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Optimization of Process Parameters for Experimental Production of Waste Frying Oil Based-Biodiesel by Taguchi Method

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Abstract: Recycling waste frying oils is an important issue concerning environmental and economic problems. Biodiesel production from waste oil is an effective method to obtain a sustainable energy source. Biodiesel attracts attention as a renewable and environmentally friendly alternative that can replace traditional fossil fuels in diesel engines. However, various process parameters such as process temperature, alcohol/oil ratio, and type of catalyst need to be carefully adjusted to increase the efficiency and optimize the quality of the biodiesel production process from waste oils. This study uses the Taguchi method to determine the most suitable process parameters for experimental biodiesel production from waste oils. The study's findings, which identified the optimum conditions for biodiesel production from waste frying oil, have the potential to impact the field of sustainable energy significantly. Three essential parameters were selected for investigation: the molar ratio of alcohol to oil, reaction temperature, and reaction time. Each parameter was examined at two levels, denoted as L-4 (23). Four experimental runs were conducted using the selected parameters and their respective levels to produce biodiesel from waste frying oil. Optimum conditions were found to be 1:6 for oil/methanol molar ratio, 60°C for reaction temperature, and 60 min for reaction time. Under optimum reaction conditions, biodiesel yield was an average of 97.7 %.

Keywords: Biodiesel, Waste frying oil, Taguchi, Design of experiment

Introduction

The rise in population and higher food consumption rates has led to a surge in waste frying oil (WFO) from various sources such as households, restaurants, hotels, schools, and industries. This issue is not just a local concern but a global one, increasingly prevalent in the TRNC (Turkish Republic of North Cyprus) and beyond. Due to its island status, Cyprus faces a particularly severe situation compared to mainland areas. Improper disposal of WFO through sinks and drains poses significant challenges for sewage treatment facilities, consequently driving up purification expenses. Indeed, WFO represents a valuable residue that holds potential as a raw material for various purposes such as soap manufacturing, energy generation through anaerobic digestion, thermal cracking, and biodiesel fuel production (Phan & Phan, 2008; Sabudak & Yıldız, 2010; Al-Shanableh et. al., 2023).

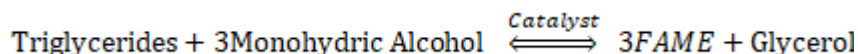
Biodiesel stands out as a popular alternative in liquid fuels, primarily due to its compatibility with conventional diesel engines with minimal or no adjustments required and its ability to be blended with petroleum diesel. Traditionally, Biodiesel has been produced from renewable sources such as edible or non-edible oils/fats (Al-Shanableh, 2017). The conversion of highly viscous oils/fats into less viscous biodiesel typically occurs through transesterification. In this process, which can be catalyzed by a base, acid, or enzyme, oil/fat reacts with an alcohol to produce fatty acid methyl esters (FAME), i.e., biodiesel, along with glycerol as a co-product with commercial value. Among the various transesterification methods, base-catalyzed transesterification (Encinar et

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al., 2002) is regarded as the most promising approach for viscosity reduction. In this process, triglycerides undergo a transesterification reaction, converting them into a mixture of esters of the fatty acid in the oil/fat, utilizing a short-chain alcohol and a catalyst. Biodiesel is derived by purifying this mixture of fatty acid methyl esters. The following general equation can represent the transesterification reaction:



Design of experiment (DOE) methods were used to determine the essential process parameters effective in the production of biodiesel from vegetable oils, such as catalyst type, alcohol type, reaction time, and reaction temperature (Priyadarshi & Paul, 2019; Yesilyurt & Cesur, 2022). The Taguchi method aims to evaluate the impact of individual parameters on processes (Taguchi & Konishi, 1987). Taguchi techniques of DOE are used to optimize various engineering applications, evaluating the most suitable and optimal condition of process parameters (Al-Shanableh et al., 2020). The method employs a set of orthogonal arrays that lead to a minimum number of experimental trials.

This study uses the Taguchi method to determine the most suitable process parameters for experimental biodiesel production from waste oils. Three parameters were selected for investigation: the molar ratio of alcohol to oil, reaction temperature, and reaction time. Taguchi orthogonal array analyzed each control parameter and their individual effects on the whole process. Each parameter was examined at two levels, denoted as L-4 (2³). Four experimental runs were conducted using the selected parameters and their respective levels to produce biodiesel from waste frying oil. Produced biodiesel samples were evaluated for quality parameters using standard analytical methods.

Materials and Methods

Materials

The waste frying oil was sourced from the cafeterias of Near East University, where approximately 18 to 20 liters of WFO were gathered daily per cafeteria. Anhydrous methanol (MeOH) with a purity of 99.8% and high-purity sodium hydroxide (NaOH) were procured from Merck for the experiments. The feedstock's fatty acid (FA) compositions were analyzed according to the EN ISO 5508 method at the TRNC Ministry of Health, Directorate State Laboratory in Nicosia, utilizing Gas Chromatography (GC). The findings from the GC analysis are presented in Table 1.

Table 1. Fatty acid compositions of WFO

Fatty acid	Molecular mass (g/mol)	% Composition of fatty acids
Caprylic acid - C8:0	144.21	0.05
Capric acid - C10:0	172.27	0.33
Lauric acid - C12:0	200.32	1.18
Myristic acid - C14:0	228.38	0.10
Palmitic acid - C16:0	256.43	39.29
Palmitoleic acid –	254.41	0.14
Stearic acid - C18:0	284.48	4.04
Oleic acid - C18:1	282.47	40.42
Linoleic acid - C18:2	280.45	13.84
Linolenic acid - C18:3	278.44	0.18

Experimental Set-up for Base-Catalyzed Transesterification

Figure 1 outlines the sequence of experimental steps which were employed to produce biodiesel through a base-catalyzed one-step transesterification reaction. While transesterification constitutes the primary phase in biodiesel production, adhering to international standards requires additional procedures such as raw material pretreatment, separation of reaction products, and purification of the resultant products.

The characteristics of used frying oils are distinct from those of virgin oils. During the frying process, triglycerides undergo hydrolysis due to the combination of heat and water, increasing the free fatty acid (FFA) content. For successful base-catalyzed transesterification, the FFA levels in a sample should be below 3% (Sharma & Singh, 2009). Triglycerides with high FFA and water content required additional preprocessing steps, such as filtration and water removal. The amount of catalyst that should be used in the process is defined by titrating WFO samples against a standardized base solution such as NaOH and calculating the amount of FFA in the oil samples with Equation 1 (Wrolstad et al., 2005).

$$\% \text{ FFA as oleic acid} = \frac{(V_{\text{NaOH}})(N_{\text{NaOH}})(MW_{\text{oleic acid}})}{\text{Weight of sample}} \quad (1)$$

where, V_{NaOH} is the volume of NaOH consumed in titration and N_{NaOH} is the normality of NaOH solution. % FFA found in WFO was 2.2 %, so base-catalyzed transesterification was suitable for WFO.

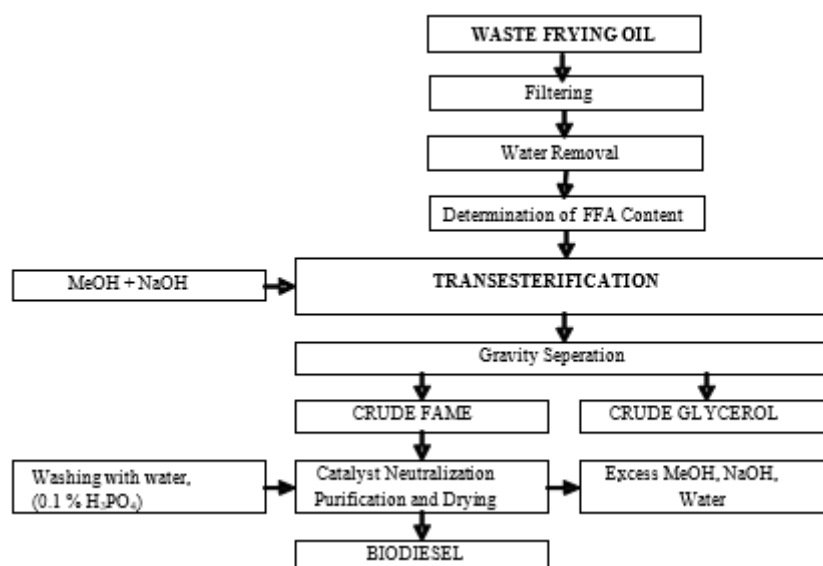


Figure 1. Flowchart of the experimental procedure for base-catalyzed transesterification

The reaction mixture was combined in a 2.0-liter three-necked flask equipped with a condenser, a magnetic stirrer, a T-type thermocouple, and a heater. The temperature of the transesterification reaction was either 50°C or 60°C. The molar ratio of methanol/oil was at two different levels of 1:4 or 1:6. The reaction time was fixed at 60 or 90 minutes. After the reaction was completed at the end of the specified time, separation and purification stages were utilized.

Design of Experiment by the Taguchi Method

The methodology devised by applying the Taguchi method to optimize process parameters for achieving the highest biodiesel yield via base-catalyzed transesterification is depicted in Figure 2 and can be readily followed. Three key control parameters were used to construct the Taguchi structure: the molar ratio of alcohol to oil, reaction temperature, and reaction time. Kim et al. (2010) and Buasri et al. (2009) worked on some other parameters, such as catalyst/alcohol type and catalyst concentration, which were utilized as control parameters, while those parameters were maintained at constant levels in this study. The three selected parameters with two levels denoted as L-4 (2^3), are listed in Table 2. Here, L-4 denotes a Latin square design with the replication number for the experiment.

Table 2. Parameters and their levels for DOE

Parameters	Levels	
	1	2
Molar ratio (oil/methanol)	1:4	1:6
Reaction temperature (°C)	50	60
Reaction time (min)	60	90

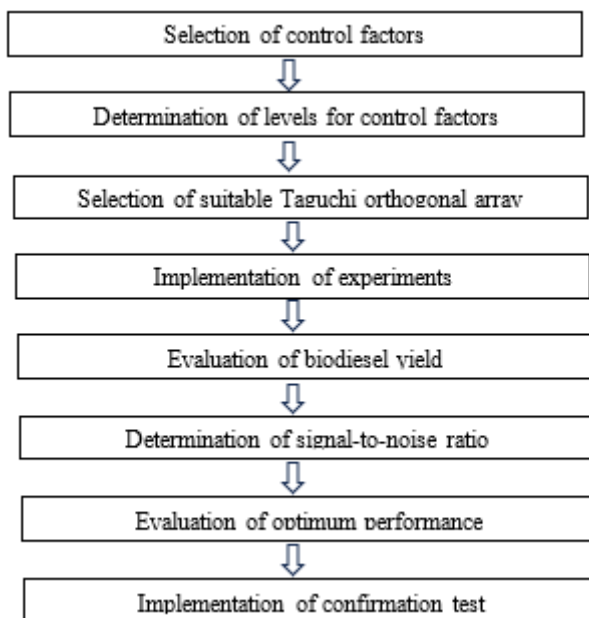


Figure 2. Taguchi's DOE procedure

To assess the impact of various control parameters on the efficiency of biodiesel production, an orthogonal array design was implemented with the experimental conditions outlined in Table 3, where numerical values represent the different levels of the parameters.

Table 3. The L-4 (23) orthogonal array for DOE of the current work

Experiment No	Control parameters and their levels		
	Molar ratio (oil/methanol)	Reaction temp. (°C)	Reaction time (min)
1	1	1	1
2	1	2	2
3	2	1	2
4	2	2	1

Using three independent variables, each with two levels would typically require 8 runs (i.e., 2³). However, by employing Taguchi's DOE, the number of experiments can be reduced to 4.

Results and Discussion

The percent yield of WFO-based biodiesel (WFOME) produced was calculated using Equation 2 (Phan and Phan, 2008) and results are shown in Table 4 as % yield.

$$\% \text{ Yield} = \frac{m_{\text{ester}}}{3 \times \frac{m_{\text{oil}}}{MW_{\text{oil}}} \times MW_{\text{ester}}} \quad (2)$$

The mass percentage of fatty acids in the feedstock and fatty acid methyl esters in biodiesel produced were obtained by GC analysis.

The yields of waste frying oil methyl esters (WFOME) obtained under four different experimental conditions are presented in Table 4. All experiments were conducted three times according to the specified conditions of control parameters outlined in Table 3. Experiment Number 4 exhibited the highest mean yield of biodiesel production, reaching 97.7%, indicating it as the optimal experimental condition. Conversely, Experiment Number 1 yielded the lowest biodiesel output at 72.4%. As per Taguchi's recommendation, achieving the optimal conditions based solely on the mean yield of biodiesel produced is insufficient. It is also imperative to determine the signal-to-noise (S/N) ratio to evaluate the quality characteristics deviating from the desired value.

The S/N ratio was calculated using the 'Larger-the-best' approach based on following Equation 3. Findings are listed also in Table 4.

$$\frac{S}{N} \text{ ratio} = -10 \log(MSD) \tag{3}$$

Where *MSD* is mean squared deviation and can be calculated as,

$$MSD = \frac{1}{n} \sum_{i=1}^n \left(\frac{1}{y_i}\right)^2 \tag{4}$$

where *n* is the number of repetitions of each experiment and *y_i* is the yield of biodiesel produced.

Table 4. WFOME yields and the S/N ratios in the four sets of experiments

Experiment no.	Yields of WFOME produced			Mean	S/N Ratio
	1 st trial	2 nd trial	3 rd trial		
1	69.4	72.4	75.5	72.4	37.183
2	72.4	73.5	78.8	74.9	37.472
3	78.6	86.6	92.2	85.8	38.613
4	97.2	97.8	98.0	97.7	39.795

The control parameters from Experiment Number 4 could be considered optimal, as they yielded the highest mean biodiesel yield and the most significant S/N ratio. The mean S/N ratio can be utilized to display the effects of each level for every parameter. Each experimental parameter should be evaluated individually, and the interactions at the assigned levels should be determined by averaging all the S/N ratios. For example, considering the oil/methanol molar ratio and its first level (1:4), the mean S/N ratio (37.328) can be computed using the values (37.183 and 37.472) obtained from Experiment numbers 1 and 2. Conversely, when its second level (1.6) is being analyzed, the mean S/N ratio (39.204) can be calculated using the values (38.613 and 39.795) from Experiment number 3 and 4, and so forth. The mean S/N ratio for each level of the three influential parameters is summarized in Table 5.

Table 5. The mean S/N ratio of the three influential parameters

Parameters	S/N Ratio	
	Level 1	Level 2
Molar ratio (oil/methanol)	37.328	39.204
Reaction temperature (°C)	37.898	38.634
Reaction time (min)	38.489	38.043

The mean S/N ratio gave a perception about which parameter should be used at which level to achieve the highest biodiesel yield. The magnitude of the S/N ratio also defined the importance of control parameters. For this research, the molar ratio of alcohol to oil had the most crucial influence on process yield and was followed by reaction temperature and the last one was reaction time. The optimal reaction conditions based on the highest S/N ratio are as follows: for the first parameter (oil/methanol molar ratio), at level 2 (1:6); for the second parameter (reaction temperature), at level 2 (60°C); and for the third parameter (reaction time), at level 1 (60 min).

Biodiesel production from refined (unused) cooking oil (RCO) was conducted under optimized conditions to validate their effectiveness. The yield of refined cooking oil methyl esters (RCOME) was determined to be 98.1%. This yield closely resembles waste frying oil methyl esters (WFOME), produced using the optimal conditions of a 1:6 oil/methanol molar ratio, 60°C reaction temperature, and 60 minutes reaction time.

Conclusion

This study used the Taguchi method to determine the most suitable process parameters for experimental biodiesel production from waste oils. Optimum conditions found due to Taguchi's DOE were a 1:6 oil/methanol molar ratio, 60°C as reaction temperature, and 60 min as reaction time. The results constitute a critical step in optimizing the biodiesel production process from waste oils and producing environmentally friendly energy sources more effectively. Future studies can focus on validating the findings on a broader range of parameters and further improving the biodiesel production process.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPHELS Journal belongs to the authors.

Acknowledgements or Notes

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