



Comparative study of graphite and titanium electrodes for electrochemical biodiesel production

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Abstract

This study comparatively investigates the effects of graphite and titanium electrodes on process performance, energy consumption, and product quality in the electrolysis-based production of biodiesel from waste cooking oil (WCO). To optimize the production parameters, a Taguchi L18 orthogonal array and Response Surface Methodology (RSM) were systematically employed. The results demonstrated that while graphite electrodes operate effectively at lower voltages (approximately 20 V), titanium electrodes require higher voltage levels (60 V) to achieve significant conversion. Under optimized conditions, titanium electrodes achieved a remarkably high biodiesel conversion yield of 93.00% with a specific energy consumption (SEC) of approximately 1.19 kWh/kg, whereas graphite electrodes yielded 83.45% with a significantly lower SEC of 0.25 kWh/kg. Statistical analysis (ANOVA) revealed that the KOH catalyst concentration was not a statistically significant factor within the narrow range tested (0.5–1.0 wt%), suggesting that electrolysis operates through a different catalytic mechanism involving in-situ ion generation. Fuel property characterizations, including GC-MS analysis and cold-flow properties (CFPP of −26.86 °C for graphite and −23.67 °C for titanium), confirmed that biodiesels produced with both electrode types show a close alignment with EN 14,214 standards. This research highlights that electrode material selection is a decisive factor influencing not only the conversion efficiency and energy intensity but also the molecular-level characteristics of the resulting fuel.

Keywords Biodiesel · Electrolysis · Waste cooking oil · Electrode materials · Taguchi method · Response Surface Methodology (RSM) · Affordable and clean energy

1 Introduction

The global energy system is undergoing a profound transformation that exposes deep-rooted fossil fuel dependencies and critical bottlenecks impeding sustainable transition (Kuzemko et al. 2024). The transformation requirements are immense, as limiting climate change necessitates reducing net CO₂ emissions to zero, requiring fundamental restructuring of energy systems that currently power the world's economies (Edmonds et al. 2024). In this context, biofuels

such as biodiesel have emerged as one of the most viable renewable alternatives to fossil fuels (Akinwale Ishola 2024) (Augusta Heavens Ikevuje et al., 2024). The challenge demands coordinated global action to address both technical limitations and systemic barriers that maintain fossil fuel dominance across interconnected energy systems.

The environmental management of waste cooking oils represents a pivotal intersection of pollution prevention and sustainable resource recovery within circular economic frameworks. Improper disposal of waste cooking oils creates significant environmental challenges, including water and soil contamination, sewage system damage, and increased biological oxygen demand in water bodies (Hamdi et al. 2022). However, when properly managed, these waste streams offer substantial opportunities for value creation and environmental benefit.

Global waste cooking oil production ranges from 29 to 67 million metric tons annually, representing an enormous untapped resource for sustainable energy production

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(Judijanto, 2025). Converting waste cooking oils into biodiesel through transesterification processes exemplifies circular economy principles by transforming waste into high-value products (Okpo and Edafiadhe 2024). This approach delivers multiple environmental advantages, including lower greenhouse gas emissions, enhanced biodegradability, and reduced dependence on fossil fuels (Sreeletha et al., 2024). Non-edible feedstock-based biodiesels have shown favorable fuel properties and engine performance when evaluated under varying compression ratios (Abishek et al. 2024a).

The conversion of waste cooking oil into biodiesel presents a clear pathway for sustainable resource recovery, addressing both waste management challenges and energy transition imperatives. Advanced biodiesel production from waste cooking oils exemplifies the convergence of technological innovation and circular economy principles. Recent developments in process intensification, including microwave- and ultrasonic-assisted transesterification systems, have achieved remarkable efficiency gains, demonstrating 94–96% biodiesel yields within 5–10 min while reducing energy consumption by 40% compared to conventional methods (Laila et al. 2025). Combustion and emission analyses of biodiesel blends have confirmed that fuel quality is directly linked to production method and feedstock selection (Abishek et al. 2024b). These technological advances carry the potential for 75–80% greenhouse gas emission reductions relative to fossil diesel. Among emerging production methods, electrolysis-assisted transesterification has attracted increasing attention due to its ability to operate under milder conditions without conventional heating requirements. The selection of electrode material is a decisive factor influencing conversion efficiency, energy consumption, and product quality yet comparative studies on electrode performance remain limited in the literature.

The traditional transesterification method for biodiesel production faces substantial limitations that compromise both economic viability and environmental sustainability. Operating at elevated temperatures of 60–70 °C with extended reaction times, this conventional approach creates significant energy demands that increase production costs and potentially undermine the environmental benefits of biodiesel (J. W. Y. Lee et al. 2022). The method's inherent inefficiencies include slow reaction rates, soap formation, and intensive pre- and post-processing requirements, ultimately leading to increased capital and operational expenses (Deshpande et al. 2017). These energy-intensive operational requirements represent persistent challenges that necessitate exploration of alternative production pathways (Jayabal 2024).

In response to these limitations, researchers have developed various promising alternatives that address the

fundamental shortcomings of traditional transesterification. Methods such as supercritical transesterification offer fast reaction rates, feedstock flexibility, and production efficiency compared to conventional alkaline catalysis (Silva and Oliveira 2014), while catalyst-free approaches demonstrate faster conversion rates with higher yields and more environment-friendly production routes (P. P. Singh et al., 2021). Additionally, innovative heating methods such as microwave-assisted transesterification can reduce reaction times by at least 60% compared to conventional approaches (Mohamad Aziz et al. 2021).

There is an extensive body of literature regarding biodiesel production using various techniques. Among these, studies focusing on conventional heating, microwave-assisted, ultrasonic-assisted, and electrolysis-assisted production are detailed below. In one such study, biodiesel was produced from corn oil and waste cooking oil using the electrolysis technique; researchers achieved a 97% conversion yield using platinum electrodes (Guan and Kusakabe 2009). In another study, biodiesel was produced from microalgae-derived oil via electrolysis in the presence of a homogeneous catalyst, attaining a yield of 95% (Moradi and Saidi 2022a). Fereidooni et al. (Fereidooni et al. 2018) utilized graphite electrodes to produce biodiesel from waste frying oil using a zeolite/chitosan catalyst and reported high conversion yields.

Motasami and Ani conducted a comprehensive review of microwave-assisted biodiesel production using various feedstocks. Their research indicated that microwave-assisted production significantly reduces reaction time compared to classical methods and allows for the extraction of high-quality biodiesel from different plant sources. Concluding their review, they calculated the energy consumption of the microwave-assisted method, asserting the system's sustainability. Consequently, they suggested that this method could be a viable approach for reducing costs in biodiesel production (Motasemi and Ani 2012).

In their work, Lin and Chen utilized an improved microwave unit to produce biodiesel from *Jatropha* oil. Due to the high fatty acid content of the *Jatropha* oil, they first reduced the acid ratio with ethyl ester before initiating the conversion process. The operating conditions were established as a 6:1 methanol/oil molar ratio, 1% catalyst load, 200 rpm stirring speed, and a reaction temperature of 65 °C. Under these conditions, the oil conversion rate reached 90% in just 10 s. Consequently, the study demonstrated that the microwave reactor drastically reduced the reaction time compared to traditional heating methods. Furthermore, the researchers emphasized that mass production could be achieved using continuous microwave reactors (Lin and Chen 2017).

Kumar et al. produced coconut ethyl ester from coconut oil using an ultrasonic method. They identified the optimum

conditions for biodiesel production as a 6:1 ethanol/oil molar ratio and 0.75% KOH. The reaction reached completion in a remarkably short time 7 min leading to the conclusion that the reaction time is significantly shortened compared to traditional methods. Additionally, a conversion yield of over 98% was observed under these conditions. It was noted that the reduction in reaction time could lower costs and help ensure sustainability (Kumar et al. 2010). Similarly, another study employed an ultrasonic synthesis method for biodiesel production from microalgae, noting a reduction in reaction time (Sivaramakrishnan and Incharoensakdi 2018).

Topare et al. utilized ultrasonic-assisted production in their study on waste soybean oil. The ultrasonication process was carried out at a frequency of 20 kHz. A 6:1 methanol/oil molar ratio and a 1.5% (by weight) calcium oxide catalyst were used. The optimum reaction temperature and time were selected as 70 °C and 60 min, respectively. Under these parameters, a biodiesel production yield of 98% was determined. Furthermore, when the resulting biodiesel was used in compression-ignition engines, the performance and emission values were found to be comparable to those of diesel fuel (Topare and Patil 2021).

Considered the inaugural study on biodiesel production using the electrolysis technique, Guan and Kusakabe examined production from corn oil and waste cooking oil (WCO) using platinum electrodes, finding a maximum biodiesel yield of 97% (Guan and Kusakabe 2009). Moradi et al. produced biodiesel from **Chlorella Vulgaris** microalgal-derived oil via electrolysis with a homogeneous catalyst, achieving a peak yield of 95% (Moradi and Saidi 2022b).

Nguyen et al. also produced biodiesel using the electrolysis technique. They set the experimental conditions at an electrolysis voltage of 22.06 V, an oil-to-methanol molar ratio of 25.09:1, a solvent amount of 7.91% by weight, and a reaction time of 2 h. Performed at room temperature, this study resulted in biodiesel with a 94.7% conversion yield. The researchers emphasized that the produced biodiesel complied with international standards. They proposed that electrolysis, incorporating a supporting electrolyte and co-solvent, is an eco-friendly, cost-effective, and efficient method for biodiesel synthesis (Chinh Nguyen et al. 2023).

In a related study, researchers produced biodiesel from non-edible seeds at room temperature using electrolysis-assisted transesterification. They determined the working parameters to be 5–15 V/cm for voltage, 1–3 h for duration, and a methanol-to-oil molar ratio of 5:1–20:1. Based on the experimental design, a second-order model was developed to investigate the effects of each parameter on biodiesel yield. The experimental results were found to be consistent with the predicted values. The highest biodiesel yield was recorded at 28% without a catalyst and 80% when using an acidic catalyst. They reported the optimum experimental

conditions as a 2-hour duration, 15 V/cm voltage, 20:1 methanol: oil molar ratio, 10:1 THF: oil molar ratio, 1% NaCl (wt), 2% distilled water (wt), and 16% catalyst (wt). Consequently, they strongly recommended the electrolysis technique as an effective method for the industrialization of biodiesel production (Karimi and Saidi 2022).

Among these emerging technologies, electrolysis-based methods represent a particularly innovative approach to addressing the temperature, time, and energy consumption challenges inherent in traditional transesterification, offering unique advantages in the pursuit of sustainable and efficient biodiesel production.

Electrolysis-based biodiesel production is an emerging green technology that utilizes electrochemical processes to convert various oil feedstocks into biodiesel fuel under mild operating conditions. This innovative method works by applying an electric field to generate active ions in-situ within the reaction mixture, facilitating the transesterification of triglycerides with methanol at room temperature (Thangaraj and Solomon 2021). The technology demonstrates remarkable versatility, successfully processing diverse feedstocks including waste cooking oil, palm oil, sunflower oil, and non-edible oils like Azadirachta India seed oil, achieving biodiesel yields ranging from 80% to 98.6% S (Rachman et al. 2018). Unlike conventional transesterification methods that require elevated temperatures (around 60 °C) and extended reaction times of 60–120 min, electrolysis processes can complete biodiesel synthesis in 1–3 h at ambient temperature [31]. The method offers significant sustainability advantages by reducing energy consumption, minimizing catalyst requirements, and in some cases operating entirely catalyst-free through the use of supporting electrolytes such as ionic liquids or deep eutectic solvents (Aregawi et al. 2024). This process intensification approach not only enhances reaction efficiency and selectivity but also contributes to environmental protection by reducing greenhouse gas emissions while providing a cost-effective pathway for commercial biodiesel production from renewable resources.

The selection of electrode material represents a fundamental determinant of success in electrolytic biodiesel production, directly influencing catalytic efficiency, electrical conductivity, and overall process economics. Electrode material choice is critical for achieving optimal yields and selectivity, as it significantly impacts electron transfer kinetics and thermodynamics (Heard and Lennox 2020). Experimental evidence demonstrates substantial performance variations across different electrode materials, with graphite electrodes achieving 87% biodiesel yield from waste cooking oil while platinum electrodes reached 90% from palm oil, highlighting material-specific catalytic effects (Darwin et al., 2023). Systematic comparisons between iron

and aluminum electrodes reveal that iron delivers superior performance with 81.12% yield at lower field strengths (125 V) compared to aluminum's 80.12% yield requiring higher voltage (138 V), demonstrating iron's better chemical stability in alkaline media (Sangsawang et al. 2025). Similarly, additive and material selection have been shown to critically affect both combustion efficiency and emission characteristics in biodiesel-fueled engines (Abishek and Kachhap 2025). Stainless steel electrodes have shown promising results, with plain stainless steel achieving 81% conversion rates and hybrid ion-exchange resin-coated electrodes reaching 87% Francois-Marie Allieux et al., 2017. Additional studies using SS304 stainless steel obtained 68% conversion efficiency in electrochemical biodiesel production (Balki 2024). The electrode surface structure and chemical stability directly govern electron transfer efficiency and reaction selectivity, with high-conductivity materials reducing energy losses and electrochemically stable compositions limiting undesirable side reactions. Current research identifies ongoing challenges including limited catalytic activity, high costs for premium materials, and corrosion susceptibility, necessitating continued research into surface modification techniques and nanostructure design to optimize electrode performance for industrial-scale biodiesel production (Dai and Meng 2025).

In this experimental design, graphite and titanium were specifically selected to represent two contrasting paradigms in electrochemical reactor design. Graphite was chosen as the representative of carbon-based materials due to its high electrical conductivity, widespread availability, and cost-effectiveness, which are critical factors for economic scalability. Conversely, titanium was selected to represent metallic electrodes, distinguished by its superior mechanical strength, exceptional corrosion resistance, and electrochemical stability under oxidative conditions. By juxtaposing these two distinct materials, this study aims to elucidate the critical trade-offs between economic feasibility represented by graphite and operational durability represented by titanium in the electrolysis-assisted biodiesel production process.

Although a growing number of studies have investigated biodiesel production using electrolysis-based methods, the influence of electrode material on process performance and fuel properties has not yet been comprehensively addressed. Existing studies predominantly focus on single electrode types, and a direct and systematic comparison between graphite and titanium electrodes under identical operating conditions is notably absent from the literature. Moreover, prior research has largely emphasized biodiesel yield, while the effects of electrode material on molecular-level fuel characteristics—such as fatty acid methyl ester (FAME) composition determined by GC–MS analysis and cold-flow

properties—remain insufficiently explored. In addition, the necessity of catalyst usage in electrolysis-assisted biodiesel production is often assumed rather than critically examined, and the potential statistical insignificance of catalyst concentration, indicating the feasibility of catalyst-free or catalyst-minimized processes, has not been adequately highlighted. Furthermore, optimization studies in electrochemical biodiesel production typically rely on a single statistical approach. The combined and cross-validated application of Taguchi design and Response Surface Methodology (RSM) for optimizing electrolysis parameters remains an underexplored area, representing a significant methodological gap. Collectively, these limitations hinder a holistic understanding of the interrelationships among electrode selection, energy consumption, and biodiesel quality.

While several studies have explored electrolysis-based transesterification, this research distinguishes itself by providing a systematic, side-by-side comparison of graphite (carbon-based) and titanium (metallic) electrodes under identical operating conditions. Unlike previous literature that primarily focuses on single-electrode systems, our study aims to bridge the gap regarding electrode selectivity and its impact on the final fatty acid methyl ester (FAME) profile. Furthermore, this study investigates the statistical significance of traditional catalyst concentrations in an electrochemical environment, aiming to demonstrate the potential for catalyst-minimized production processes.

The primary objective of this study is to comparatively evaluate the effects of graphite and titanium electrodes on process performance, energy consumption, and product quality in the electrolysis-based production of biodiesel from waste cooking oil. Within this scope, optimum process parameters for biodiesel production using each electrode type are determined, and the energy–yield trade-off between the applied voltage and biodiesel conversion efficiency is quantitatively analyzed. Furthermore, the influence of electrode material on the chemical composition of biodiesel is investigated through GC–MS analysis, while fuel quality parameters are assessed in alignment with EN 14,214 standards, including cold-flow properties. Finally, statistical models developed using Taguchi and Response Surface Methodology (RSM) are validated against experimental data, and the reliability of these models is systematically evaluated.

2 Materials and methods

2.1 Material preparation

2.1.1 Feedstock

Waste cooking oil (WCO) was collected from local restaurants and household kitchens in Sinop, Turkey. The collected oil was subjected to a multi-stage purification process to remove solid particles and moisture. Initially, the oil was filtered through cellulose-based filter papers to eliminate suspended solid impurities. Subsequently, the filtered oil was heated to 110 °C and maintained at this temperature for 30 min under atmospheric pressure to evaporate free water content. This thermal treatment was critical to prevent saponification reactions during transesterification by reducing the water concentration below 0.5 wt%. The pretreated oil was cooled to room temperature and stored in sealed amber glass bottles at 4 °C to minimize oxidative degradation prior to biodiesel synthesis.

2.1.2 Chemicals

Methanol ($\geq 99.9\%$ purity, HPLC grade) and potassium hydroxide (KOH, analytical reagent grade, $\geq 85\%$ purity) were procured from Merck KGaA (Darmstadt, Germany). Acetone ($\geq 99.5\%$ purity) was obtained from Sigma-Aldrich and employed as a co-solvent at a mass ratio equivalent to 50% of the methanol volume. The primary function of acetone in the reaction mixture is to reduce the interfacial tension between the immiscible oil and methanol phases, thereby creating a more homogeneous reaction environment. This improved miscibility significantly increases the effective contact area between the reactants and the electrode surface, facilitating methanol diffusion into the viscous oil phase and accelerating the transesterification reaction. Additionally, acetone contributes to reducing overall solution viscosity and enhancing ionic conductivity of the electrolyte mixture. Distilled water with an electrical conductivity of $< 2 \mu\text{S}/\text{cm}$ was utilized to further enhance ionic transport during electrolysis. All chemicals were used as received without additional purification.

2.1.3 Electrolyte preparation

The electrolyte solution was formulated through a systematic mixing protocol to ensure homogeneous composition and optimal ionic conductivity. First, a predetermined quantity of KOH catalyst was completely dissolved in methanol under continuous magnetic stirring at 300 rpm for 5 min, forming a potassium methoxide solution. The co-solvent acetone was then added at a mass ratio equivalent to 50%

of the methanol volume to reduce solution viscosity and improve mass transfer characteristics. Finally, distilled water was incorporated at 3 wt% relative to the oil mass to generate electrolytically active hydrogen and hydroxide ions in situ through water hydrolysis. This electrolyte mixture was stirred for an additional 10 min at room temperature (25 ± 2 °C) to achieve complete miscibility before introducing the pretreated waste cooking oil. The oil was added last and mixed at 400 rpm for 3 min to establish a pseudo-homogeneous emulsion suitable for electrochemical transesterification.

2.2 Experimental setup and equipment

2.2.1 Electrolysis cell and electrode configuration

The electrolytic biodiesel production experiments were conducted in a custom-designed electrochemical reactor system. Two distinct electrode materials were evaluated: graphite plates ($25 \times 40 \times 2$ mm, electrical resistivity $< 10 \mu\Omega \cdot \text{m}$) and titanium plates ($25 \times 40 \times 1$ mm, Grade 2 commercially pure titanium, ASTM B265).

Both electrode types shared identical planar dimensions (25×40 mm) and were operated at a submerged depth of 30 mm. This configuration yielded a total active submerged surface area of 15.0 cm^2 per electrode, accounting for the electrochemical contribution of both sides (7.5 cm^2 per side). The use of identical surface areas for both graphite and titanium electrodes ensures that any observed performance variations are fundamentally attributable to intrinsic material properties rather than geometric factors. To characterize the electrochemical drive under optimized operating conditions, the average current density (J) was evaluated. The resulting current densities were $43.3 \text{ mA}/\text{cm}^2$ for the graphite electrode (at $\sim 0.65 \text{ A}$) and $80.0 \text{ mA}/\text{cm}^2$ for the titanium electrode (at $\sim 1.2 \text{ A}$).

To ensure reproducible electrode positioning and maintain a constant inter-electrode gap, custom-designed electrode holders were fabricated using polylactic acid (PLA) filament via fused deposition modeling (FDM) on a 3D printer (resolution: 0.1 mm layer height). These holders maintained a fixed electrode spacing of 10 mm throughout all experiments, which was critical for consistent electric field distribution and current density. Figure 1 shows the electrode holder and the electrodes used in the experiments.

The electrolysis cell consisted of a 100 mL borosilicate glass beaker (Schott Duran, thermal expansion coefficient: $3.3 \times 10^{-6} \text{ K}^{-1}$) that served as the reaction vessel. Continuous agitation was provided by a magnetic stirrer operating at a constant speed of 400 rpm to promote uniform reactant distribution and minimize concentration polarization at the electrode surfaces. Temperature monitoring was performed



Fig. 1 Graphite (left) and titanium (right) electrode plates with custom 3D-printed PLA holders used in electrolysis experiments. Electrode dimensions: 25×40 mm

using a K-type thermocouple (± 0.5 °C accuracy) inserted into the reaction mixture. During electrolysis, the reactor temperature equilibrated between 35 and 50 °C depending on the applied voltage, with no external heating required. This mild temperature regime represented a significant energy efficiency advantage compared to conventional thermal transesterification. The electrolysis process facilitates the in-situ generation of active catalytic species through electrochemical reduction. At the cathode, the reduction of methanol and the incorporated water leads to the formation of methoxide (CH_3O^-) and hydroxide (OH^-) ions, which serve as the primary nucleophiles for the transesterification of triglycerides at ambient temperatures. The selection of electrode material is a decisive factor in this mechanism as it governs the overpotential and electron transfer kinetics. Graphite's high surface conductivity allows for effective ion generation at lower operating voltages, whereas titanium electrodes require a significantly higher voltage to overcome the activation barrier for initiate the same catalytic activity.

2.2.2 Power supply and control system

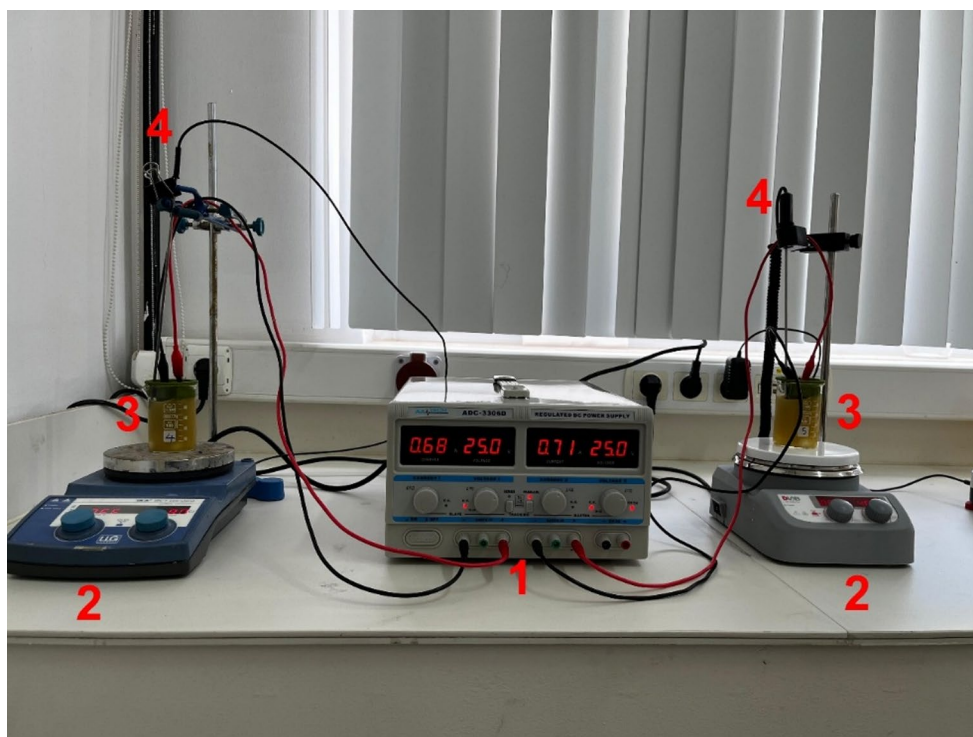
Direct current (DC) power was supplied using a programmable dual-channel DC power supply (AATech ADC-3306D) with the following specifications: voltage range 0–30 V, current range 0–6 A per channel, voltage regulation $\leq 0.01\%$, and current regulation $\leq 0.2\%$. The power supply featured independent voltage and current control with four-digit LED displays providing real-time monitoring of both output parameters. Voltage and current stability were maintained within $\pm 0.1\%$ throughout each experiment. The programmable dual-channel DC power supply (AATech ADC-3306D) offers three operating modes: independent mode (0–30 V / 0–6 A per channel), parallel mode (0–30 V / 0–12 A), and series mode (0–60 V / 0–6 A). Graphite electrode experiments were conducted in independent mode at up to 30 V, while titanium electrode experiments requiring

60 V were performed using the built-in series mode of the same unit. The output current was monitored in real-time through the four-digit LED displays of the DC power supply to ensure process stability. While the current was observed to remain highly stable within the instrument's regulation limits ($\pm 0.1\%$) throughout the reaction, an automated data logging system was not utilized for continuous recording during this phase of the research. Consequently, the reported electrochemical parameters focus on the controlled voltage levels and the resulting process efficiency. The electrodes were connected to the power supply terminals with copper wires (14 AWG, oxygen-free high conductivity) to minimize resistive losses. The entire experimental setup was operated at ambient laboratory conditions (25 ± 2 °C, $45 \pm 5\%$ relative humidity) to ensure consistency across all experimental runs. Figure 2 shows the power supply and experimental setup used. Figure 2. General view of the experimental system, illustrating the primary equipment: (1) Programmable dual-channel DC power supply (AATech ADC-3306D), (2) Magnetic stirrers, (3) Borosilicate glass beakers (electrolysis cells) with custom 3D-printed PLA electrode holders, and (4) K-type thermocouples.

2.2.3 Analytical instrumentation

Biodiesel density measurements were conducted at 15 °C using a high-precision digital pycnometer (Anton Paar DMA 4500 M, measurement uncertainty: ± 0.00005 g/cm³). Each sample was measured in triplicate, and the mean value was reported. Fatty acid methyl ester (FAME) composition was determined by gas chromatography-mass spectrometry (GC-MS) using an Agilent 7890B system equipped with a DB-23 capillary column (60 m $\times$ 0.25 mm i.d., 0.25 μ m film thickness). The GC oven temperature program started at 50 °C (held for 1 min), ramped to 175 °C at 25 °C/min, then to 230 °C at 4 °C/min (held for 5 min). Helium was used as the carrier gas at a constant flow rate of 1 mL/min. MS

Fig. 2 General view of the experimental system



detection was performed in electron ionization (EI) mode at 70 eV with a scan range of m/z 50–550. Peak identification was accomplished by comparing retention times and mass spectra with NIST library standards. Kinematic viscosity at 40 °C was measured using a Cannon-Fenske routine viscometer (ASTM D445) with automatic timing. Cold filter plugging point (CFPP) determinations followed the EN 116 standard protocol using a cloud and pour point tester (Tanaka AFP-102).

2.3 Experimental design and optimization

2.3.1 Taguchi orthogonal array design

To systematically investigate the effects of process parameters on biodiesel yield and quality, a Taguchi L_{18} ($2^1 \times 3^3$) orthogonal array was employed for each electrode type. This robust experimental design methodology enabled efficient screening of four critical variables across mixed-level factorial space while requiring only 18 experimental runs per electrode instead of the 54 runs demanded by full factorial design. The Taguchi approach thus provided substantial reductions in experimental effort, material consumption, and analysis time without sacrificing statistical rigor.

For graphite electrode experiments, the four independent variables and their respective levels were: (1) methanol-to-oil mass ratio: 35%, 40%, 45%; (2) reaction time: 30, 45, 60 min; (3) KOH concentration: 0.5%, 0.75%, 1.0% (wt% relative to oil); and (4) electrolysis voltage: 20, 40, 60 V.

For titanium electrode experiments, preliminary screening trials indicated that higher voltages were required to initiate effective electrolysis due to titanium's different electrochemical characteristics. Consequently, the voltage levels for titanium were adjusted to 40, 50, 60 V while maintaining identical levels for the other three parameters. All other process conditions (electrode spacing, stirring speed, initial oil mass of 50 g, co-solvent and water additions) were held constant across both electrode series to enable valid inter-electrode comparisons.

2.3.2 Response surface methodology (RSM)

Following Taguchi screening analysis, Response Surface Methodology (RSM) was implemented to model the nonlinear relationships between process variables and responses (biodiesel yield and density) and to identify global optimal operating conditions. Second-order polynomial regression models were developed according to:

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j + \varepsilon$$

where Y represents the predicted response, β_0 is the constant term, β_i are linear coefficients, β_{ii} are quadratic coefficients, β_{ij} are interaction coefficients, x_i and x_j are coded independent variables, and ε is the random error. Model adequacy was assessed using adjusted coefficient of determination (R^2_{adj}), adequate precision ratio (signal-to-noise ratio > 4), and residual diagnostic plots. Numerical optimization was

performed using the desirability function approach to simultaneously maximize biodiesel yield and minimize density within the experimental design space. Three-dimensional response surface plots were generated to visualize parameter interactions and identify optimal process windows. All statistical analyses, ANOVA calculations, and RSM modeling were executed using Minitab 19 Statistical Software (Minitab LLC, State College, PA).

2.4 Analytical methods and quality assessment

2.4.1 Biodiesel recovery and purification

Upon completion of each electrolysis experiment, the reaction mixture was immediately transferred to a separatory funnel and allowed to settle for 12 h at room temperature to achieve complete phase separation. The denser glycerol-rich bottom phase was drained and discarded. The upper biodiesel-rich phase was then subjected to a two-stage purification protocol. First, dry washing was performed using magnesol (magnesium silicate, 3 wt% relative to crude biodiesel) under gentle stirring for 15 min to absorb residual glycerol, soap, and catalyst. The mixture was subsequently centrifuged at 3500 rpm for 10 min to remove the adsorbent. Second, the supernatant biodiesel was filtered through Whatman No. 1 filter paper and dried over anhydrous sodium sulfate (Na_2SO_4) for 24 h to eliminate trace moisture. The purified biodiesel was then decanted, weighed, and stored in sealed amber glass vials under nitrogen atmosphere at 4 °C prior to characterization.

2.4.2 Conversion efficiency and density measurements

The biodiesel conversion efficiency was determined gravimetrically by calculating the mass ratio of the purified and dried biodiesel obtained after the reaction to the initial mass of the crude oil used. Density measurements at 15 °C were conducted in triplicate using the digital pycnometer described in Sect. 2.2.3, with results expressed as the mean $\pm$ standard deviation. The measured density values were evaluated against the EN 14,214 standard specification range of 860–900 kg/m³. The obtained results were systematically compared with these standards to assess fuel quality, with minor deviations in density being further analyzed in relation to conversion purity and feedstock characteristics.

2.4.3 Statistical analysis

Signal-to-noise (S/N) ratios were calculated according to the Taguchi method to evaluate parameter effects on response robustness. For biodiesel yield (larger-is-better characteristic), the S/N ratio was computed as $-10 \log_{10}(\Sigma(1/y^2)/n)$,

where y represents the measured yield and n is the number of observations. For density (smaller-is-better characteristic), the S/N ratio was calculated as $-10 \log_{10}(\Sigma y^2/n)$. Higher S/N ratios indicated more desirable and stable performance. Analysis of variance (ANOVA) was performed at a 95% confidence level ($\alpha=0.05$) to determine the statistical significance of each parameter and their contributions to total variation. Parameters were considered statistically significant when their p -values were less than 0.05 and their F -values exceeded 4.0. The percentage contribution of each parameter was calculated from the sum of squares (SS) values obtained from ANOVA tables. Regression model adequacy for RSM analysis was confirmed when adjusted R^2 values exceeded 0.80 ($\geq 80\%$), indicating that the model could explain at least 80% of the response variability.

2.4.4 Fuel property characterization

Selected biodiesel samples obtained under optimized conditions for each electrode type were subjected to comprehensive fuel property evaluation according to EN 14,214 specifications. Beyond density and FAME composition (GC-MS), the following parameters were measured: kinematic viscosity at 40 °C (EN ISO 3104), cold filter plugging point/CFPP (EN 116), cetane number (calculated from FAME profile using empirical correlations), and iodine value (calculated from fatty acid composition according to AOCS Cd 1c-85). Each measurement was performed in duplicate, and results were compared against the EN 14,214 limits to evaluate fuel quality. The calculated fuel properties enabled a comparative assessment of how electrode material selection influences the molecular-level composition and resulting performance characteristics of the produced biodiesel.

3 Results

3.1 Experimental design and biodiesel production

This section presents the comprehensive statistical evaluation of electrolysis-assisted biodiesel production using graphite and titanium electrodes. The experimental data obtained from the Taguchi L_{18} orthogonal array were systematically analyzed through signal-to-noise (S/N) ratio calculations, analysis of variance (ANOVA), and response surface methodology (RSM) to identify significant process parameters, develop predictive models, and determine optimal operating conditions for each electrode material.

The $L_{18}(2^1 3^3)$ design was used in biodiesel production using Taguchi experimental design. Biodiesel conversion was carried out using the electrolysis method with domestic waste frying oil as the catalyst, along with a solvent and

alcohol. The project aimed to determine the optimum operating parameters of graphite and titanium-based electrodes in biodiesel production. Potassium hydroxide (KOH) was used as the catalyst, acetone as the solvent, and methanol as the alcohol. The catalyst and alcohol ratios were added as a percentage by weight relative to the weight of the crude oil. Fifty grams of domestic waste cooking oil was used in each experiment. In each experiment, half the amount of methanol used as the solvent was added as acetone, and 3% of the oil was added as distilled water to increase conductivity. First, KOH was dissolved in 100 cc of methanol calculated based on the 50 g oil ratio. Then, half the amount of acetone used in the methanol and 3% distilled water of the oil were added to the methoxide mixture and stirred in a mixer at 300 rpm. Finally, 50 g of domestic waste frying oil was added to this prepared mixture. All experiments were carried out at an average ambient temperature of 25 °C.

Following the tests, the samples were subjected to washing and drying processes as specified in the materials and methods section. Similar processes were carried out for each experiment, and the biodiesel conversion yield of the biodiesels produced was calculated and their densities were measured and recorded using a densimeter. The Taguchi L18(2¹ 3³) experimental design for biodiesel production with graphite electrodes and the results obtained are given in Table 1.

According to the TS EN 14,214+A1 standard, the density of biodiesel (at 15 °C) is specified within the 860–900 kg/m³ range. Table 1 shows that the results obtained (839–843 kg/m³) demonstrate a close alignment and near-compliance with these values. Based on these results, it is considered that the biodiesel produced in all experiments

in the experimental design is suitable for optimization, as the properties show high practical feasibility and align with the standard's objectives. Experiments were conducted with titanium electrodes according to the methods applied for graphite electrodes. When determining the levels in the experimental designs, trials were first conducted considering the data in the literature. As seen, it was observed that the voltage value increased for the titanium electrode compared to the graphite electrode for the electrolysis process to occur. This is attributed to the distinct physical and electrical properties of the electrode materials, such as overpotential and surface conductivity.

This phenomenon can be further elucidated by the inherent surface characteristics and electrical properties of these materials. Titanium surfaces naturally develop a passive titanium dioxide TiO_2 layer, which exhibits semiconducting properties and creates a significant electrical resistance. This oxide layer acts as a kinetic barrier for charge transfer, necessitating a higher overpotential to initiate and maintain the electrochemical transesterification process (Feltham et al. 2023; Kociubczyk et al. 2015). In contrast, graphite possesses high electrical conductivity and a relatively porous structure that provides a larger effective surface area. These attributes reduce the activation energy required for the reaction, allowing the system to achieve efficient biodiesel conversion at considerably lower voltage levels (Abdollahi Asl et al. 2020; Im et al. 2016). Consequently, while titanium offers higher peak yields, graphite stands out as a more energy-efficient alternative due to its lower voltage requirements."

Table 2 shows the experimental design and biodiesel results.

Table 1 Taguchi L18(2¹ 3³) experimental design and results for biodiesel production using graphite electrodes

Experiment Number	Methanol (wt %)	Reaction time (min)	KOH (wt %)	Electrolysis voltage (V)	Biodiesel conversion yield (%)	Density (kg/m ³)
1	35	30	0.5	20	74.11	870
2	35	30	0.75	25	71.47	888
3	35	30	1	30	63.35	890
4	35	45	0.5	20	77.35	848
5	35	45	0.75	25	76.50	850
6	35	45	1	30	66.94	849
7	35	60	0.5	25	68.68	881
8	35	60	0.75	30	63.15	885
9	35	60	1	20	71.52	838
10	40	30	0.5	30	69.71	867
11	40	30	0.75	20	72.44	860
12	40	30	1	25	71.42	865
13	40	45	0.5	25	82.94	853
14	40	45	0.75	30	84.72	861
15	40	45	1	20	81.45	841
16	40	60	0.5	30	74.88	856
17	40	60	0.75	20	80.13	840
18	40	60	1	25	73.26	845

Table 2 Taguchi L18(2¹ 3³) experimental design and results for biodiesel production with a titanium electrode

Experiment Number	Methanol (wt %)	Reaction time (min)	KOH (wt %)	Electrolysis voltage (V)	Biodiesel conversion yield (%)	Density (kg/m ³)
1	35	30	0.50	40	57.73	885
2	35	30	0.75	50	65.76	870
3	35	30	1.00	60	72.09	860
4	35	45	0.50	40	76.96	852
5	35	45	0.75	50	83.45	843
6	35	45	1.00	60	90.12	831
7	35	60	0.50	50	78.56	844
8	35	60	0.75	60	86.82	835
9	35	60	1.00	40	83.85	841
10	45	30	0.50	60	88.70	829
11	45	30	0.75	40	79.45	845
12	45	30	1.00	50	85.82	832
13	45	45	0.50	50	90.12	835
14	45	45	0.75	60	93.46	816
15	45	45	1.00	40	90.17	838
16	45	60	0.50	60	88.92	814
17	45	60	0.75	40	86.02	825
18	45	60	1.00	50	87.96	815

Upon examination of the table, it is observed that the density results obtained fall within the values specified in the TS EN 14,214+A1 standard. Based on these results, the biodiesel produced from all experiments in the experimental design complies with the standards. All results have been utilized in the optimization process. Using the Taguchi L18 experimental design for optimizing parameters in biodiesel production with graphite and titanium electrodes, 18 experiments were conducted for each electrode instead of a total of 54 experiments. Conducting fewer experiments saved time and prevented excessive chemical usage. Subsequently, the response surface method was used to determine the optimal experimental conditions for each electrode type. The preferred experimental variables for biodiesel production with graphite and titanium electrodes were optimized parametrically within themselves using S/N analysis. While adjusting for maximum biodiesel yield, the approach of minimum density was adopted, and S/N ratios were calculated using Minitab software, followed by variance analysis. The S/N ratio derived for the biodiesel conversion yield of biodiesel obtained in experiments conducted according to the experimental design with graphite electrodes revealed that the variables most affecting the biodiesel conversion yield in graphite electrode production were, in order, 45% methanol by weight, 45 min reaction time, 0.5% KOH, and 20 volts electrolysis voltage.

In the S/N analysis, the experimental variables that most affected the biodiesel density were 45% methanol, 45 min reaction time, 1% KOH, and 20 volts electrolysis voltage. When the S/N ratios for biodiesel conversion yield and density were evaluated, it was determined that the electrolysis voltage was 20 volts for both. Similarly, it was observed

that 45% methanol use was the most effective alcohol ratio for density and yield. However, the difference in base concentration occurred in the KOH ratio. While the best ratio for density was 1%, a 0.5% KOH concentration was effective for biodiesel conversion yield. Another important point considered to determine whether the experimental data was statistically significant was the Adeq Precision (adequate precision) value. In the variance analysis performed with the Minitab programmed, a high “f” value and a low “p” value are expected [22]. Another value that makes the variable meaningful and requires attention is the sum of squares (SS). An increase in this value indicates an increase in the significance of the variable. An F value higher than 4 indicates that the effect of the relevant experimental variable is significant [23]. The effect of the experimental variables on the biodiesel conversion yield of biodiesel produced by the graphite electrode electrolysis method was examined by variance analysis, and the ANOVA table generated by Minitab is given in Table 3.

Upon examining Table 4, it was determined that the p-value of all experimental parameters except KOH was less than 0.05, and that the F-value of variables other than KOH was higher than 4. These results indicate that methanol, reaction time, and electrolysis voltage are statistically significant for biodiesel conversion efficiency in the graphite electrode experiment. Furthermore, when the contribution values were examined, it was determined that reaction time had the most significant effect on biodiesel conversion, with a contribution rate of 32.31%. Table 5 presents the variance analysis for the density of biodiesel obtained from the graphite electrode experiments.

Table 3 Analysis of variance for biodiesel conversion yield (for experiments with graphite electrodes)

Variables	Degrees of freedom	Sum of Squares (SS)	Contribution (%)	F-Value	P-value
A- Methanol (wt %)	1	186.12	28.52	16.81	0.002
B- Reaction time (min)	2	210.85	32.31	9.52	0.005
C- KOH (wt %)	2	44.94	6.89	2.03	0.182
D- Electrolysis voltage (V)	2	99.90	15.31	4.51	0.040
Error	10	110.72	16.97		
Total	17	652.53	100.00		

Table 4 Variance analysis for biodiesel conversion yield (for experiments with titanium electrodes)

Variables	Degrees of freedom	Sum of Squares (SS)	Contribution (%)	F-Value	P-value
A- Methanol (wt %)	1	504.35	33.47	23.22	0.001
B- Reaction time (min)	2	536.03	35.57	12.34	0.002
C- KOH (wt %)	2	70.21	4.66	1.62	0.247
D- Electrolysis voltage (V)	2	179.13	11.89	4.12	0.049
Error	10	217.25	14.42		
Total	17	1506.96	100.00		

Table 5 Variance analysis for biodiesel density (for experiments with graphite electrodes)

Variables	Degrees of freedom	Sum of Squares (SS)	Contribution (%)	F-Value	P-value
A- Methanol (wt %)	1	684.5	14.19	6.51	0.029
B- Reaction time (min)	2	1662.1	34.47	7.91	0.009
C- KOH (wt %)	2	301.4	6.25	1.43	0.283
D- Electrolysis voltage (V)	2	1123.4	23.30	5.35	0.026
Error	10	1050.8	21.79		
Total	17	4822.3	100.00		

When examining Table 5, it is observed that all variables except the KOH experiment variable are significant variables for the density value, as is the case with biodiesel conversion yield. The P value is less than 0.05, while the F value is greater than 4. It was determined that the reaction time contributed the most (34.47%) to achieving the low-density value. According to the S/N and variance analysis results, it was determined that the levels of KOH selected as the experimental variable in biodiesel production from domestic waste frying oil using the graphite electrode electrolysis method were not statistically significant in terms of either biodiesel conversion yield or density. The results

of the S/N analysis, where KOH was found to be 0.5% for biodiesel conversion yield and 1% for density, were consistent with the results of the variance analysis. Although KOH is one of the most effective factors in biodiesel conversion yield in traditional biodiesel production, it was found that the selected KOH levels were not very effective in the electrolysis method. The electrolysis voltage used with graphite electrodes was found to be 20 volts, which is the lowest level selected for both biodiesel conversion yield and density. It was determined that applying 20 V electrolysis voltage with graphite electrodes to a mixture containing 45% methanol by weight for 45 min was the most suitable experimental variable for biodiesel conversion yield and density.

The results obtained from the biodiesel production experiments from domestic waste frying oil using the electrolysis method with a titanium electrode according to the Taguchi experimental design were used in statistical analysis. The results of the S/N and variance analysis performed for both biodiesel conversion yield and density using Minitab were examined. The levels of the variables effective in achieving the highest biodiesel conversion yield in biodiesel production experiments with titanium electrode were determined to be the highest S/N ratios, respectively, at 45% methanol, 45 min reaction time, 1% KOH, and 60 volts electrolysis voltage.

The levels of the experimental variables that most affected the density of the biodiesel obtained from the titanium electrode experiments (with the highest S/N ratio) were determined to be 45% methanol, 60 min reaction time, 1% KOH, and 60 volts electrolysis voltage. It was observed that the only variable that yielded different results between biodiesel conversion yield and density was the reaction time. The highest biodiesel conversion yield was achieved in 45 min, while the lowest biodiesel density was observed at a reaction time of 60 min. This result is considered reasonable for production processes.

The variance analysis for the biodiesel conversion yield of biodiesel produced from titanium electrode experiments is presented in Table 4.

In titanium electrode experiments, it was observed that the experimental variable KOH was statistically insignificant according to the P and F values. It was determined that the reaction time provided the highest contribution to achieving the highest biodiesel conversion yield. The variance analysis performed for the density value is presented in Table 6.

Upon examining Table 6, it is observed that the P value is less than 0.05 and the F value is greater than 4, indicating that the experimental variables are methanol, reaction time, and electrolysis voltage. Furthermore, it was determined that methanol contributed 42.06% to achieving the lowest

Table 6 Variance analysis for biodiesel density (for experiments with titanium electrodes)

Variables	Degrees of freedom	Sum of Squares (SS)	Contribution (%)	F-Value	P-value
A- Methanol (wt %)	1	2496.9	42.06	47.90	0.000
B- Reaction time (min)	2	1918.1	32.31	18.40	0.000
C- KOH (wt %)	2	148.8	2.51	1.43	0.285
D- Electrolysis voltage (V)	2	851.4	14.34	8.17	0.008
Error	10	521.2	8.78		
Total	17	5936.4	100.00		

density value. As in other experiments, KOH was found to be statistically insignificant for the density value.

In conclusion, according to the S/N and variance analysis, the effect of KOH was not statistically significant within the tested range of 0.5–1.0 wt% in the transesterification process carried out with two different electrodes using the electrolysis method. This finding should be interpreted cautiously, as the tested range was intentionally narrow. Broader concentration ranges, including catalyst-free conditions, should be investigated in future studies before drawing definitive conclusions about the role of KOH in electrolysis-assisted transesterification. Furthermore, it was determined that a higher electrolysis voltage is required when using a titanium electrode compared to a graphite electrode. Moreover, it was determined that a high proportion of methanol must be used to achieve the highest biodiesel conversion yield and the lowest density value.

3.2 Response surface methodology (RSM) for regression and optimization

In this study, a second-degree regression model calculating the change in biodiesel conversion yield and density depending on the experimental variables was developed using Minitab RSM analysis. Furthermore, a comprehensive optimization was performed using RSM, considering all experimental variables together. In this section, the results obtained from the graphite and titanium electrode experiments for biodiesel are examined under separate headings. All results obtained from experiments using graphite electrodes and the electrolysis method on domestic waste frying oil were combined and general optimization was performed using RSM. The optimization was designed to achieve the highest biodiesel conversion yield and the lowest density value. The visual representation of the result obtained from the RSM optimization performed using Minitab is shown in Fig. 3.

Based on the response surface graphs provided in Fig. 3, it can be clearly observed that increasing the electrolysis voltage and reaction time initially drives a sharp upward trend in the conversion yield while simultaneously decreasing the density. However, these curves tend to flatten out or show a slight reversal beyond their specific optimum thresholds. Consequently, the optimal experimental conditions for achieving the highest biodiesel yield and the lowest density value were determined to be 45% methanol by weight, a reaction time of 48.18 min, 0.74% KOH by weight, and an electrolysis voltage of 20.3 volts. Under these conditions, the expected biodiesel conversion yield is 84.37%, while the density is expected to be 837.94 kg/m³. Table 7 presents

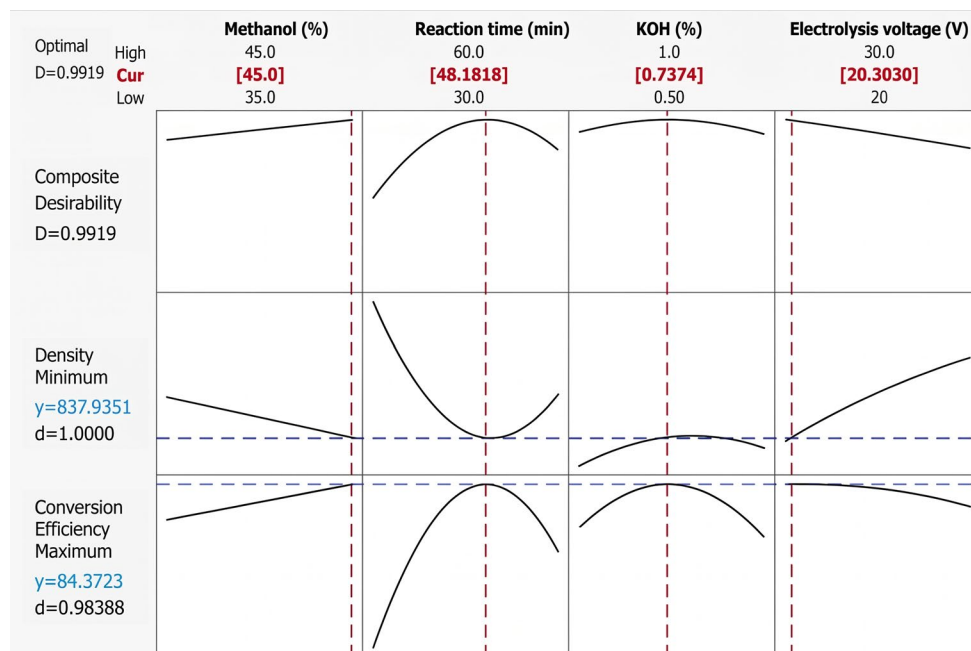
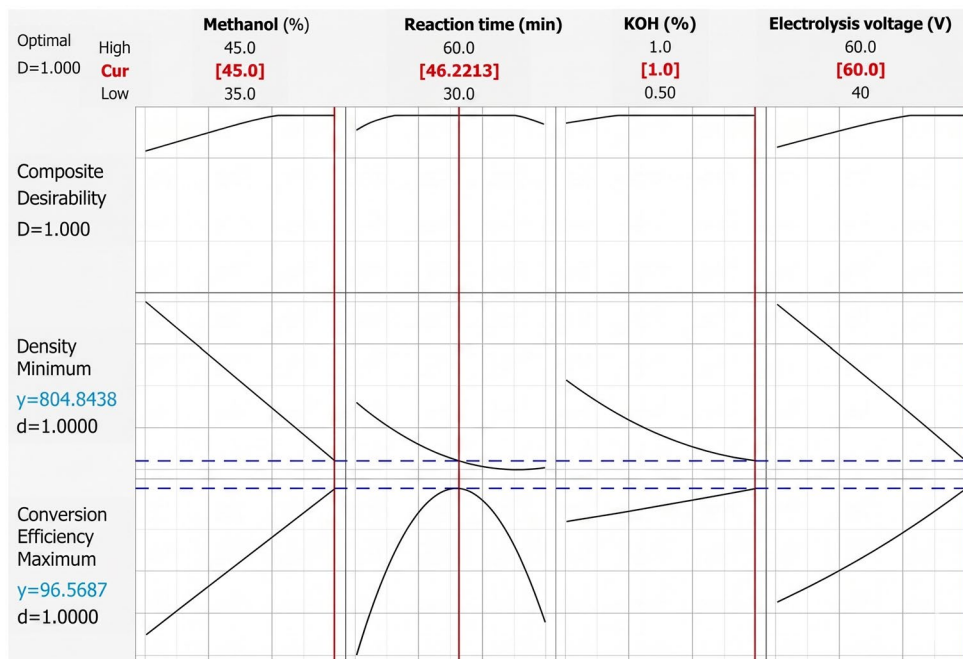
Fig. 3 RSM optimization results for biodiesel production using graphite electrodes

Table 7 RSM optimization summary for graphite electrode experiments

Optimum points of variables			Model Fit Values (y)			Composite Desirability
Methanol (wt %)	Reaction Time (min)	KOH (%)	Electrolysis voltage (V)	Biodiesel Conversion Efficiency (%)	Density (kg/m ³)	
45	48.18	0.73	20.303	84.37	837.94	0.991

Fig. 4 RSM optimization results for biodiesel production using a titanium electrode

the target values predicted by the RSM optimization model for conversion yield and density.

As a result, the optimal experimental parameters for achieving the ideal conversion yield and density in biodiesel production using the electrolysis-assisted transesterification method with graphite electrodes from domestic waste frying oil have been determined. Accordingly, methanol should be obtained by dissolving 0.74% KOH in 45% methanol relative to the weight of the waste frying oil to be used in biodiesel production. Then, acetone should be added as a solvent in an equal amount to half the amount of methanol. Distilled water should be mixed into this mixture in an amount equal to 3% of the oil. Fifty grams of filtered domestic waste frying oil should be added to this mixture, which is stirred at 300 rpm. Electrodes consisting of an anode and a cathode, positioned 10 mm apart, should be immersed in the mixture using a power source. The power source voltage should be set to 20.3 volts, and the experiment should be conducted for 48 min. Experiments should be conducted at ambient temperature. Biodiesel production was carried out under these determined optimal parameters. According to the experimental results, biodiesel with a conversion yield of 83.45% and a density of 843 kg/m³ was obtained. As can be seen, the results are very close to the estimated values. Some fuel properties were calculated numerically based on

GC/MS fatty acid distributions and are presented in the following section.

Figure 4 shows a summary graph of the multiple optimization results obtained using the response surface method (RSM).

Based on the response surface graphs provided in Fig. 4, it is evident that increasing the electrolysis voltage exhibits a strong, continuous upward trend in conversion yield up to the maximum tested limit of 60 V, accompanied by a steady decrease in density. Consequently, according to the optimization results, which targeted the highest biodiesel conversion yield and the lowest density value, it was determined that the optimal conditions were 45% methanol by weight, 1% KOH by weight, a reaction time of 46.22 min, and an electrolysis voltage of 60 volts. The results of the RSM optimization performed based on the titanium electrode biodiesel results are summarized in Table 8.

The optimal experimental conditions found for producing biodiesel using the electrolysis method with a titanium electrode can be utilized. Experiments conducted under these conditions to test the accuracy of the model resulted in a biodiesel conversion yield of 93% and a biodiesel density of 818 kg/m³. The results were found to be close to the target values shown in Fig. 4.

Table 8 Summary of RSM optimization for experiments with titanium electrodes

Optimum points of the variables			Model Fit Values (y)			Composite Desirability
Methanol (wt %)	Reaction Time (min)	KOH (%)	Electrolysis voltage (V)	Biodiesel Conversion Efficiency (%)	Density (kg/m ³)	
45	46.22	1	60	96.56	804.84	1

Table 9 Fatty acid distributions of biodiesels produced with graphite and titanium electrodes

Fatty Acid (Component Name)	Biodiesel Produced with Graphite Electrode (%)	Biodiesel Produced with Titanium Electrode (%)
Nervonic acid (C24:1)	0.71	0.15
Tridecanoic acid (C13:0)	0.01	0.01
Eicosapentaenoic acid (C20:5n3)	0.23	0.28
Lauric acid (C12:0)	0.01	0.00
Butyric acid (C4:0)	0.06	0.01
Linoleic acid (C18:2n6c)	44.09	35.79
Palmitoleic acid (C16:1)	0.04	0.01
Undecanoic acid (C11:0)	0.03	0.02
Docosadienoic acid (C22:2)	0.14	0.39
Arachidonic acid (C20:4n6)	0.07	0.06
Eicosadienoic acid (C20:2)	0.15	0.04
Pentadecanoic acid (C15:0)	0.02	0.01
Tricosanic acid (C23:0)	0.08	0.18
Oleic acid (C18:1n9c)	23.04	22.55
Elaidic acid (C18:1n9t)	0.04	0.01
Pentadecenoic acid (C15:1)	0.01	0.01
Eicosatrienoic acid (C20:3n6)	0.03	0.79
Heicosanoic acid (C21:0)	0.17	1.13
Arachidic acid (C20:0)	0.11	0.01
Heptadecenoic acid (C17:1)	0.05	0.02
Capric acid (C10:0)	0.02	0.03
Heptadecanoic acid (C17:0)	0.22	0.08
Alpha-linolenic acid (C18:3n3)	0.12	0.02
Erucic acid (C22:1n9)	23.73	12.36
Stearic acid (C18:0)	0.06	0.19
Caproic acid (C6:0)	0.46	0.08
Docosahexaenoic acid (C22:6n3)	0.05	0.79
Eicosatrienoic acid (C20:3n3)	0.07	8.9
Behenic acid (C22:0)	0.13	0.06
Myristic acid (C14:0)	0.07	0.17
Gamma-linolenic acid (C18:3n6)	0.22	0.22
Eicosenoic acid (C20:1)	0.24	4.55
Lignoceric acid (C24:0)	5.35	4.90
Palmitic acid (C16:0)	0.01	6.13
Myristoleic acid (C14:1)	0.08	0.03

The predictive accuracy of the developed RSM models was statistically validated. To ensure a concise presentation, the extensive second-order polynomial equations were omitted, while their performance indicators were evaluated. For the graphite electrode experiments, the adjusted coefficient of determination (R_{adj}^2) reached 95.8% for conversion yield and 88.0% for density. The titanium electrode models

exhibited even higher correlation, with R_{adj}^2 values of 98.2% and 95.5% for yield and density, respectively. These values, being significantly above the 80% threshold, indicate that the regression models provide an excellent fit to the experimental data and offer high reliability for predicting optimal process conditions.

3.3 Comparison of fuel properties

This section presents the gas chromatography-mass spectrometry (GC-MS) analysis results of samples obtained from biodiesel production experiments conducted using graphite and titanium electrodes. Gas chromatography-mass spectrometry (GC-MS) analyses are a powerful technique that enables the qualitative and quantitative determination of the fatty acid composition in biodiesel samples. Thanks to these analyses, the ratios of saturated and unsaturated fatty acids present in biodiesel can be clearly determined. The percentage distribution of fatty acids in biodiesel according to both electrode types was determined using GC-MS analyses. Table 9 shows the average area percentage values for each fatty acid component.

Table 9 shows the comparative average percentage distributions of the fatty acid profiles obtained from GC-MS analyses according to electrode type. In this analysis, the fatty acids detected in the biodiesel samples were determined based on their carbon chain lengths and double bond numbers. Although similar fatty acid compositions were observed in biodiesel samples obtained using graphite and titanium electrodes, significant differences emerged in terms of ratios. For example, based on the GC-MS analysis, significant differences were observed in the fatty acid ratios despite similar compositions. Linoleic acid (C18:2n6c), the most dominant unsaturated component, was found at 44.09% in biodiesel produced with graphite electrodes and 35.79% with titanium electrodes. This suggests that the graphite electrode environment may favor the transesterification of polyunsaturated fatty acids. Additionally, high proportions of other unsaturated components, such as oleic acid (23.04% for graphite and 22.55% for titanium) and erucic acid (23.73% for graphite and 12.36% for titanium), were recorded. Regarding saturated fatty acids, palmitic acid (C16:0) was notably higher in the titanium samples (6.13%) compared to graphite (0.01%), which is thought to be associated with the slightly higher density observed in

Table 10 Fuel properties of s based on oil acid distribution

Fuel properties	Graphite Electrode	Titanium Electrode	EN 14,214
Cetane Number	52.03	53.25	min. 51
Viscosity (mm ² /s)	3.53	3.65	3.5–5
Cold Flow Point (CFPP, °C)	-26.86	-23.67	-
Density (kg/m ³)	839.62	843.18	860–900
Iodine value (g I ₂ /100 g oil)	102.35	90.21	max. 120

titanium-based biodiesel. These components are saturated fatty acids that affect the oxidative stability and density of biodiesel and, being present in higher proportions in titanium electrode biodiesel, can be associated with higher density. Therefore, the ratios of saturated and unsaturated fatty acids directly affect not only the yield of biodiesel but also fuel properties such as viscosity, density, and fluidity.

In our study, the fuel properties of biodiesel samples produced using different electrode materials (graphite and titanium) were experimentally determined through analyses conducted in a laboratory environment. For both samples, basic fuel parameters such as cetane number, viscosity (at 40 °C), cold flow point (CFPP), density (at 15 °C) and iodine number were tested using methods compliant with international standards. The measurements revealed that the biodiesel samples obtained with graphite and titanium electrodes showed a close alignment with the limit values specified in the EN 14,214 European biodiesel standard. While the density values (839–843 kg/m³) were slightly below the lower limit of the standard (860 kg/m³), this deviation is attributed to the high conversion purity and the thermal history of the waste cooking oil. However, the values obtained for other critical parameters, such as viscosity (3.5–5.5 mm²/s) and iodine value (90–120 g I₂/100 g oil), fall strictly within the required range, indicating that the biodiesel is suitable in terms of engine performance and storage stability. These experimental findings reveal that the type of electrode used has a decisive effect on fuel quality. Comparative data on the fuel properties in question are presented in detail in Table 10.

Within the scope of the study, the fuel properties of biodiesel samples obtained using two different electrodes, graphite and titanium, were tested in a laboratory environment. As a result of the experimental analyses performed, critical performance parameters such as cetane number, viscosity (at 40 °C), cold flow point (CFPP), density (at 15 °C) and iodine number were obtained and are presented in Table 10. Both samples largely meet the limit values specified in the EN 14,214 biodiesel standard. To maintain experimental precision, density and fuel property measurements were performed in triplicate and duplicate, respectively. The observed variations in fatty acid profiles between electrode types are thought to be influenced by the material-specific

overpotentials and surface structures. Such differences suggest that the electrode material may potentially function as a selective electro-catalyst, thereby affecting the molecular distribution of the resulting biodiesel. In particular, the cetane number was determined to be 52.034 for biodiesel produced with a graphite electrode and 53.251 for biodiesel produced with a titanium electrode, both values being above the minimum limit of 51. This result indicates that biodiesel will exhibit shorter ignition delays and smoother combustion characteristics in diesel engines. The viscosity values were measured as 3.535 mm²/s (graphite) and 3.653 mm²/s (titanium) for the two samples, respectively. Both values fall within the range of 3.5–5 mm²/s and are at a level that will provide good atomization compatible with the injection system. The cold flow point (CFPP) was determined to be -26.86 °C for biodiesel with graphite electrodes and -23.67 °C for biodiesel with titanium electrodes. These low CFPP values indicate that the fuel can flow without clogging filters and operate smoothly in vehicles, particularly in cold climatic conditions. In terms of density, both samples fell slightly below the 860–900 kg/m³ range specified in the EN 14,214 standard; biodiesel produced with graphite electrodes had a density of 839.621 kg/m³, while biodiesel produced with titanium electrodes had a density of 843.184 kg/m³. This may be due to the high unsaturated fatty acid content and the linear nature of the methyl ester chain structure. Regarding the fuel density, the measured values (839–843 kg/m³) were observed to be slightly below the lower limit of the EN 14,214 standard (860 kg/m³). This deviation is attributed to the high biodiesel conversion yield of the electrochemical process, which effectively transforms complex triglycerides into high-purity Fatty Acid Methyl Esters (FAME). Furthermore, the thermal history of the waste cooking oil used as a feedstock contributed to a lighter molecular weight distribution in the final product. From a technical standpoint, this lower density can be considered an advantage for diesel engine performance, as it typically enhances fuel atomization during injection. Smaller fuel droplets lead to better air-fuel mixing and more complete combustion, potentially reducing soot and particulate emissions. Therefore, despite the minor numerical deviation, the produced biodiesel demonstrates high suitability for practical engine applications. Finally, the iodine value was determined to be 90.216 g I₂/100 g oil for biodiesel produced with a titanium electrode and 102.352 g I₂/100 g oil for biodiesel produced with a graphite electrode. Both values are below the 120 limit and are acceptable in terms of oxidative stability. This parameter is particularly important in predicting the risk of biodiesel degradation during storage. These results show that the electrode material used has a decisive effect not only on the biodiesel conversion yield but also on the fuel quality of the biodiesel obtained. The titanium electrode enables the

production of more stable and viscosity-balanced biodiesel, while the graphite electrode offers advantages, particularly in terms of cold weather performance.

To evaluate the industrial viability of the electrochemical transesterification process, the Specific Energy Consumption (SEC) was calculated for both electrode materials under optimized conditions. The SEC (kWh/kg) was determined using Eq. (1);

$$SEC = \frac{V \cdot I \cdot t}{1000 \cdot m_{biodiesel}} \quad (1)$$

where V represents the applied voltage (V), I is the average current (A), t is the reaction time (h), and $m_{biodiesel}$ is the mass of purified biodiesel obtained from 50 g of waste cooking oil. For the graphite electrode, operating at 20.3 V for 48 min with an average current of ~ 0.65 A, the SEC was approximately 0.25 kWh/kg biodiesel. In contrast, the titanium electrode, which required a higher voltage of 60 V for 46 min with an average current of ~ 1.2 A, resulted in an SEC of approximately 1.19 kWh/kg. From a practical feasibility standpoint, graphite offers a clear advantage for low-cost, energy-efficient production, making it suitable for small-scale or decentralized energy systems where minimizing operational costs is prioritized. Conversely, titanium electrodes, despite having ~ 4.7 times higher SEC, achieved a significantly higher conversion yield of 93% compared to graphite's 83.45%. This high-yield performance of titanium justifies its use in industrial applications where high purity and maximum feedstock recovery are paramount, potentially offsetting the higher energy costs through improved product throughput and lower post-processing requirements.

4 Conclusion and recommendations

This study successfully demonstrated and compared the efficacy of graphite and titanium electrodes for the electrolysis-assisted production of biodiesel from waste cooking oil using Taguchi L18 orthogonal design and Response Surface Methodology, revealing critical trade-offs between energy efficiency and maximum conversion yield.

- Graphite electrodes operated optimally at a low voltage of 20.3 V for 48 min, achieving a satisfactory experimental biodiesel yield of 83.45% with an exceptional energy efficiency of approximately 0.25 kWh/kg Specific Energy Consumption.
- Titanium electrodes required a significantly higher overpotential of 60 V for 46 min to overcome the passive oxide layer, resulting in a superior experimental conversion yield of 93%, but at the cost of an approximately

4.7 times higher Specific Energy Consumption of 1.19 kWh/kg.

- Comprehensive fuel property characterizations, including cetane number, kinematic viscosity, and iodine value, confirmed that both electrode materials produced biodiesel closely aligning with EN 14,214 standards. Both exhibited minor acceptable deviations in density and provided excellent cold-flow properties with a CFPP of -26.86 °C for graphite and -23.67 °C for titanium.
- Statistical analysis revealed that within the 0.5 to 1.0 wt% range, the KOH catalyst concentration was statistically insignificant for both electrodes, suggesting that the electrochemical generation of methoxide and hydroxide ions heavily drives the reaction.
- Graphite is highly recommended for decentralized, cost-sensitive, and energy-efficient operations, whereas titanium is better suited for industrial-scale applications where maximizing waste-to-fuel recovery justifies higher initial and operational energy expenditures.
- To fully validate the sustainability and scalability of this method, future research must incorporate comprehensive Techno-Economic and Life Cycle Assessments. Furthermore, investigating catalyst-free conditions, the effect of electrode spacing, and the ratio of auxiliary solvents like acetone will be critical next steps in process optimization.
- Long-term durability and stability tests of both electrode materials under continuous operation cycles are highly recommended to evaluate the structural integrity of graphite and the passivation behavior of titanium over extended periods.
- Transitioning from a batch reactor approach to a continuous flow electrochemical cell design would provide essential engineering data for commercial implementation and continuous byproduct recovery such as high-purity glycerol.

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Data availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical approval Not applicable. This study does not involve human participants or animals.

Consent for publication Not applicable.

Consent to participate Not applicable.

Competing interests The authors declare no competing interests.

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