

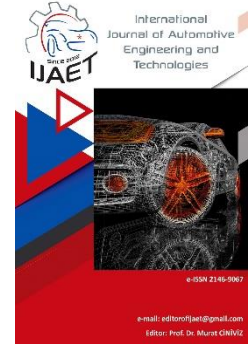


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Original Research Article

Biodiesel production from waste frying oil by electrochemical method using stainless steel electrode



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ABSTRACT

Biodiesel production from waste frying oil is important in terms of effectively utilizing waste and reducing production costs. It is important that the production method of biodiesel is environmentally friendly, economical, and sustainable. For this purpose, electrochemical transesterification process with stainless steel electrodes instead of rare metal electrodes was preferred in this study for biodiesel production. In this study, where SS304 class AISI 304 stainless steel was used as an electrode, biodiesel was produced from waste frying oil by electrochemical method. An electrolyte was prepared with 8:1 molar ratio of methanol, 2% by weight distilled water, 2% THF, 0.5% NaCl and waste frying oil. The conversion efficiency and fuel properties of biodiesel produced in the electrochemical process lasting 3 hours with a reaction voltage of 20 V were determined. The results were compared with biodiesel produced by conventional methods. According to the results, a conversion efficiency of 68% was achieved in the electrochemical process. Moreover, it has been determined that biodiesel properties are compatible with EN 14214.

Keywords: Biodiesel, Waste frying oil, Electrochemical method, Conventional method, Steel electrode.

1. Introduction

Today, humankind mostly meets the energy it needs from fossil sources. Reasons such as the increasing effect of fossil fuels on global warming and the rapid depletion of their resources have increased the tendency towards renewable alternative energy sources [1]. Biofuels are preferred as an alternative energy source in internal combustion engines. Biodiesel is used as an alternative to diesel in compression ignition (CI) engines. Vegetable

and animal oils and waste frying oils are preferred in biodiesel production. Waste frying oil is considered as a biodiesel raw material source due to reasons such as the environmental damage it causes, the decrease in fossil fuel resources, and the increase in diesel fuel prices. Four basic techniques are used to reduce the high viscosity of oils and ensure their use in the engine. These are direct use and blending, microemulsions (co-solvent mixing), thermal cracking (pyrolysis) and

transesterification (alcolysis) [2]. Transesterification is the most used method to reduce the viscosity of animal and vegetable oils. This biodiesel production technique has been reported to have certain advantages over other processes. These; easy reaction conditions, environmentally friendly process, and the ability to process a wide variety of raw materials [3, 4].

Transesterification is the esterification reaction of fatty acids with alcohol in the presence of a basic catalyst. When triglycerides in oil react with alcohol during the transesterification process, it forms a mixture of fatty acid esters (biodiesel) and glycerin. In the literature, the chemical reaction process in the transesterification method is commonly carried out in three different ways: conventional, microwave and ultrasonic [5]. However, research on biodiesel production by electrochemical method has increased recently. This method is also known as electrolysis and electrolytic transesterification in the literature. Electrochemical method is a synthesis method in which low voltage is applied with anode and cathode electrodes in aqueous solutions. Guan and Kusakabe [6] are the first researchers to use this method in the

production of biodiesel from waste frying oil. They found that it exhibited a high fatty acid methyl ester (FAME) yield (>97%) even when a low sodium concentration was used in the presence of a relatively high-distilled water content [6]. The variables and results of some studies in the literature on the production of biodiesel from different raw materials by electrochemical method are summarized in Table 1. As can be seen in Table 1, the type of electrode used in electrochemical biodiesel production is very effective in biodiesel conversion efficiency. It has been observed that graphite and titanium electrodes provide higher conversion efficiency compared to the use of steel electrodes. However, stainless steel electrodes stand out due to reasons such as being resistant to corrosion, not reacting with electrolyte, being a good conductor, and being able to withstand high temperature and voltage. Additionally, low-cost stainless-steel electrodes can provide better production rate of H₂ and O₂ gas during the reaction [15, 16]. In terms of conversion efficiency in the transesterification process, it was observed that the methanol/oil ratio (molar) and reaction voltage were inversely proportional, as seen in Table 1.

Table 1. Literature summary of studies on biodiesel production by electrochemical method

Raw materials (oil)	Alcohol type and ratio (molar)	Catalyst type and ratio (by weight)	Solvent type and ratio	Distilled water ratio (by weight)	Electrode type	Distance between electrodes	Reaction voltage (V)	Reaction time and temperature	Con. Eff. (%)	Ref.
Waste frying oil	Methanol 6:1	KOH 0.5%	Acetone 10%	0.2%	Graphite (20mm×20 mm×1mm)	10 mm	50 V	2 h Ambient temperature	96%	[7]
Corn oil	Methanol 24:1	NaCl 0.56%	THF/methanol (0.25)	%2	Platinum plate (20mm×20 mm)	12 mm	18.6 V	2 h Ambient temperature	97%	[8]
Soybean	Methanol 24:1	Cholinechloride/Ethylene Glycol synthesis (1:2 molar) 7%	none	1.5%	Platinum plate (60mm×30 mm)	10 mm	21 V	2 h Ambient temperature	95%	[9]
Waste frying oil	Methanol 6:1	NaOH ¹ KOH ² 1%	Acetone ¹ THF ² MTBE ³ 10%	2%	Graphite (22mm×22 mm)	10 mm	40 V	2 h Ambient temperature	98% ¹ 98% ² 98% ³	[10]
Waste frying oil	Methanol 12:1	Iron-containing CaO	Acetone, 20% NaCl, 0.6%	2%	Graphite (20mm×20 mm*5mm)	10 mm	29 V	50 min.	92%	[11]
Waste frying oil	Methanol 6:1	Phosphomolybdic acid/graphene oxide 0.85%	THF 0.5:1 (molar)	2%	Graphite (20mm×20 mm*5mm)	10 mm	60 V	15 h Ambient temperature	90.3%	[12]
Waste frying oil	Methanol 9:1	Phosphomolybdic acid/clinoptilolite 3%	Nail polish 0.5:1 (molar)	2%	Graphite (22mm×22 mm*10mm)	10 mm	21V	4 h Ambient temperature	96%	[13]
Canola oil	Methanol 10:1	NaCl 0.3%	none	1%	Stainless steel (20mm x 20mm)	30 mm	20V	5 h 40°C	62%	[14]

It seems that the reaction voltage must be increased in order to achieve high conversion efficiencies at low alcohol ratios. Moreover, the rate of distilled water used in the electrochemical method was generally 2% of the electrolyte. It is observed that TDH and acetone are generally used as solvent types. It is observed that the solvent ratio increased to a maximum of 3% and is generally used at low levels. It has been observed that NaCl and CaO are used as catalysts in biodiesel production, unlike traditional and lower-cost products such as NaOH and KOH. It is found that the distance between electrodes is mostly 10 mm. It is observed that the reaction time is generally between 2 and 5 hours.

Today, due to the high cost of platinum, one of the rare elements, and the low impact resistance of graphite, it is important to investigate the use of SS304 class AISI 304 stainless steel electrodes in biodiesel production. Although the conversion efficiency is low, stainless-steel electrodes was preferred to reduce the production cost. In this article, which investigates biodiesel production with easily accessible and low-cost steel electrode, it is aimed to determine the effect of electrochemical production on fuel properties in the production of biodiesel from waste frying oil. The use of steel electrodes in electrochemical biodiesel production stands out in terms of low-cost production in order to increase efficiency in the biodiesel production process and ensure sustainable production. Although biodiesel production with high conversion efficiency can be achieved with titanium and graphite electrodes, steel electrodes attract attention with their durability and long life. Reducing energy costs in the biodiesel production process reduces environmental impact. When the literature was examined, it was determined that the biodiesel production of electrochemical methods from waste frying oil with steel electrodes was limited. This study aims to produce biodiesel from waste frying oil using a low-cost electrochemical production method.

2. Materials and Methods

In this study, biodiesel production was made with domestic waste frying oil. High purity (99.7%) methanol for transesterification

processes, potassium hydroxide (KOH), tetrahydrofuran (THF), sodium chloride (NaCl) and distilled water were used. A schematic view of the preferred electrochemical method for the transesterification process is given in Fig.1.

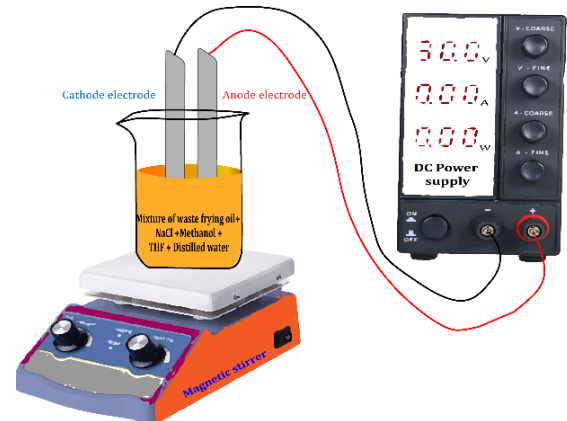


Fig.1. Schematic view of the transesterification method with the electrochemical method

Before biodiesel production, the waste frying oil was filtered, and the free fatty acid was measured as 0.82%. Free fatty acid in terms of oleic acid was determined according to EN ISO 660 standard. Since the free fatty acid is below 1%, there is no need for any pre-treatment to reduce the acid value.

In the production process of biodiesel by electrochemical method, the electrode material that directs the electric current and transmits it into the electrolyte liquid has an important role. In this process, the electrodes must provide a surface where chemical reactions occur for biodiesel production as well as facilitate electron transfer [17]. Moreover, the presence of free electrons in the atomic structure of the electrode material used affects the magnitude and speed of the current given to the electrolyte during the reaction. Factors such as the surface structure of the electrode material, surface area, and surface roughness also affect the reaction rate [17–19]. Steel electrodes may be preferred due to their cost and lifetime. If high-cost materials are preferred, production costs may increase, which may challenge the economic sustainability of biodiesel production.

In this method, power supply, SS304 class AISI 304 stainless steel electrodes, electrical connection apparatus, magnetic stirrer, methanol, auxiliary solvent, sodium chloride, a small amount of distilled water, glass beaker

and thermometer were used. Experimental parameters were determined using the literature data in Table 1. The anode and cathode electrodes, measuring 20x20x2mm, were fixed at 10 mm and adjusted so that at least 60% of them remained in contact with the electrolyte. In the experiments, 2% distilled water by weight was added to 50 g of waste frying oil and 8:1 molar methanol was mixed. THF as solvent was added at 2% by weight. NaCl was added to this prepared electrolyte at a rate of 0.5% by weight to improve the electrochemical reactions. The reaction voltage of the experiments was provided by a high-precision dual-output DC power supply with an operating range of 0-30 V, Figure 1. According to the literature data in Table 1, the reaction voltage in the electrochemical process was determined as 20 V. The electrochemical reaction process was carried out under 20 V voltage and without heating, at a stirring speed of 300 rpm, in a reaction time of 3 hours. Meanwhile, the temperature inside the reaction remained in the range of 35-45°C. In order to compare the properties of biodiesel, conventional biodiesel production was also carried out under experimental conditions accepted in the literature. For this, 20% methanol and 1% KOH by weight were mixed into 100 g of waste frying oil. The reaction process was carried out at a stirring speed of 400 rpm, a temperature of 60 °C and for 1 hour. The conversion efficiencies of biodiesels obtained by both methods were calculated and their fatty acid distributions were determined. Fatty acid determination was made with GC-MS device according to TS 4664 EN ISO 5508 standards. Then, some physical and chemical fuel properties were calculated numerically according to fatty acid distributions. These features will be compared with EN 14214:2012+A2:2019 biodiesel standard data.

3. Results and Discussion

In this section, the experimental and calculation results of biodiesels produced by electrochemical and conventional methods are compared. With the experimental data, firstly, the conversion efficiencies of biodiesels were calculated and then the fatty acid distributions determined by GC-MS were presented. Finally, some fuel properties were calculated

numerically with fatty acid distributions and compared.

Biodiesel conversion efficiency refers to the efficiency in the process of converting feedstock (waste frying oil) into biodiesel. Biodiesel conversion efficiency was calculated with equation (1).

$$\eta_{conversion} = \frac{m_{biodiesel}}{m_{oil}} \times 100 \quad (1)$$

Biodiesel conversion efficiency ($\eta_{conversion}$) was calculated by the ratio of the weight of biodiesel produced by transesterification ($m_{biodiesel}$) to the used waste frying oil (m_{oil}). In this study, the conversion efficiencies of biodiesels obtained by traditional and electrochemical methods are presented in Fig.2.

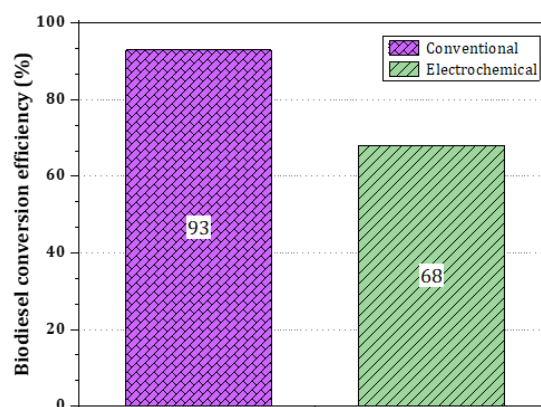


Fig.2. Comparison of biodiesel conversion efficiencies

When Figure 2 is examined, it is seen that a conversion efficiency of 93% is achieved in conventional biodiesel production. It is found that the electrochemical conversion efficiency is 68%. It was observed that there was an improvement compared to the 62% conversion efficiency found in a study on biodiesel production with a similar electrode [14]. It is thought that this improvement in the conversion efficiency in the electrochemical method is due to the high ratio of NaCl and distilled water.

Fatty acid contents were determined on a GC/MS gas chromatography mass spectrometer device (Thermo Scientific ISQ LT model). A Trace Gold TG-WaxMS capillary column (Thermo Scientific code: 26088-1540) with a length of 60 m, an inner diameter of 0.25 μm and a film thickness of 0.25 μm was used for this analysis. Fatty acids were determined by comparing the arrival

times of the standard FAME mixture consisting of 37 components. The fatty acid distributions of waste frying oil and biodiesels produced by two different methods were determined by this method. The fatty acid distributions of waste frying oil are summarized in Table 2.

When Table 2 is examined, it is seen that unsaturated fatty acids are 68.3%. The conversion efficiency is expected to be high in this type of oil. The cloud point of biodiesel derived from crude oil containing highly saturated fatty acids may increase to an undesirable level. It is known that saturated fatty acids in fatty acids negatively affect the viscosity and cold flow properties of the fuel, while unsaturated fatty acids determine fuel stability [20–22].

Alviso et al. performed regression analysis with a genetic algorithm to predict fuel properties as a function of fatty acid composition, using an experimental database. Equations that can calculate the kinematic viscosity (KV), flash point (FP), cloud point (CP), pour point (PP), cold filter plugging point (CFPP), cetane (CN) and iodine numbers (IN) of biodiesel through regression analysis are presented below [23].

For the higher heating value (HHV) and density of the fuel, the equations (Eq.9 and Eq.10) derived by Kumbhar et al. were used according to the fatty acid distributions [24]. In this model, the coefficients of determination are 0.86 and 0.9918, respectively [24].

$$KV = 4.264 + 0.0275 \cdot X_4 + 0.004 \cdot X_5 - 0.00218 \cdot X_6 - 0.0695 \cdot X_{10} \quad (2)$$

$$FP (^\circ C) = 176.318 - 1.727 \cdot X_4 + 0.1 \cdot X_4 \cdot X_7 - 0.4574 \cdot X_{10} \quad (3)$$

$$CP (K) = 268.444 + 0.2 \cdot X_3 + 0.666 \cdot X_4 \quad (4)$$

$$PP (K) = 267.303 + 0.3 \cdot X_3 + 0.505 \cdot X_4 - 0.1 \cdot X_6 - 0.1 \cdot X_7 \quad (5)$$

$$CFPP (K) = 259.051 + 0.72834 \cdot X_3 + 0.5 \cdot X_4 + 7.71255 \cdot X_8 \quad (6)$$

$$CN = 58.445 + 0.266 \cdot X_2 - 0.101 \cdot X_4 - 0.143 \cdot X_6 - 0.21 \cdot X_7 + 0.24 \cdot X_9 \quad (7)$$

$$IN = 42.649 + 7.029 \cdot X_2 - 0.424 \cdot X_3 - 9.09 \cdot X_8 + 0.448 \cdot X_5 + 1.23 \cdot X_6 + 1.994 \cdot X_7 \quad (8)$$

$$\text{Density} = 877.47 + 21.35 \cdot X_1 + 0.619 \cdot X_2 - 0.0367 \cdot X_3 - 0.3740 \cdot X_4 - 0.0599 \cdot X_5 + 0.0007 \cdot X_6 + 0.3532 \cdot X_7 \quad (9)$$

$$HHV = 31.42 + 4.18 \cdot X_1 + 0.011 \cdot X_2 + 0.0916 \cdot X_3 + 0.0112 \cdot X_4 + 0.0656 \cdot X_5 + 0.0740 \cdot X_6 + 0.0947 \cdot X_7 \quad (10)$$

Table 2 Fatty acid distributions of waste frying oil

Saturated fatty acids (C _n :0)		Unsaturated fatty acids (C _n :x)	
Acid type	Area (%)	Acid type	Area (%)
Tridecanoic acid (C13:0)	0.00	Nervonic acid (C24:1)	1.15
Lauric acid (C12:0)	0.26	Linolelaidic acid (C18:2n6t)	1.17
Butyric acid (C4:0)	0.00	Eicosapentaenoic (C20:5n3)	0.15
Undecanoic acid (C11:0)	0.00	Linoleic acid (C18:2n6c)	29.16
Pentadecanoic acid (C15:0)	0.07	Palmitoleic acid (C16:1)	0.13
Trichosanoic acid (C23:0)	0.09	Docosadienoic acid (C22:2)	0.34
Heneicosanoic acid (C21:0)	0.13	Arachidonic acid (C20:4n6)	0.21
Aracidic acid (C20:0)	1.47	Eicosadienoic acid (C20:2)	0.15
Capric acid (C10:0)	0.02	Oleic acid (C18:1n9c)	33.25
Heptadecanoic acid (C17:0)	0.18	Elaidic acid (C18:1n9t)	0.64
Stearic acid (C18:0)	11.46	Pentadecenoic acid (C15:1)	0.02
Caproic acid (C6:0)	0.02	Eicosatrienoic acid (C20:3n6)	0.03
Behenic acid (C22:0)	2.68	Heptadecenoic acid (C17:1)	0.16
Myristic acid (C14:0)	0.51	Alpha linolenic acid (C18:3n3)	0.39
Lignoceric acid (C24:0)	1.32	Erucic (C22_1n9)	0.02
Palmitic acid (C16:0)	12.77	Docosahegzaenoic (C22:6n3)	0.14
Caprylic acid (C8:0)	0.70	Eicosatrienoic acid (C20:3n3)	0.09
		Gamma linolenic (C18:3n6)	0.05
		Eicosenoic acid (C20:1)	1.07
		Myristoleic acid (C14:1)	0.01

TOTAL (%) 31.70**TOTAL (%) 68.30**

Table 3 Some fatty acid distributions of biodiesels produced with conventional and electrochemically

Formula Coding	Fatty acid types	Biodiesel of conventional method	Biodiesel of electrochemical method
X ₁	Lauric acid (C12:0)	0.25	0.21
X ₂	Myristic acid (C14:0)	0.51	0.58
X ₃	Palmitic acid (C16:0)	13.46	12.15
X ₄	Stearic acid (C18:0)	11.74	13.45
X ₅	Oleic acid (C18:1n9c)	34.16	37.56
X ₆	Linoleic acid (C18:2n6c)	29.14	27.45
X ₇	Alpha linolenic acid (C18:3n3)	0.35	0.25
X ₈	Behenic acid (C22:0)	0.21	0.21
X ₉	Eicosadienoic acid (C20:3n3)	0.11	0.05
X ₁₀	Erucic (C22_1n9)	0.01	0.12

Table 4 Some fuel properties of biodiesels produced by conventional and electrochemical methods

Fuel specifications	EN 14214	Biodiesel of conventional method	Biodiesel of electrochemical method
Kinematic viscosity (cSt)	3.5-5	4.66	4.71
Flash point (°C)	min.101	156.46	153.37
Cloud point (°C)	-	5.80	6.68
Pour point (°C)	-	1.17	1.82
Cold filter blocking point (°C)	-	3.15	3.09
Iodine number	max.120	90.46	90.75
Cetane number	min. 51	53.18	53.27
Density (kg/m ³)	860-900	876.23	874.70
Higher heating value (MJ/kg)	-	38.24	38.09

The values of fatty acids used to determine the fuel properties of biodiesels obtained by conventional and electrochemical processes are presented in Table 3. Additionally, the names of fatty acids corresponding to "X_i" in the equations used in Eq.2-10 are shown in this table.

Fuel properties such as kinematic viscosity (KV), flash point (FP), cloud point (CP), pour point (PP), cold filter plugging point (CFPP), cetane (CN), iodine numbers (IN), density and higher heating value (HHV) were calculated theoretically by using the fatty acid types given in Table 3 in the equations in Eq.2-10, and are presented in Table 4.

When Table 4 is examined, it is observed that biodiesels produced by conventional and electrochemical methods comply with the EN 14214 biodiesel standard. It is observed that the viscosity of biodiesel produced by the electrochemical method is higher compared to the other method. It is also found that its higher heating value remained lower. The term fuel

lower heating value is a fuel characteristic used to calculate and compare the specific energy consumption of different fuels used in an engine. The higher this value of the fuel, the higher the possible energy output. It is also used in calculating thermal efficiency. In this regard, it is necessary to measure or approximately estimate the lower heating value of the fuel rather than the HHV. For this purpose, the lower heating values of the biodiesels produced according to the model developed by Erdoğan were determined [25]. Accordingly, the biodiesel of the conventional method and the biodiesel of the electrochemical method were estimated to be 37.54 MJ/kg and 37.27 MJ/kg, respectively. In a study where a similar method was applied, the effect of NaCl on the biodiesel conversion efficiency for waste frying oil was investigated. According to this study using platinum electrodes, it is seen that more than 1% NaCl by weight must be used to achieve a conversion efficiency of 68%. Accordingly, in

my article, an improvement was detected compared to the other study, with 68% efficiency being achieved with 0.5% NaCl. Moreover, the methanol content remained low [26]. It has been determined that the conversion efficiency is lower compared to the study using a circular cross-section rod graphite electrode, which is brittle at the same reaction voltage and similar methanol/oil ratio. The use of CaO as a catalyst in the relevant study is thought to be a factor in this [27]. In terms of the fuel properties found in this study, the results are similar to other studies [28, 29] using the electrolysis method.

According to the results, it is found that biodiesel production from waste frying oil by the electrochemical method using steel electrodes is an effective method. This method encourages more efficient use of renewable energy resources by reducing the environmental impact of waste frying oils. This method can evolve into important processes in the energy sector by providing sustainable solutions in the fields of waste management and biofuel production. It also appears to be environmentally and economical.

4. Conclusions

It is important that the production method of biodiesel is economical, and the electrodes are efficient and reusable. In this study, a reusable, non-fragile and easily accessible electrode was preferred. The results obtained in this study, in which biodiesel production by electrochemical method was investigated by using low-cost SS304 class AISI 304 stainless steel electrodes instead of high-cost electrodes, are presented below.

- It is shown that biodiesel production is possible with low-cost steel electrodes.
- The use of steel electrodes in the electrochemical process resulted in a low conversion efficiency of 68%. However, it was found that the properties of the produced fuel comply with the EN 14214 standard.
- Compared to the conventional method, it required the use of more methanol and a longer reaction time.
- Waste production in the electrochemical method can be reduced as it offers a more environmentally friendly option compared to the conventional method.

- Steel electrodes offer a long-lasting solution thanks to their high durability. Moreover, it can provide stable results if adapted to continuous production processes.

- With this method, costs in biodiesel production can be reduced. It can be an important road map for a more sustainable future in the energy sector.

The experimental variables used should be optimized to improve the fuel properties of biodiesel produced by the electrochemical method and increase the conversion efficiency.

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