Environmental and Chemical Changes in Palm Oil Biodiesel using Homogeneous Catalysts

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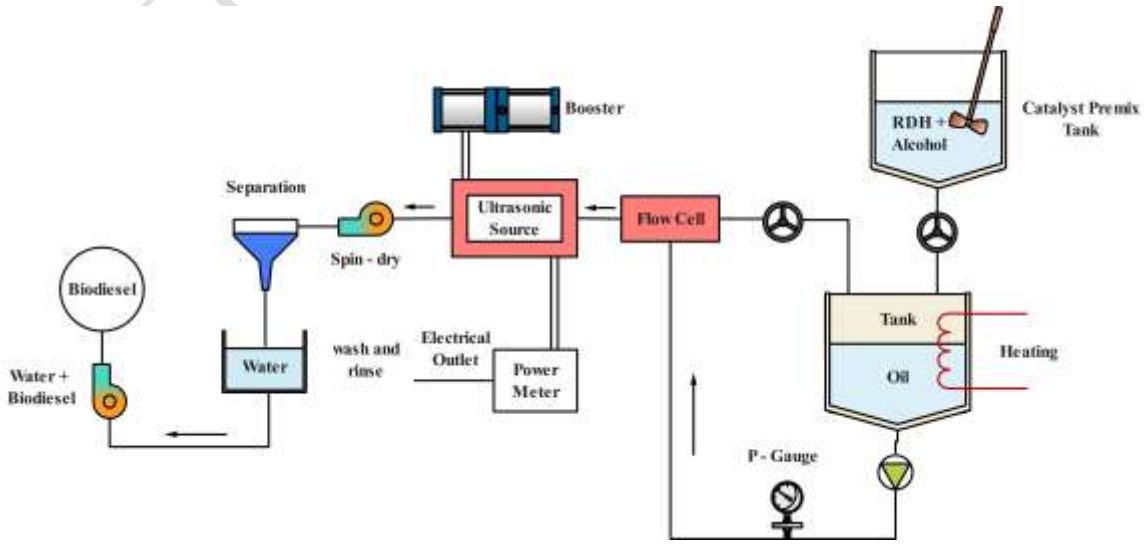
GRAPHICAL ABSTRACT



Abstract:

The production of biofuels in several nations has increased dramatically in recent decades. Their characterization necessitates the use of distinct assessment methods. A commercial diesel specimen is compared to the properties of the biodiesel using electrochemical impedance spectroscopy, linear scanning voltammetry, and open circuit potential. During the biodiesel production process using homogeneous catalysts, emissions of volatile organic compounds (VOCs) and particulate matter can occur. VOCs and particulate matter are harmful air pollutants that can have negative health impacts on humans and the environment. Here, ultrasound was employed to propel a transesterification process among palm oil and methanol. High performance liquid chromatography (HPLC) measures triglyceride conversion into biodiesel; a molar ratio of 7:1 of methanol to oil (M:O) and a reaction period of 80 minutes at 60˚C yields a 94.16% yield. There was 1g of potassium hydroxide catalyst for every 100g of oil. The open circuit potential (OCP) values for biodiesel samples were frequently below 810 mV, and the stabilization periods were less than 2 minutes, as shown by linear sweep voltammograms (LSV). In order to better understand the properties of biofuels and related materials, electrochemical impedance spectroscopy (EIS) was created and it discovered a capacitive system with a typical impedance of MΩ cm^2 at low frequencies.

Keywords: electrochemical; conversion; voltammetry; impedance; potential; biodiesel; sustainable fuels;

1. Introduction

Use of a heterogeneous natural mineral rock (NMR) catalyst in the conventional and ultrasonic generation of biodiesel from used cooking oil (UCO) [1]. Ultrasonic transesterification was evaluated by authors [2] for its potential to produce biodiesel from Syagrus cearensis almonds, also known as Catolé. To make this process more wide applicable on an industrial scale, researchers are investigating a number of its components, including catalyst utilization, influencing parameters, starting materials, reaction stages, kinetics, and modelling [3].

Creation of biodiesel from vegetable oil using ultrasonic aided transesterification is rapidly gaining attention as a superior method due to its high efficiency, reliability, and

speed [4]. To make biodiesel from Manilkara zapota seed oil (MZO), the ultrasonic process is the best option [5]. Using ultrasonic energy to refine oleander oil into biodiesel is the most efficient process [6]. The highest yield of neem biodiesel (98.01%by weight) and improved physiochemical properties were both achieved with the ultrasonic transesterification method [7].

The mahua oil (Madhuca indica) used to make the biodiesel underwent a two-step esterification and transesterification procedure to remove the fatty acids and create a more usable form of the oil. [8]. The highest conversion yield for the flax seed biodiesel was 93%, and the impact of varied moisture content on production showed that the sample with 4% moisture content gave the best biodiesel yield [9]. Ultrasonic transesterification of WCO into FAME has been reported, along with its kinetics [10]. Both acid-esterification and base-transesterification are required in the two-step process that yields biodiesel [11], [12].

By subjecting epoxidized oleic acid to continuous hydroxylation, it was possible to convert palm oleic acid into a polyol with a high yield [13]. Reusability studies have shown that the heterogeneous catalyst may be recycled at least nine times. To minimize the impact on air quality, it is important to use catalysts that are environmentally friendly and comply with regulations. The use of solid heterogeneous catalysts can be an alternative to homogeneous catalysts, as they generally produce fewer emissions and are easier to separate and reuse. [14]. Under the 40 % FFA conditions, the FFA was substantially reduced from 89.16wt% to 1.75wt%. Amberlyst-15, methanol-to-oil molar ratio of 5:1, stirring speediness of 300 rpm. Amberlyst-15's esterification reaction catalyzing properties suggest it may be used to lessen the FFA content of POME [15]. Low grade crude palm oil (LGCPO) seems like a great choice as feedstock for biodiesel synthesis because it is cheap and doesn't compete with food resources [16].

Authors [17] examines methanol-oil molar ratio, catalyst concentration, baffles, reaction temperature, reaction volume and total reactant flow rate were all investigated as they pertained to FAME yield. The biodiesel synthesis process was investigated by researchers [18], who used three different heterogeneous catalysts—zeolite, zinc oxide,

and aluminium oxide—to trans-esterify palm oil and methanol. Used oil from the kitchen has a high monetary value that can be recovered through recycling. [19].

The tandem synthesis catalytic performance of TsOH-H2SO4 was comparable to that of TsOH, and it was recoverable for reuse, whereas TsOH was not [20]. Microreactor Pd-supported catalyst preparation for renewable bio-hydrogenated diesel (BHD) [21].

The byproduct of the crude palm oil (CPO), palm fatty acid distillate (PFAD), has great potential use because of it production in large quantities. The researchers [22] concentrated on Saturated fatty acid concentrations at the sn-2 location and triacylglycerol compositions both showed randomization of the fatty acids.[23]. Traditional methods of producing fatty acid ethyl ester involve transesterification using a heterogeneous alkali catalyst [24]. NaOH and KOH are examples of homogeneous catalysts, and CaO, super base CaO, and Zeolite are examples of heterogeneous catalysts[25]. Since the fatty acid content of the blends was stable, undesired processes including degradation and trans isomer formation were prevented. [26]

Authors [27] examined the impact on activated biochar's iodine number of two activating agents, four acid concentrations, and five activation timeframes (from coconut shell and Randu wood). Researchers [28] reported that 65°C temperature, 1:6 mole ratio, 120 minutes of reaction duration, and a 5% (w/w) heterogeneous catalyst ratio yielded ideal operation conditions for biodiesel generation employing the KIO_3 heterogeneous catalyst.

Catalysts were examined using gas sorption analysis, FTIR, and XRD [29]. Laboratory-scale batch reactor experiments were performed to analyze the transesterification of palm oil with methanol catalyzed by KOH by authors [30] look into how tetramethylammonium bromide (TMAB) improves the final biodiesel product's wash ability by reducing foam. According to research [31], the usage of a heterogeneous acid catalyst will greatly facilitate the esterification of PFAD to biodiesel. The unique process of producing high-quality biodiesel from palm olein was analysed and shown to be more environmental friendly by literatures [32]. According to the results of an activity test conducted by authors [33] it was determined that the bio-naphtha created using the suggested process has only trace amounts of aromatic components and almost no sulphur.

Authors [34] looked at the effect of a strong base catalyst, sodium silicate produced from Si-rich geothermal sludge, in the transesterification of palm oil with methanol. Co/ZSM5 as a catalyst for the translation of CPO to Fatty Acid Methyl Ester is still inferior to basic catalyst, according to research [35]. By contrast, when the catalyst was employed in the transesterification process with UCO, the output of FAME increased to 41.87%. CaO was produced as a catalyst by reacting methanol with calcined quick lime as studied by authors [36]

Using innovative Si-based heterogeneous catalysts generated from Imperata cylindrica, authors [37] analyzed the biodiesel produced from palm oil mill sludge (POMS). To reduce reliance on palm oil, researchers [38] establishes papaya and rambutan seed oils' viability as biodiesel feedstock. Esterification of palm fatty acid distillate (PFAD) to create PFAD methyl ester / biodiesel was studied in depth by authors [39] who looked at the impact of microwave irradiation energy. Ultrasonic analysis was performed by researchers [40] before KOH, Na2SiO3, palm oil, and methanol were heated in a water bath to 60°C.

Biodiesel generation using commercial heterogeneous catalysts like calcium oxide (CaO) and potassium phosphate (K_3PO_4) [41] using an ultrasound-assisted reactor (US). Trimethylolpropane ester (TMP ester), derived from palm oil, was analyzed by authors [42], and found to have an iodine value of 66.4 g/100 g after being epoxidized.

Biodegradable base-stock for bio-lubricant may be found in Trimethylolpropane (TMP) esters [43]. It has been speculated that using heterogeneous catalysts for the manufacture of TMP esters is the key to achieving environmental friendliness. Based on their investigation, Authors [44] concluded that transesterification of high oleic palm oil methyl esters (POME) with trimethylolpropane(TMP) is the chemical pathway leading to TMPTE production.

Vegetable oil methanol transesterification was studied, and a SO42-/ZnO catalyst was developed [45]. By fine-tuning the reaction time and the catalyst/oil weight ratio, we were able to maximize the biodiesel (fatty acid methyl ester) production. For more pure glycerol, the free ions from the salt and catalyst were analyzed by researchers [46-47] and

removed by an ion exchange method employing Amberlite resins. FTIR analysis was also performed to ensure sample purity and identify any remaining impurities [48-49].

This experiment used a linear sweep voltammeter, open-circuit potential, and EIS with the aid of a potential stat- Galvano stat to assess biodiesel. Ultrasound and KOH were used to catalyze transesterification between palm oil and methanol at two variant temperatures.

2. Experimental materials and methods

2.1. Materials

Commercially available palm oil contains the following fatty acid percentages by weight: stearic (2.95%), palmitic (6.2%), oleic (17.35%), palmitoleic (0.05%), and linoleic (73.45%). The reagents used were of analytical quality. The resistivity of the aqueous solutions was measured using MilliQ water, which was 18.2 MΩ cm. Anhydrous methanol (CH3OH), containing not more than 0.003% water, and anhydrous potassium hydroxide (KOH), containing greater than 99.97% KOH, were used to transform palm oil into biodiesel.

2.2. Analysis of Physicochemical process

ISO 660, ISO 3104, and ISO 3675 were used to determine the biodiesel's acidity index, density, and viscosity, respectively. Density readings for the biodiesel used in the experiment were obtained in g/cm^3 using the international standard ASTM D4052. This standard sets the density at 20°C as 0.860-0.900 g/cm^3. The ASTM specification D613 was used to calculate the kinematic viscosity at 40°C, and the results were given in units of mm^2s^{-1}. By determining how much potassium hydroxide (0.05 N) was required to neutralize the free fatty acids (oleic acid) in 1 gm of material, the acid number was calculated. Reversed-phase HPLC with a UV detector at 205 nm was employed to calculate the triglyceride conversional rate. For the HPLC study, authors utilized a system from German manufacturer Agilent, the 1100. Need a degasser and reservoir tray to run Chemistation version 15 in addition to a Windows PC. To analyse the biodiesel's triglyceride composition, 38 µL of the fuel was blended with 962 µL of a hexane/propanol combination (4 parts hexane/5 parts 2 propanols) in 1 cc vials with

screw lids made for HPLC machines. Triglycerides in oil and biodiesel can be converted into their respective percentages by measuring the area between them.

2.3. Production of Biodiesel

In an Erlenmeyer flask, 12.5 mL of methyl alcohol was mixed with 0.41 g of potassium hydroxide (KOH) for each test. Authors then added 37.5 mm of palm oil to the resulting potassium methylate and brought the total amount to 50 mm. The UP 200S ultrasonic instrument was used to combine the ingredients. The Sonotrode S26d14 was used to administer the ultrasonic treatment, which was carried out at a frequency of 24 kHz for 20, 40, 60, and 80 minutes. At each of the three temperatures (23°C, and 60°C), the tests were performed in triplicate. The specimen was centrifuged at 3000rpm for 3 minutes in an IEC centrum CL2 centrifuge. Separation and analysis of the resulting glycerin and biodiesel followed.

Separation was achieved by diluting the biodiesel with water (1:1) and centrifuging it at 3000 rpm for 2.5 minutes. Data on each sample's lab performance, physicochemical analysis, and rate of conversion was entered into Excel and analyzed using Microsoft 365's built-in statistical tools to yield descriptive statistics like mean and standard deviation. Figure1 depicts a typical setup for producing biodiesel on a small scale. A 20-liter processing vessel, 1.5-kilowatt heating element, 2-liter stirring catalyst premix tank, 1-horsepower (HP) centrifugal pump with variable-speed drive are all needed for this approach, as well as a back-pressure regulator for controlling pressure in the stream cell and a sup-pressor. High density polyethylene was used to make the processing and premix tanks. The system included a centrifuge for separating the biodiesel and glycerin and for blending the wash water and biodiesel.

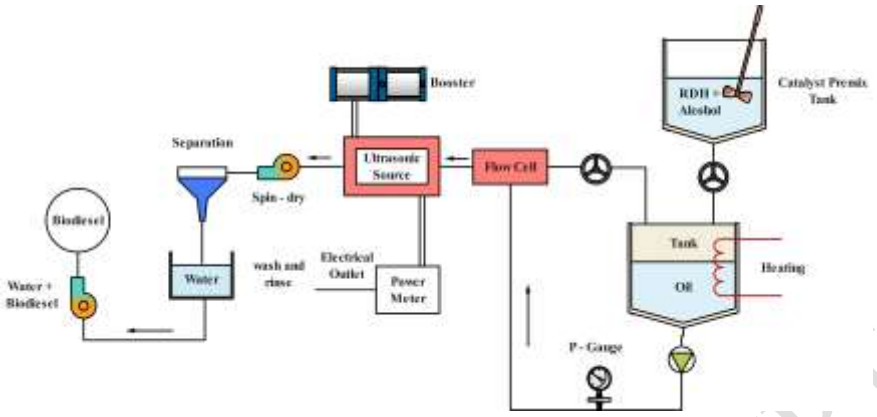


Figure 1. Schematic view to generate biodiesel using ultrasound.

2.4. Analysis of Electrochemical methods

In a situation where oxidation / reduction rates are balanced, no net current will flow through the system, and this is the electric potential measured by an open circuit tester. Using linear sweep voltammetry, authors were able to detect the electroactive types present in biodiesel and conventional diesel. The experiments were performed by changing the OCP to anodic and cathodic potentials. Experiments were started at the OCP of each sample and a 1.0 V voltage sweep was performed at 2 mVs^{-1}. Because the majority of electrochemical reactions can be observed at a difference of only 1 V from the OCP, this potential range was selected. Since the electrochemical impedance test may only be done in a static setting, the sweep rate was selected accordingly. An electrochemical impedance spectroscopy (EIS) was conducted by spread on a sinusoidal disturbance of 200 mV root mean square at 30 kHz, 1 Hz, and 7 points per decade in between. This disarray enabled experiments to be conducted while the system was in a steady state and also caused enough disturbance to be detectable with a potentiostat. We let each system five minutes to stabilize before taking any readings.

Sampling was performed at 60°C, and linear sweep voltammetry, open circuit potential, and EIS were used to test the material's stability. Researchers purchased regular fossil fuel to use as a reference. All samples were stabile for one hour to warm up to room temperature before any measurements were taken. Both electrodes were placed in a glass electrochemical cell that held 12 mL of solution and had a compartment constant of 0.014 cm^{-1}. Due to the lack of anticipated electrochemical reactions and the desire to

design a cheap cell, 304 stainless steel sheets were employed to make the electrodes. The studies were run on a potentiostat - galvanostat manufactured by Gamry and controlled by Gamry Framework software.

3. Results and Discussion

3.1. Results of Electrochemical properties

3.1.1. Impact of Electrochemical Impedance Spectroscopy analysis

The Figure 2 depicts the Nyquist plots of synthetic biodiesel and regular fossil diesel.

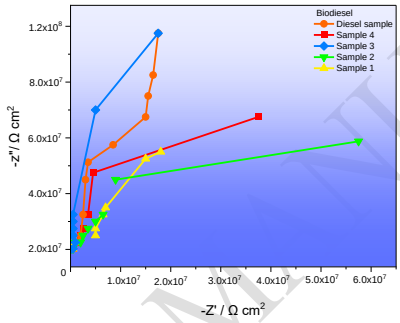


Figure 2. experimental data with an electrochemical impedance spectrum

The spectra shown in Figure 2 were fitted using the Levenberg-Marquardt technique, and even in the worst-case scenario, the chi-squared value reached 0.08. All of the systems in Figure 2 have low-frequency impedances of the order of M cm^2 or less. Impedance spectroscopy can be used to variation among biodiesel, pseudo-binary and diesel mixes in the less-frequencies domain. High-frequency analysis is required for biodiesel because diesel resistance predominates in the impedance at these frequencies. Capacitive impedance makes the largest contribution to the whole system in the mid-range frequency range; hence this is helpful information for assessing the capacitance capacity of the materials. Investigating the system's charge transfer resistance in the low-frequency band reveals information about the ease with which electrochemical procedures can be carried out. Diesel resistance (R1) is parallel to the nonideal double-layer capacitance, and the resistance to charge transfer (R2) due to electrochemical

processes is connected in series with R1. Figure 2 displays, after considering all of the solution's parameters, the R1 and for the electrochemical process.

Diesel solutions were consistently at a 10^5 Ωcm^2 order of magnitude, as seen in Figure 3. This means that there were no conductive species present in any of the samples. The secondary axis further demonstrates the extremely high charge transfer resistance seen in all cases, with values of 10^8 Ωcm^2 corroborated by voltammetry in circumstances when no electroactive chemicals were detected. It has been observed that the charge transfer resistance of systems containing electroactive species is on the order of 10^3 Ωcm^2, but can reach as high as 10^9 Ωcm^2 depending on the measuring parameters, such as the exposure period. Biofuels and comparable materials can be better characterized with the use of this data. A single frequency measurement, like that of an electrolytic conductivity meter, can provide some insight into a material's electrochemical composition.

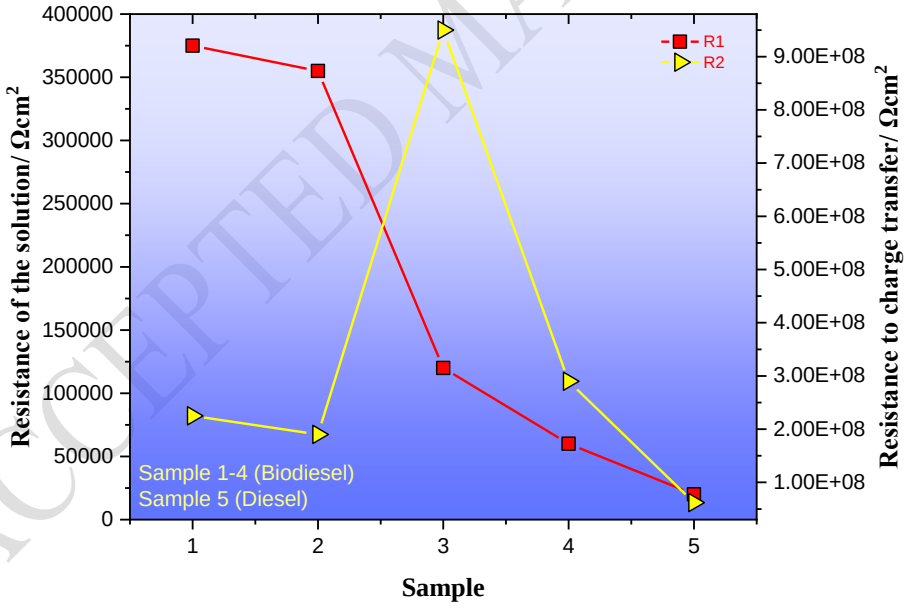


Figure 3. Measured Bio-diesel sample resistance were fitted to measure diesel and charge transfer resistance.

3.1.2. Impact of Linear Sweep Voltammetry analysis

Results from a linear sweep voltammetry (Sweep speed 2 mV s^{-1}) are shown in Figure 4. Cathodic and anodic scans up to 1.0 V both showed nanoampere currents, which can be explained by lingering pollutants or electroactive components. That biodiesel and other fuels don't exhibit distinctive reaction peaks in the same possible windows is to be expected. Sample 1's low resistance in the open circuit potential experiment suggests that it will exhibit the highest values. Low current densities may be seen in the voltammograms of samples 2, 3, and 4. The reduced current values seen in the voltammograms of the commercial sample are consistent with the hypothesis that these chemicals are even less electroactive or inert.

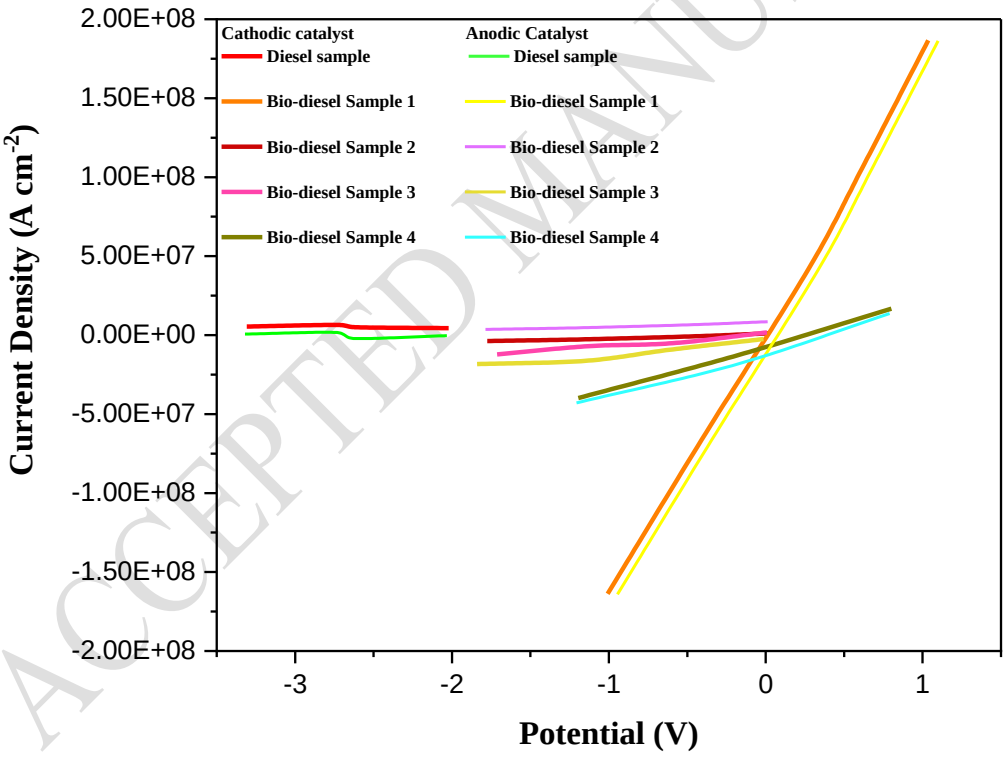


Figure 4. Comparision of voltage output between diesel and biodiesel.

Certain natural oils have been shown to have current densities three orders of magnitude bigger than our systems (μA/cm^2). The results of this study have ramifications

for the categorization of natural and synthetic biofuels, fuels, and oils. The Nano Ampere is an appropriate current density unit for this substance.

3.1.3. Impact on Potential of Open Circuit

Potential behavior in open circuits is depicted in Figure 5 for all samples. All samples reached equilibrium before 120 s, demonstrating that the system quickly stabilizes under any conditions. This rule out the possibility of time-consuming, complicated electrochemical reactions or methods involving the cumbersome reassignment of loads in the double layer. According to reports, a longer period of time is required to stabilize a more complicated system. Since stabilization took place in such a short period of time, it is reasonable to assume that no reactions took place at the interfaces; nevertheless, this conjecture will need to be confirmed using linear sweep voltammetry to establish its validity. When the potential was held constant, authors found that Bio-diesel of Sample 1 had a value of around 40 mV, Sample 2 was around 260 mV, Sample 3 was around 810 mV, Sample 4 was around 730 mV, and the diesel sample was over 2100 mV. These appears to be a significant decline in potential for samples 2, 3, and 4. No potential change was seen between the electrodes in sample 1, as would be expected when utilizing two electrodes of the same type. It's safe to presume that the first sample contains species that add to the sample's electrolytic conductivity. The charge resistivity step was higher in samples 2, 3, and 4, suggesting that they contained fewer electroactive substances. Compared to older samples, newer ones have a drop potential that is roughly three times as great because the electroactive species composition is dropping. Hydrocarbon components that give higher electrolyte resistances are likely to reason for these large variations from the findings published for commercial diesel.

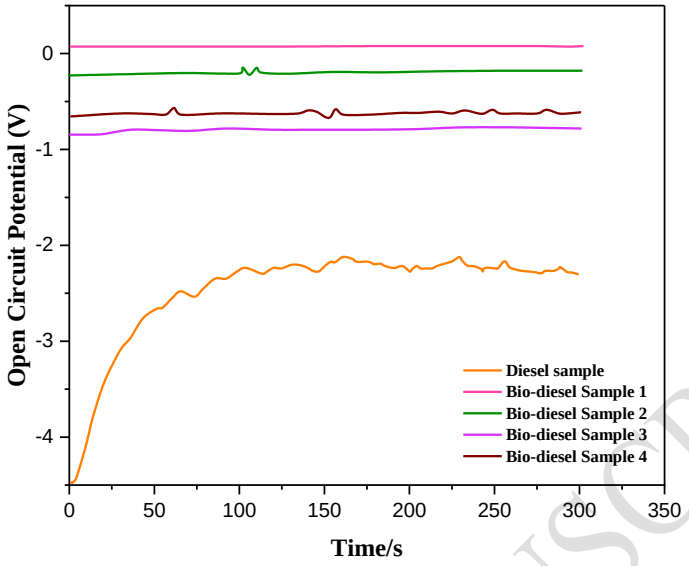


Fig. 5. Obtained open circuit potential.

3.2. Results of Chemical and Physical Characteristics

An outcome of ultrasonic transesterification at 23°C and 0.41g of KOH per sample are shown in Table 1. As a result of the triglyceride-to-methyl-ester conversion reaction, mono- and diglycerides are produced as byproducts; the percentage of conversion, X, represents the magnitude of this fraction (%). Though the density percentage was somewhat higher than ES 14214 in the 20-minute tests.

Table 1. Outcome of trans-esterification at 23°C.

Sample number	Time (minutes)	Biodiesel Density	Acidity Index	Kinematic Viscosity	Percentage of Oil Yield	Conversion of Triglycerides
		20°C (g cm^{-3})	(mg KOH g^{-1})	40 °C (mm^2 s^{-1})	(wt.%)	(wt.%)
1	20	0.93	0.43	4.93	64.06	98.81
2	40	0.91	0.41	4.65	66.42	99.26
3	60	0.96	0.39	4.51	67.28	99.45
4	80	0.88	0.36	4.32	69.05	99.59

Table 2 displays the results of ultrasonic transesterification at 60°C. Initial results from a 20-minute test specimen show that the triglyceride conversion percentage, X (%),

is >99 %, resulting to a density parameter of 0.91g cm^{-3} and a kinematic viscosity of 4.23 mm^2s^{-1}, both of which are within the range allowed by ES 14214.

Using ultrasound transesterification in a batch reactor, authors showed that a with a drastic cut in reaction time and excellent conversion efficiency, producing biodiesel with a purity of 99.9% is feasible. The longer the test ran, the higher the percentage (by volume) of glycerin generated, which meant more biodiesel was made. It took 60 minutes to reach the ideal density of 0.81g cm^3. After only 90 minutes of reaction time, authors found biodiesel yields of 92%.

Table 2. Outcomes of transesterification at 60°C

Sample Number	Time	Biodiesel Density	Acidity Index	Kinematic Viscosity	Percentage of Oil Yield	Conversion of Triglycerides
		20°C (g cm^{-3})	(mg KOH g^{-1})	40°C (mm^2 s^{-1})	(wt.%)	(wt.%)
1	20	0.91	0.23	4.23	76.28	99.23
2	40	0.88	0.19	4.05	85.26	99.75
3	60	0.86	0.18	3.94	92.35	99.62
4	80	0.81	0.13	3.83	94.16	99.71

Figure 6 shows the yields of biodiesel from ultrasonic transesterifications conducted at 23˚C and 60˚C. Though the biodiesel content varies, it is always within ES 14214 was the allowable range. The best biodiesel generated at 94.16%, or 5.84% glycerol, which is just a smidgen minimum than the number. Table 2 shows that the findings are consistent with those obtained using ultrasound at 60°C, with a conversion rate of 99.71% and a yield of 94.16%.

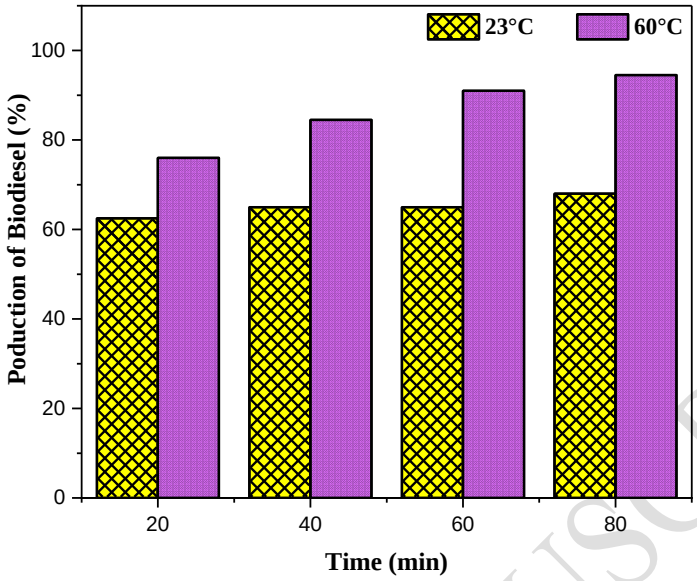


Figure 6. Quantities of biodiesel produced in percentages.

Table 3. Ultrasound settings for transesterification of palm oil at 250 watts.

Oil	Ultrasonic Power	Frequency	Ultrasound Time	Temperature	Molar Ratio Methanol/Oil	Potassium hydroxide Catalyst	Conversion
	(W)	(kHz)	(min)	°C		(weight%)	(%)
Palm oil	250	26	65	60	06-ene	1	99.83

All of the parameters used in this investigation were replicated, including the methanol/oil molar proportion, power, and Potassium hydroxide weight percentage. Based on these findings, authors know that raising the frequency by 4 kHz, the duration of the ultrasound by 20 minutes, and the temperature by 5°C will result in a 99.83 % conversion rate. The use of several raw materials in homogeneous catalysis. Creating biodiesel with pig fat and MeOH as a solvent yields a 99.0% conversion rate, but doing the same with ethyl alcohol and leather training waste yields just a 98.9% conversion rate.

4. Conclusions

An result of this analysis suggests that when ultrasound is utilized as the energy source, the efficiency and physicochemical properties of the biodiesel vary with exposure duration and temperature, even though the electrical power and frequency stay constant. It is possible to obtain the same efficiency in converting triglycerides to biodiesel at room temperature (as opposed to 60°C in 20 minutes) if the exposure period to ultrasound is increased to more than 80 minutes. The goal of industrial biodiesel production is to maximize output in the least amount of time possible, and one strategy for doing so is to raise the reaction temperature by means of renewable energy. The short stabilization period of less than two minutes observed in OCP, LSV, and EIS analyses of biodiesel properties is indicative of the rapid organization of the electrochemical double layer. At least within the investigated possible range, it was also found that the solution and charge transfer impedance measurements revealing extraordinarily high resistance values (on the order of 10^5 and 10^8 Ωcm^2) are evidence against the presence of electrochemical processes.

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