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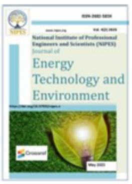

Transesterification of Soybean Acid Oil with Al_2O_3 Catalyst: Effects of Catalyst Concentration and Reaction Time on Biodiesel Yield and Quality

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Abstract

Environmental concerns such as increased global warming have encouraged the use of biofuels in industrial settings. This paper studies the effect of varying catalyst concentration and reaction time in biodiesel production from soybean acid oil, a by-product of the acid degumming process in soybean oil refining via alumina-catalyzed transesterification. The results from transesterification revealed a progressive increase in biodiesel yield with increase in both reaction time and catalyst concentration. Fuel properties such as cetane number, antiknock index, density and acid number of four biodiesel samples obtained with ≥ 90% yield were determined. The best fuel properties of 52.80 cetane number, 95.20 research octane number and 90.20 anti-knock index were observed for the biodiesel sample produced in 75 minutes using 5wt% Al_2O_3*, with an overall free fatty conversion of 99.90%. FT-IR spectra of resultant biodiesel indicated the production of Fatty Acid Methyl Esters (FAME).*

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1. Introduction

Rapid industrialization has not only contributed to the processing of beneficial products for the consumption of man but also a spike in energy demands and environmental pollution, with the processing sector contributing to 21% of global CO_2 emissions [1]. In Nigeria, [2] explored the prospects of the country's energy transition being propelled by its fuel subsidy reforms, tying its possibility to increased awareness on adverse environmental and socio-economic impacts of fossil fuels as well as willingness to explore key renewable energy resources [3,4,5].

Meanwhile, agro-processing companies have been identified to discharge significant varieties of by-products which can be processed into bio-fuels that can serve as fossil energy alternatives or complementary energy sources [6][7]. Such companies include edible oil extraction plants, sugar processing plants and rice mills, among others [8,9,10,11].

1.1 Acid Oil

Acid Oil is a by-product of the chemical degumming process conducted after solvent-based extraction of vegetable oil. It is typically entrained in a viscous sludge generated at about 6% of the daily crude oil output [12]. A number of research works have investigated the production of biodiesel from Soybean Acid Oil; however, most of them are characterized by stringent reaction conditions, low yield and cumbersome reaction processes [6,9,12,13,14]. In transesterification with homogenous base catalysts, an additional bottleneck is soap formation due to the hydrolysis of free fatty acids, often necessitating an esterification step before transesterification and subsequent purification steps [9].

1.2 Biodiesel: Production Processes and Catalysts

Biodiesel is commonly produced by transesterification of vegetable oil or animal fat with short-chain alcohols in the presence of homogenous or heterogenous catalysts, resulting in a fuel with higher oxygen content characterized by zero sulfur emissions, less smoke and fewer particulates [15,16]. The use of biodiesel facilitates improved combustion and can be used in compression-ignition (diesel) engines with little or no modification, thereby contributing to improved engine performance and reduced greenhouse gas emissions [17,18].

Numerous studies have established that aluminium oxide has various favorable catalytic properties such as large surface area, suitable pore size distribution and high thermal stability [20]. In a comparative study by [21], Al_2O_3 outperformed Zeolite and ZnO catalysts in the production of biodiesel from the methanol transesterification of palm oil. Likewise, γ-Al_2O_3 nanoparticles have been used as catalyst support in the production of no-glycerol biodiesel, as reported by [22]. The chemical activity of γ-Al_2O_3 stems from the presence of Al^{3+} ions on the acid site surfaces with the capacity to donate a proton and accept an electron pair [23]. In terms of availability, it is readily available from chemical suppliers and can be synthesized from aluminium can waste at suitable conditions [24].

In this study, the viability of biodiesel production from soybean acid oil using heterogenous Aluminium Oxide Catalyst was investigated under varied conditions, noting the effect of reaction time and catalyst concentration on the biodiesel product yield as well as its fuel properties.

2. Methodology

Acid oil, obtained from Fortune Oil Mills Nig. Ltd. located at No. 59 Tafawa Balewa Road, Kano, was procured and characterised. Proximate analyses were conducted to determine the moisture content, oil content, free fatty acid content, acid value, and density of the acid oil. The oil was extracted using centrifugation following the procedure outlined by [12]. The extracted oil was subsequently transported to the Centre for Renewable Energy and Sustainability Transitions (CREST) at Bayero University Kano for biodiesel production and characterisation.

Chemicals and reagents (Analytical Grade) were procured from Haddis Chemicals. They include: H_2SO_4 acid (98% purity), Methanol HPLC Grade (99.9% purity), Ethanolic KOH Solution (0.1N), Diethyl ether and Al_2O_3 granules. Distilled water and Ethanol Absolute were obtained from Centre for Renewable Energy and Sustainability Transitions (CREST), Bayero University Kano.

The equipment used were accessed at Fortune Oil Mills Quality Control Laboratory and Centre for Renewable Energy and Sustainability Transitions (CREST), Bayero University, Kano. They include: Centrifuge, Analytical weighing balance, Moisture Analyzer, Magnetic stirrer set-up with temperature control, Titration setup – retort stand and burette, Density Bottle, Biodiesel separator, Titrando Tiamo^{TM} software and Petroleum Quality Analyzer.

2.1 Extraction and Characterisation of Acid Oil

In the preliminary laboratory procedure, acid oil was extracted according to the procedure by [12]. Soybean acid degumming sludge and distilled water were mixed in the ratio 5:3, with 13.51 vol% of 20% H_2SO_4 added dropwise with continuous stirring. This facilitated the separation of the mixture into 3 layers after centrifuging. The top layer was contained acid oil, the middle layer contained impurities solubilized in water while the bottom layer contained phospholipid-containing sludge [24].

The determination of free fatty acid concentration was done by weighing 1g of the acid oil sample in a beaker and adding 50ml of diethyl ether to it. Two drops of phenolphthalein were added to the mixture which was titrated against 0.1N KOH while being stirred with a magnetic stirrer. The titre value obtained at obvious colour change was recorded. The FFA% was then calculated using the formula:

$$FFA\% = \frac{t_v \ x \ N \ x \ M}{2m} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where tv = titre value, N = Normality, m = Sample Mass, M = Molar Mass

The Acid Number was determined as 2 (FFA%) mg/mgKOH. Density and moisture content were determined using density bottle and moisture analyzer respectively. The oil sample was dried to remove excess moisture. Thereafter, Fourier Transform Infrared Analysis (FTIR) was conducted on the dried acid oil to note its functional groups. A marked difference was noticed between the spectra in the acid oil and that of standard soybean oil reported by [25], indicating the presence of impurities.

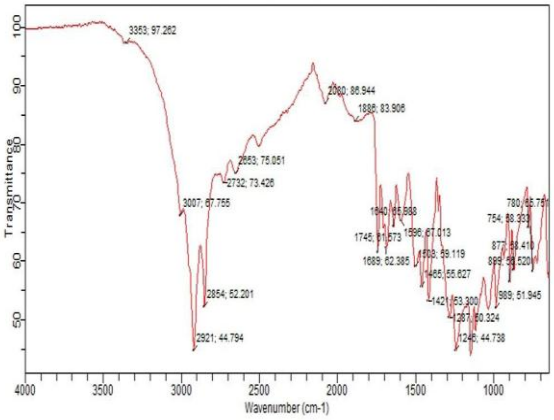

Figure 1: FT-IR Spectrum of Acid Oil

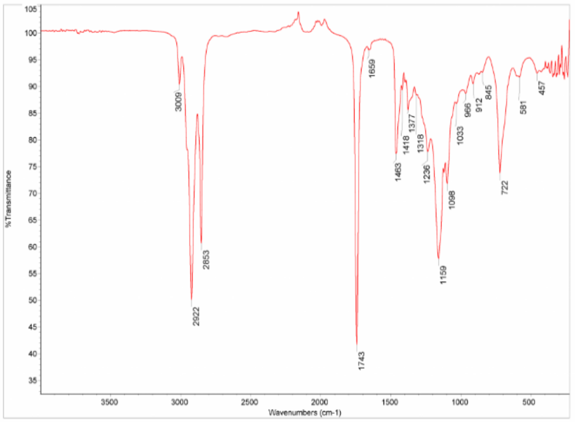

Figure 2: FT-IR Spectrum of Fresh Soybean Oil

2.2 Acid Oil Pretreatment

Pretreatment of acid oil was conducted by esterification using the procedure by [26]. The esterification was conducted by employing the use of 0.85 vol% H_2SO_4 and 1:1 Methanol to Oil ratio stirred at 450 rpm for 70.2 minutes at 60^{o}C. After completion of the reaction, the resulting mixture was left to stand in a separator overnight, resulting in two layers of methanol-water mixture and esterified oil layer. The esterified oil was dried at 105^{o}C to remove residual moisture and then analysed using Titrando Tiamo^{TM} software to determine Acid Value and confirm FFA reduction.

2.3 Biodiesel Production

To convert the triglycerides present in the oil sample into fatty acid methyl esters, appropriate amounts of catalysts, methanol and oil were measured or weighed. Catalyst weight percent was determined by adopting density of Al_2O_3 to be 3.99 g/cm^3. Acid oil was preheated to 60^{0}C, while a pre-determined amount of catalyst was added to methanol and stirred, then the mixture was poured into the oil. The reaction mixture was placed on magnetic stirring apparatus and stirred at 400 rpm for the stipulated reaction duration.

Transesterification was conducted with varying catalyst concentration and reaction time, while keeping temperature at 60^{o}C in the presence of excess methanol. Catalyst concentrations used were 3% and 5% Al_2O_3, while the reaction duration was varied from 60 minutes to 75 minutes, 90 minutes, 105 minutes and 120 minutes

3. Results and Discussion

The experimental results indicated that the Free Fatty Acid (FFA) content and Acid Number of raw acid oil obtained from Fortune Oil Mills Nig. Ltd. were 15.3% and 30.6 mgKOH/mg respectively. The density of the sample was found to be 0.95 g/cm^3. Moisture content was determined to be 0.98% before drying, after which the moisture content became negligible (~0.1%).

3.1 Oil Pretreatment

Pretreatment of the acid oil was done by esterification. A 2-layer product was obtained after overnight settling of the esterification products. The Oil fraction settled at the bottom while Water-Methanol mixture remained at the top. The esterified oil was further dried at 105^{o}C to remove residual methanol and water. The Acid Number of the esterified oil was determined to be 0.85 mgKOH/mg using Titrando Tiamo^{TM} software. This corresponds to a Free Fatty Acid value of 0.425%, which is half the Acid number, and is an indicator of the percentage of free fatty acids in the oil sample.

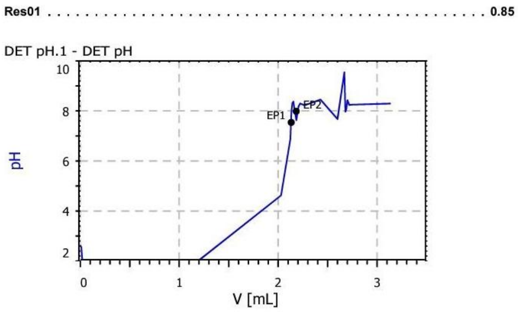

Figure 3: Acid Number determination results of esterified oil obtained using Titrando Tiamo^{TM}software

Based on initial acid number of 30.6 mgKOH/mg and final acid number of 0.85 mgKOH/mg, the free fatty acid conversion percentage amounts to 97.22%. The substantial drop in acid number value makes it suitable for transesterification.

3.2 Transesterification of Acid Oil with Al_2O_3

After transesterification, the Al_2O_3 became relatively darker in colour and compacted in a mixture with a small amount of glycerol, enabling easy separation of biodiesel. This is in line with the potential of Alumina to produce no-glycerol biodiesel [21]. To confirm biodiesel production, FT-IR analysis was conducted on the biodiesel sample produced with Al_2O_3. Yield for each run was calculated as

$$Y = \frac{Volume\ of\ Biodiesel}{Volume\ of\ Oil\ Sample}\ x\ 100\% \ \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

3.2.1 Transesterification Yield Results

Catalyst concentration and reaction time were varied in the production of biodiesel, with results presented in *Table 1* and *Table 2*.

3.2.1.1 Biodiesel Production with 3% Al_2O_3

Table 1 shows the results obtained from the transesterification of soybean acid oil in the presence of 3% Al_2O_3 at 60^0C.

Table 1: Results from transesterification of soybean acid oil with 3% Alumina

Run	**Time (minutes)**	**Yield (%)**
A	60	77.50
B	75	87.50
C	90	92.50
D	105	93.30
E	120	93.75

3.2.1.2 Biodiesel Production with 5% Al_2O_3

Table 2 shows the results obtained from the transesterification of soybean acid oil in the presence of 5% Al_2O_3 at 60^0C.

Table 2: Results from transesterification of soybean acid oil with 5% Alumina

Run	Time (min)	Yield (%)
F	60	80.00
G	75	90.00
H	90	95.00
I	105	96.25
J	120	96.67

3.2.1.3 FT-IR Spectra

Figure 4 shows the FT-IR Spectrum of Biodiesel produced from Soybean Acid Oil using Al_2O_3. Major expected functional groups in the FT-IR spectra are ester group, carboxylic acid group and sometimes methanol group. The peaks observed corresponded to the expected functional groups present in biodiesel, as outlined in standard FTIR tables. Hence, the presence of the 1745cm^{-1} peak indicated a strong presence of esters, corresponding to the carbonyl stretching vibration in the biodiesel's ester group. Likewise, there was a presence of O-H stretch peaks within the 3200-2500 cm^{-1} range, indicating small, broad or medium presence of carboxylic acids. The absence of peaks in the 3550-3200 range indicated the absence of the alcohol group, implying that the methanol solvent had reacted completely and wasn't present in the biodiesel sample.

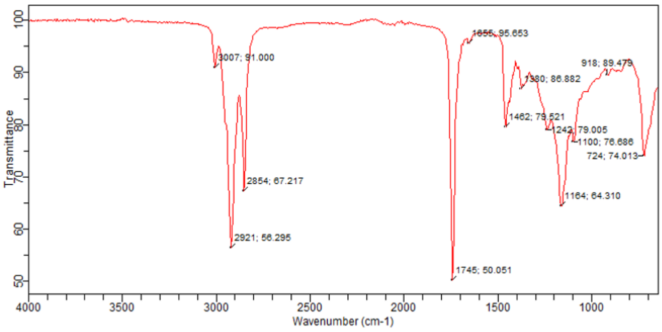

Figure 4: FT-IR Spectrum of Acid Oil Biodiesel Produced after Test Run I using Aluminium Oxide Catalyst

Meanwhile, peaks further confirming the presence of carboxylic acids and esters are often hidden in the fingerprint region, such as the alkyl stretching of $O-CH_3$, represented by an absorbance in the 1300 to 1000 cm^{-1} range which is typical of biodiesel. According to [27] this was represented by a peak at 1196 cm^{-1} while for this work, it was observed at 1162 cm^{-1}.

3.3 Biodiesel Fuel Characterisation

The runs of interest were narrowed down to four (C, D, G and H) runs which produced greater than biodiesel yield at the lowest reaction time to determine the optimal reaction conditions within the prevailing constraints. Fuel properties of Produced Biodiesel (Acid Number, Saponification Value, Cetane Number, Octane Number, etc.) in these runs were determined. Results from characterization of fuel properties are presented in *Table 3*.

Table 3: Biodiesel fuel properties

Run	Acid Number (mgKOH/g)	Saponification Value (mgKOH/g)	Cetane Number	Research Octane Number	Antiknock Index	Density (kg/m^3)
C	0.11	198.00	40.1	80.8	78.9	875
D	0.13	193.77	45.8	91.9	87.4	885
G	0.03	217.30	52.8	95.2	90.2	865
H	0.27	199.91	45.3	91.7	87.3	875
Standard	ASTM D664	ASTM D5558-95	ASTM D613	ASTM D2699	-	EN ISO 3675
Limits	**0.50 max.**	**370 min**	**40 min**	**87min**	**-**	**860 -900**

3.4 FT-IR Spectra

The Fourier Transform Infrared Spectroscopy aids in confirming the presence of Fatty Acid Alkyl Esters in biodiesel samples [28]. Hence, according to standard FT-IR Tables, to confirm the formation of fatty acid methyl esters (biodiesel), C=0 Stretching (Ester Carbonyl) groups (about 1740 cm^{-1}) are be expected from the FT-IR spectra, along with C-H Stretching (Alkyl Chain) peaks at 2800-3000 cm^{-1}. In some cases, O-H Bending (Alcohol) broad peak around 3200-3500 cm^{-1} is observed due to incomplete consumption of methanol. The FT-IR spectra for the different samples confirmed the formation of fatty acid methyl esters.

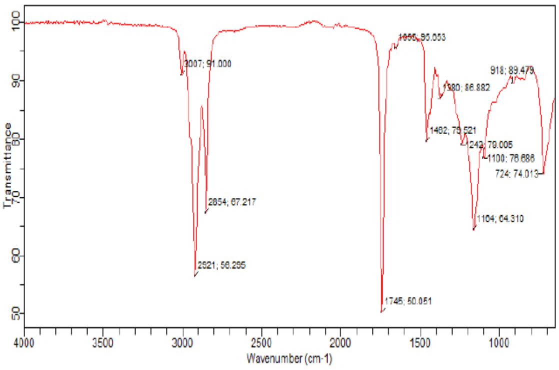

Figure 5: FT-IR Spectrum for Sample C

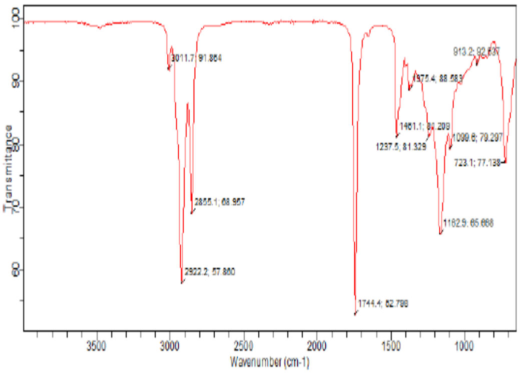

Figure 6: FT-IR Spectrum for Sample D

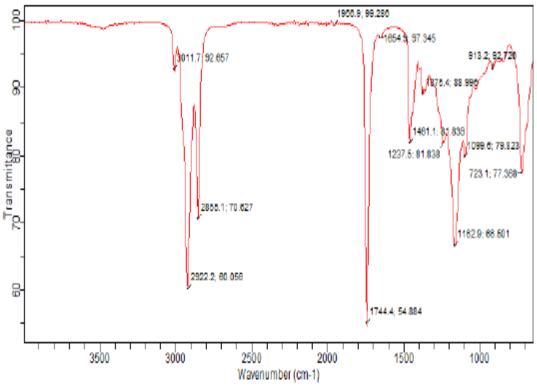

Figure 7: FT-IR Spectrum for Sample G

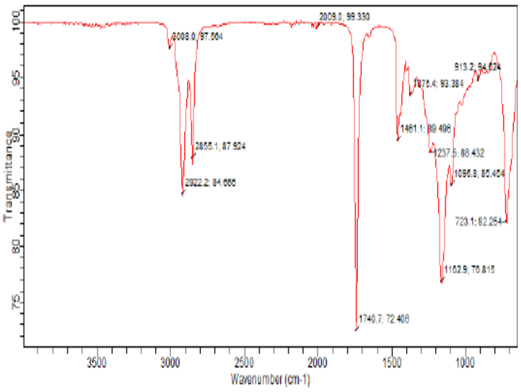

Figure 8: FT-IR Spectrum for Sample H

3.5 Biodiesel Yield

Factors affecting biodiesel yield include reaction temperature, catalyst concentration, alcohol-oil ratio and type of alcohol used [29]. A marked increase in yield was noticed with increase in both catalyst concentration and reaction time. This is in line with a range of previous research studies [30,31,32].

3.5.1 Effect of Reaction Time

Adequate reaction time is required to facilitate the complete conversion of triglycerides to fatty acid alkyl esters; yield generally increases with reaction time [33]. However, prolonged reaction time often results in a reduction of actual biodiesel yield by triggering the reverse reaction or facilitating the formation of soap, hence causing depletion of esters [34]. From the results obtained after transesterification of soybean acid oil catalyzed by both 3% Al_2O_3 and 5% Al_2O_3, the yield increases progressively with increase in reaction time, but the rate of yield increase dropped substantially after 90% yield in both cases.

3.5.2 Effect of Catalyst Concentration

Biodiesel yield generally increases with increase in catalyst amount by favouring the completion of the reaction in the presence of more active sites, thereby resulting in higher biodiesel yield in shorter reaction time [31]. Therefore, efforts are made by researchers to optimize catalyst percentage along with reaction time to minimize costs due to cost of catalysts and energy resources [16].

Table 4 compares the yield obtained using 3wt% Alumina and 5wt% Alumina during the same reaction time, affirming that biodiesel yield increases with catalyst concentration at reasonable limits. However, excessive catalyst concentration, in addition to wastage of resources, affects biodiesel yield by resulting in unfavourable mass transfer limitation due to increase in viscosity which decreases surface contact between the reactants and the catalyst’s active sites [31].

Table 4: Variation of biodiesel yield with catalyst amount at 60⁰C

S/N	Time (minutes)	Yield with 3wt% catalyst	Yield with 5wt% catalyst
1	60	77.5	80
2	75	87.5	90
3	90	92.5	95
4	105	93.3	96.25
5	120	93.75	96.67

From the data obtained and shown in *Table 4*, it can be noted that a significant increase in biodiesel yield accompanies increase in catalyst amount (weight %) at the same reaction conditions. The highest biodiesel yield is obtained using 5wt% Al_2O_3 after 120 minutes. A comparative graph of the yields using the two different catalyst concentrations are shown in *Figure 9*.

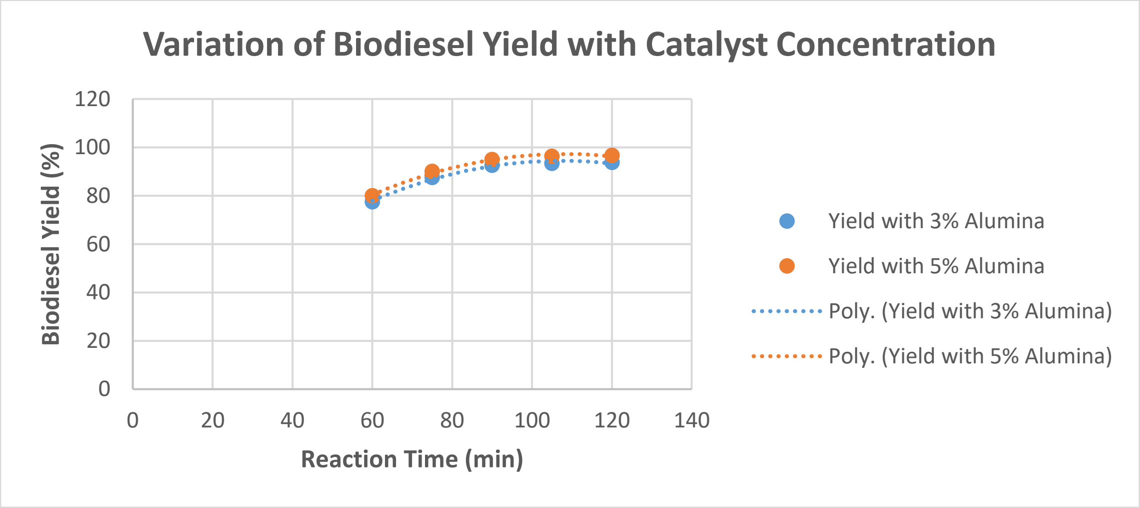

Figure 9: Effect of Catalyst Concentration on Biodiesel Yield

3.6 Biodiesel Quality

To assess the properties of the produced biodiesel, the fuel properties of the biodiesel samples produced with ≥90% yield at the shortest reaction times for both catalyst concentrations (two for each) were determined. The saponification value, cetane number, acid number, octane number, anti-knock index and density for samples C, D, G and H along with standard limits for the parameters are presented in *Table 3*. It may be recalled that sample C refers to the biodiesel produced using 3% Al_2O_3 in 90 minutes, sample D refers to biodiesel produced with 3% Al_2O_3 in 105 minutes, sample G refers to biodiesel produced with 5% Al_2O_3 in 75 minutes and sample H refers to biodiesel produced with 5% Al_2O_3 in 90 minutes.

3.6.1 Acid Number Analysis

From the results in Table 3, all samples conformed with the biodiesel limits of acid number, implying that the produced biodiesel is of high quality. However, the highest acid number was observed in sample H, which was produced with 5wt% catalyst in 90 minutes, while the lowest value was observed in sample G produced with 5wt% catalyst in 75 minutes. The final acid number value obtained for sample G as well as the free fatty acid conversion are shown in *Figure 10* and *Equation (ii)* respectively.

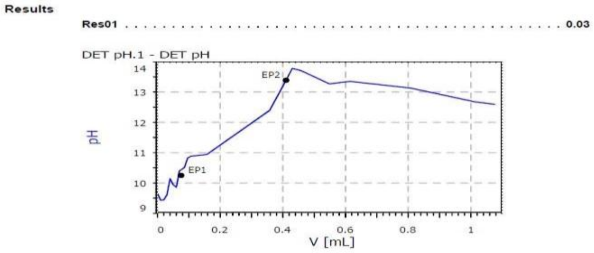

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Figure 10: Acid number result for Sample G (produced with 5wt% Alumina in 75 minutes)

$$F\% = \frac{30.6 - 0.03}{30.6} \; x \; 100\% = 99.9\% \; \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

3.6.2 Density Analysis

The results from density analysis showed the least density attained at the lowest reaction time of 75 minutes as observed in sample G. The density increased progressively with the same value in samples C and H which were both produced in 90 minutes, though with different catalyst concentrations, while the highest value was observed in sample D which had the longest reaction duration.

3.6.3 Saponification Analysis

In terms of saponification value, a sharp contrast to the results from acid number determination was observed, with the highest saponification value noted in sample G while the lowest observed in sample D.

3.6.4 Petroleum Property Analysis

The variations in the other fuel properties of Cetane Number, Research Octane Number and Anti-Knock Index obtained using a Petroleum Analyzer followed the same pattern with the highest values observed in sample G, followed by samples D, H and C in that order.

3.6.5 Best Quality Sample

Building on results from conducted analyses, it can be surmised that Sample G produced with 5% Al_2O_3 in 75 minutes featured the highest Research Octane Number as well as Cetane and Anti-Knock Indices, while having the lowest density and acid number. Though it has the highest saponification value which is still below the stipulated limit of 370 mgKOH/g, it surpasses the other biodiesel samples in terms of fuel properties.

3.6.6 Comparison with Previous Research

A comparison of properties of biodiesel produced from sample G with results presented in previous research publications yields the data in *Table 5.*

Table 5: Comparison of biodiesel production results

Feedstock	Methodology	Results	Source
Soybean Oil	**Cation exchange resins** 6wt% catalyst, 363K at 600rpm for 120 min	66.3% free fatty acid conversion	[6]
Soybean Sludge and Palm Kernel Free Fatty Acid	**2-step Homogeneous Trans-esterification** with HCl and KOH Reaction at 65 °C for 2hrs	85.96 max. yield	[7]
Soybean soapstock	**Acid Esterification** T: 80°C for first hour 95°C for 4 hours Pressurized 4kgf/cm^2 reactor	94% FFA conversion	[12]
Acidified Soybean Soapstock	**Transesterification** T: 70 °C for 5 hrs 7wt% lignin-derived Catalyst	97% Free Fatty Acid conversion	[13]
High-Acid Oil from Soybean Soapstock	**Supercritical Methanol Trans-esterification** T: 350 °C for 30 mins	45.49 Cetane Index 0.8mgKOH/mg acid Number	[14]
Jatropha Oil	**Heterogeneous Transesterification** Reaction with 1wt% catalyst for 90 minutes	94.38 max. yield	[15]
Sludge Palm Oil	**Enzymatic Transesterification** Ethanol-Oil ratio: 4:1 10U Enzyme loading 40°C at 250rpm speed for 24hr	62.3% max yield	[35]
Palm Fatty Acid Distillate	**Esterification** Reaction with 10wt% acidic catalyst at 60^{0}C for 4hrs	82.5% max conversion	[36]
Soybean Acid Oil (extracted from soybean soapstock)	**Heterogenous Transesterification of Esterified Oil** Transesterification conducted with 5wt% Al_2O_3 for 75 minutes at 60°C	96.67% max. yield (J). 52.8 cetane number, 0.03mgKOH/mg acid number and 99.9% FFA conversion (G).	This study

4. Conclusions

From the research outcomes, it can be inferred that hybrid two-step transesterification consisting of a homogeneous esterification and heterogeneous transesterification of high free-fatty-acid-oil results in reduced reaction time and substantial yields when compared to the successive homogeneous two-step transesterification. Biodiesel yield increased progressively with both reaction time and catalyst concentration. However, the rate of yield increase declined considerably with extended reaction time, in agreement with the findings by [26]. Some of the observed fuel properties of biodiesel product didn't vary linearly with catalyst concentration or reaction duration. Density was found to increase with reaction time while Cetane number, octane number and anti-knock index increased in the order C < H < D < G. The reaction conditions for sample G, which exhibited the best fuel properties of 52.8 cetane number, 95.2 research octane number and 90.2 anti-knock index were 60^{0}C reaction temperature, 5wt% Al_2O_3, stoichiometric excess of methanol reacted with 30ml of esterified oil sample for a 75-minute reaction time. FT-IR spectra results showed strong presence of fatty acid methyl esters, thereby confirming biodiesel production.

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