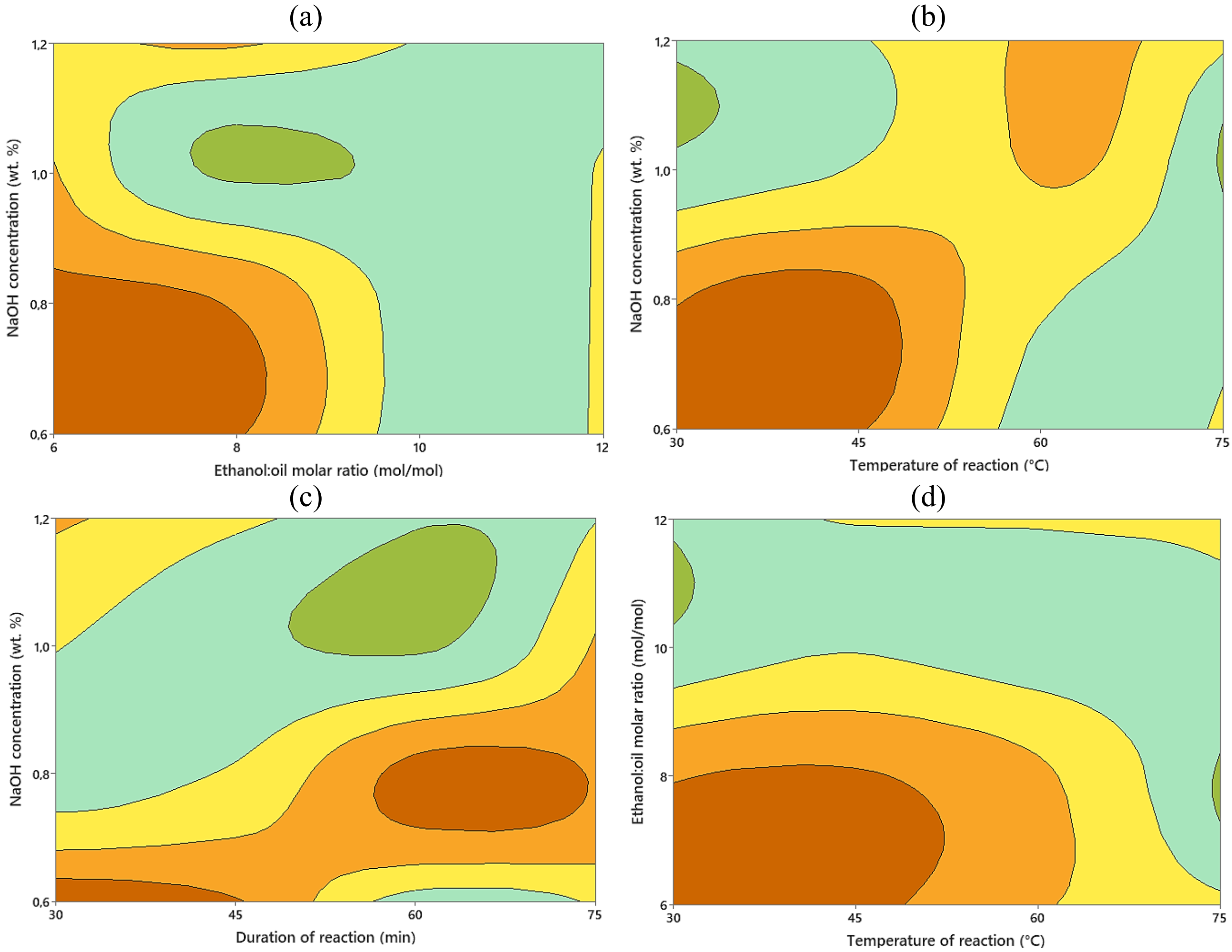


Figure 3. SNR$_L$ values for each factor at various levels

The effects of NaOH concentration (0.60 wt.%, 0.80 wt.%, 1.00 wt.%, and 1.20 wt.%), ethanol: oil molar ratio (6:1, 8:1, 10:1, and 12:1), temperature of reaction (30 °C, 45 °C, 60 °C, and 75 °C), and duration of reaction (30 min, 45 min, 60 min, and 75 min) on the transformation of LOEE are illustrated in Figure 4. In the existing research, the goal is to raise the LOEE transformation efficiency by using the Taguchi technique, which is a statistical technique, i.e. the focus is on maximizing biodiesel yield. The transformation yields of the added oil to the ethyl ester increased up to 1% NaOH loading and 10 mol/mol ethanol: oil molar ratio and reached the peak value. For higher values, especially for higher catalyst loading, the LOEE yield decreased because of the increased tendency of soap formation. When the effect of catalyst loading was evaluated, it was observed that the concentration of NaOH was the dominant factor in the yield of LOEE production. On the contrary, an increase in NaOH concentration by more than 1.00 wt.% led to an immediately noticeable reduction in LOEE transformation yield. According to general opinion, besides the increase in NaOH loading (e.g. increasing to 1.2% by wt.), an increase in soap formation can be observed depending on the FFA content of the added oil and thus the LOEE yield also decreases. Another aspect related to this that reduces the ester efficiency is that it's difficult to separate glycerin from the ester. To increase the conversion efficiency in ethyl ester formation, a critical balance must be maintained in the transesterification reaction [46]. Increases in the ethanol: oil molar ratio (above 10 mol/mol) and the duration of the reaction (above 60 min) above a certain value led to an increase of the glycerin percentage in the samples. This caused the glycerin to foaming more, a phenomenon that leads to less ester transformation. Although the increase in the reaction temperature from 30 °C to 45 °C seems to negatively affect the ester yield, the main reason for this phenomenon is the low values of NaOH concentration and ethanol: oil molar ratio chosen for those temperature of the reaction. Furthermore, raising the temperature of the reaction from 45 °C to 75 °C had an undeniable effect on the transesterification method, corresponding to an increase in conversion efficiency to ethyl ester. The maximum temperature used in the research (75 °C) provided the best transformation efficiency. Since the boiling point of ethanol added at the ester production stage is 78.3 °C, the maximum temperature of the reaction in the tests was determined as 75 °C. If the temperature of the reaction is set very near, at the same or higher than the boiling point of ethanol, the alcohol will evaporate before the ester conversion is completed, and in this case, the triglycerides in the raw material will trigger the formation of soap and thus the ethyl ester yield will decrease [47].


(a)
(b)
(c)
(d)

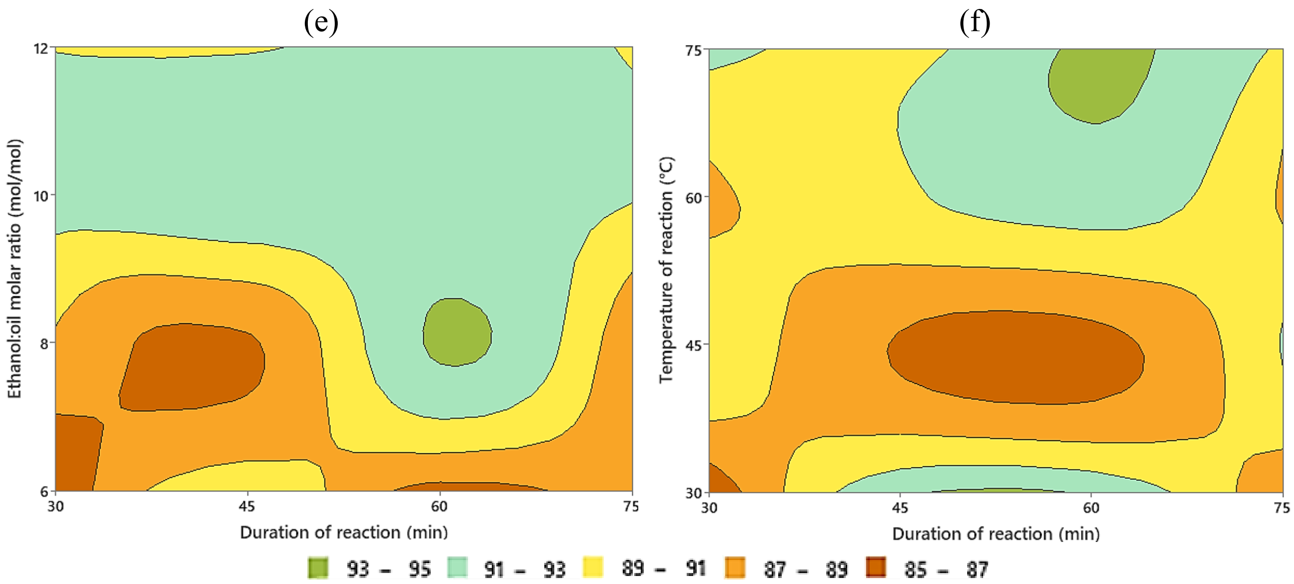


Figure 4. Contour Plots of (a) A vs. B, (b) A vs. C, (c) A vs. D, (d) B vs. C, (e) B vs. D, and (f) C vs. D

3.2. Analysis of Variance (ANOVA)

Transesterification to produce ethyl esters from linseed oil was conducted using the L16 orthogonal array approach. LOEE efficiency was evaluated as a performance characteristic. After each run, the data were statistically analyzed through ANOVA results of the transesterification process, tabulated in Tables Table and Table , to predict the impacts of all process factors on response and the significance of the model. All four factors (A, B, C, and D) are statistically "significant" as expressed in Table 5 because the P-value associated with each of them is smaller than 0.05. The factors can be ranked as follows according to their F-values, which give the relative importance of each factor: B (F-value: 54.31), A (F-value: 24.76), C (F-value: 20.23), and D (F-value: 10.65). Since the model explains 99.10% of the variance in the data (R-sq value), it can be stated that it shows a very good fit. The adjusted R-square (95.49%) and the estimated R-square (74.36%) are also quite high. These high values point to predictive ability and good model performance. The residual has a relatively small sum of squares (0.8229) compared to the other four factors, suggesting that most of the variation in the data is explained by the factors in the model. High F-values and low P-values for all factors, together with high R-sq values, indicate that the model is valid [48]. Furthermore, thanks to these values, it is concluded that all factors are important in explaining the variation in LOEE efficiency.

Table 5. Percentage contribution of the operating parameters

Factors	Degrees of Freedom (DF)	Sum of Squares (SS)	Mean Square (MS)	Contribution Factor (%)	F-Value	P-Value	
A	3	20.3761	6.7920	22.32	24.76	0.013	Significant
B	3	44.6930	14.8977	48.95	54.31	0.004	
C	3	16.6500	5.5500	18.24	20.23	0.017	
D	3	8.7619	2.9206	9.59	10.65	0.042	
Residual	3	0.8229	0.2743	0.90			
Total	15	91.3038	24.8846	100.00			

In Table 6, the root mean square error (RMSE) is entered as 0.523727. This value represents the standard error of the regression, also known as the standard error of the prediction. The coefficient of variance (CV) is often expressed as a percentage and represents the ratio of the RMSE to the mean of LOEE yields. This low CV (0.58%) indicates that there is relatively low variability in the data compared to the mean, suggesting consistent results across the experiments.

Table 6. Model summary

RMSE	Mean of LOEE yields (%)	CV (%)	R-squared (%)	R-squared (adjusted) (%)	R-squared (predicted) (%)
0.523727	89.944	0.58	99.10%	95.49%	74.36%

3.3. Validation of Optimized Conditions

The contribution factor data of the process parameters tested in the current research on LOEE efficiency, expressed as a percentage, are tabulated in Table 5. As can be seen, the largest contribution of LOEE on conversion efficiency is B with 48.95%, followed by A with 22.32%, C with 18.24%, and D with 9.59%. The theoretical conversion efficiency for LOEE was found to be 95.9844% by using the highest coefficient for each factor and substituting the factor coefficients in Table 7 in Equation (2), or in other words, by using the Taguchi technique in the optimum reaction variables. A careful look at Table 7 shows that the theoretical efficiency with the highest percentage can be found by including the highest value of each factor next to the 89.944% constant in Equation (2). According to the regression equation, the coefficient values to be summed with 89.944% can be listed as follows; 1.418% (coefficient of NaOH concentration), 1.916%

(coefficient of Ethanol: oil molar ratio), 0.809% (coefficient of reaction temperature) and 1.108% (coefficient of reaction time). The result of this process will give the best LOEE yield of 95.9844% according to the analysis. This is the experiment with the factors corresponding to the highest coefficients used for this process, i.e. the optimum conditions, which is 1% (wt.) NaOH concentration, ethanol: oil molar ratio of 10 mol/mol, temperature of reaction of 75 °C and reaction time of 60 minutes. The highest LOEE yield was theoretically determined by Minitab Statistical Software using the coefficients in Table 7 using the input data. Accordingly, three replicate experiments were conducted under real-world conditions using the highest setting of the residual operating variables. LOEE efficiencies were measured as 94.89%, 95.46%, and 95.25% in these three experiments, respectively. The results of these three experiments were then averaged to obtain a value of 95.20%. The agreement between the predicted and experimental values can be observed from the negligible difference between them. This negligible difference between the two values could be attributed to the unpredictable influence of the parameters [49].

Table 7. Coefficients

Term	Coefficient	T-Value	P-Value
Constant	89.944	686.96	0.000
NaOH concentration (wt. %)			
0.6	-1.279	-5.64	0.011
0.8	-0.917	-4.04	0.027
1.0	1.418	6.25	0.008
Ethanol: oil molar ratio (mol/mol)			
6	-2.222	-9.80	0.002
8	-0.969	-4.27	0.024
10	1.916	8.45	0.003
Temperature of reaction (°C)			
30	-0.554	-2.44	0.092
45	-1.137	-5.01	0.015
60	0.093	0.41	0.709
Duration of reaction (min)			
30	-0.809	-3.57	0.038
45	0.213	0.94	0.417

60	1.108	4.89	0.016

$$
\begin{aligned}
\text{LOEE yield (\%)} = 89.944 &- 1.279 \ \text{NaOH concentration (wt. \%)\_0.6} \qquad (2)\\
&- 0.917 \ \text{NaOH concentration (wt. \%)\_0.8}\\
&+ 1.418 \ \text{NaOH concentration (wt. \%)\_1.0}\\
&+ 0.778 \ \text{NaOH concentration (wt. \%)\_1.2}\\
&- 2.222 \ \text{Ethanol:oil molar ratio (mol/mol)\_6}\\
&- 0.969 \ \text{Ethanol:oil molar ratio (mol/mol)\_8}\\
&+ 1.916 \ \text{Ethanol:oil molar ratio (mol/mol)\_10}\\
&+ 1.276 \ \text{Ethanol:oil molar ratio (mol/mol)\_12}\\
&- 0.554 \ \text{Temperature of reaction (°C)\_30}\\
&- 1.137 \ \text{Temperature of reaction (°C)\_45}\\
&+ 0.093 \ \text{Temperature of reaction (°C)\_60}\\
&+ 1.598 \ \text{Temperature of reaction (°C)\_75}\\
&- 0.809 \ \text{Duration of reaction (min)\_30}\\
&+ 0.213 \ \text{Duration of reaction (min)\_45}\\
&+ 1.108 \ \text{Duration of reaction (min)\_60}\\
&- 0.512 \ \text{Duration of reaction (min)\_75}
\end{aligned}
$$

3.4. Fuel Properties of Linseed Ethyl Ester

The outputs showing the main physicochemical properties of linseed oil-derived biodiesel are tabulated in Table 8 below. As mentioned in the paragraphs above, three experiments were repeated under optimum conditions, resulting in an average LOEE efficiency of 95.20%. Therefore, the basic fuel properties of the LOEE with an efficiency of 95.25%, which is the closest to this efficiency value in the three trials, were analyzed. For the present research, some key fuel properties of ethyl ester from linseed oil were determined, which include ash content, acid number, water content, cloud point, higher heating value, pour point, kinematic viscosity, cetane number, density, etc. and are listed in the Table below. In addition, the physicochemical properties of a few of the properties mentioned in the previous sentence and its continuation in Table 8 have been tested by following the global biodiesel standards and most of them are in compliance with the standard ranges given in the table. Most of the fuel characteristics measured for LOEE produced under optimal conditions meet the standards, indicating that LOEE can be an ultimate substitute for conventional fuel for CI engine fueling.

Table 8. The fuel properties of linseed oil ethyl ester

Fuel properties	Units	ASTM D6751	EN14214	Linseed oil ethyl ester
Chemical formula	-	-	-	$C_{17.49}H_{30.58}O_2$
Higher heating value	kJ/kg	-	-	38,385
Density (at 15 °C)	kg/m^3	880	860-890	872
Kinematic viscosity (at 40 °C)	mm^2/s	1.9-6.0	3.5-5.0	3.19
Flash point	°C	Min. 93	Min. 101	>120
Cloud point	°C	-3 to -12	-	-1.92
Pour point	°C	-15 to -16	-	-9.60
Cold filter plugging point	°C	Max. 5	-	-4.81
Copper strip corrosion (3 h at 50°C)	Degree of corrosion	Class 3	Class 1	1a
Iodine value	g I_2/100 g	-	Max. 120	186.89
Acid value	mg KOH/g	Max. 0.5	Max. 0.5	0.38
Cetane number	-	Min. 47	Min. 51	31.40
Saponification number	mg KOH/g	Max. 370	-	201.03
Oxidation stability	hour	Min. 3	Min. 8	12.79
Ash content	% (m/m)	0.02 max	0.02 max	0.01
Water	wt. %	Max. 0.05	Max. 0.05	<0.05
Carbon	wt. %	77	-	77.03
Hydrogen	wt. %	12	-	11.22
Oxygen	wt. %	11	-	11.68
Odor	-	-	-	Agreeable
Color	-	-	-	Yellow Orange
Polyunsaturated (>= 4 double bonds) ethyl ester	% (m/m)	-	Max. 1	0
Linolenic acid ethyl ester	% (m/m)	-	Max. 12	1.76

3.5. Elemental Analysis of Optimized Ethyl Ester

The data on the elemental analysis of ethyl ester obtained from linseed oil are summarized in Table 9. Phosphorous (P), manganese (Mn), iron (Fe), and zinc (Zu) were undetected in linseed ethyl ester, and the concentrations of chromium (Cr), cobalt (Co), nickel (Ni), and copper (Cu) were recorded to be less than 1 ppm, as reported in the Table. However, the most abundant element in the ethyl ester is aluminum (Al) with 18.65 ppm, closely behind sodium (Na) with 11.40 ppm.

Table 9. The basic elemental analysis findings for LOEE

Na (ppm)	Mg (ppm)	Al (ppm)	K (ppm)	Ca (ppm)	Cr (ppm)	Co (ppm)	Ni (ppm)	Cu (ppm)
11.40	1.97	18.65	6.13	8.56	0.11	0.01	0.56	0.03

4. CONCLUSIONS

Through these extensive research studies and analyses, the L16 orthogonal design of the Taguchi design for the optimization of the most critical parameters consisting of catalyst (NaOH) concentration (0.6 wt.%, 0.8 wt.%, 1.0 wt.%, and 1.2 wt.%), ethanol:oil molar ratio (6:1, 8:1, 10:1, and 12:1), temperature of reaction (30 °C, 45 °C, 60 °C, and 75 °C), and duration of reaction (30 min, 45 min, 60 min, and 75 min) in the production of ethyl ester from linseed oil has been first applied and introduced to the literature. Until this research, the optimization of the operating variables for the production of new biodiesel from linseed oil has been investigated with the L9 design of the Taguchi technique, usually with KOH catalyst and methanol, although linseed oil has been applied in the preparation of ethyl ester in several articles. Remarkable conclusions based on the findings are summarized below, item by item:

- The optimal test conditions required to maximize ethyl ester efficiency through the transesterification process were defined by the Taguchi method as 1% NaOH concentration, 10:1 ethanol:oil molar ratio, 75 °C temperature of the reaction, and 60 minutes duration of the reaction.

- The highest contribution factor of 48.95% was recorded for the ethanol:oil molar ratio. It was next closely followed by NaOH concentration with 22.32% and the temperature of the reaction with 18.24%, respectively. The least effective contribution factor was the duration of the reaction with 9.59%.

- With the Taguchi technique involving the L16 orthogonal array, the percentage theoretical efficiency of LOEE under the optimal conditions of reaction was estimated to be 95.98%.

- In order to verify the theoretical yield, experiments were carried out in three replicates under the conditions stated in the previous paragraphs and it was concluded that the average ethyl ester efficiency of LOEE was 95.20%, which is very similar to the theoretical yield of 95.98%.

- The fuel specifications were examined and it was confirmed that all but a few of the specifications met the international standard requirements.

For the aforementioned reasons, the current research can conclude that linseed oil ethyl ester is a cost-effective, eco-friendly, and highly efficient alternative with substitution potential for CI engines, due to its potential to reduce the energy crisis that conventional diesel fuel can cause by alleviating ecological deterioration. Furthermore, to the authors' knowledge, there is no focused literature on the production of ethyl ester from linseed oil at optimal conditions identified in the current research, and also no research article on the optimal conditions for the production of ethyl ester from linseed oil utilizing the Taguchi method's L16 orthogonal set, indicating the need for more research on the investigated feedstock. In addition, the ethyl ester could be investigated for other fuel characteristics (such as free and total glycerol, total impurity, and ester content) which were not determined in the current study. Moreover, thermodynamic and kinetic investigations could be carried out in the production of ethyl ester from linseed oil. Finally, blends of pure LOEE with conventional diesel fuel could be investigated in a CI engine at constant engine speed, maximum power or torque rpm, or different load conditions to determine exhaust pollutants, combustion behavior, and engine performance.

DECLARATION OF ETHICAL STANDARDS

The author of the paper submitted declares that nothing which is necessary for achieving the paper requires ethical committee and/or legal-special permissions.

CONTRIBUTION OF THE AUTHORS

Zeki Yılbaşı: The experimental studies, analysis, and writing of the manuscript in this study were carried out entirely by the author.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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