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Abstract

Due to the awareness of adverse effects of conventional fuels on environment and the frequent rise in crude oil's price, the need for sustainable and environment friendly alternate source of energy has gained importance in recent years. Biodiesel has proved to be a good replacement for petro-diesel because of its unique properties like significant reduction in greenhouse gas emissions, non-sulfur emissions, non-particulate matter pollutants, low toxicity and biodegradability. In this study, calcined cow bone was used as heterogeneous catalyst in the transesterification of palm kernel oil (PKO). The catalyst was calcined in a muffle furnace at 900°C for 2 hours and 30 minutes. The catalyst was characterized via Fourier Transform Infrared Radiation (FTIR) Spectroscopy. Transesterification was carried out by varying the parameters such as methanol/oil ratio (6:1, 9:1, 12:1 and 15:1), reaction temperature (40, 50, 60 and 70 °C), catalyst particle size (1mm, 2mm and 4mm mesh sizes) and reaction time (2, 3, 4, and 5h) using an agitation stirring speed of 300rpm. The optimal conditions for the transesterification obtained are reaction time of 60mins, catalyst particle size of 1mm, methanol to oil ratio of 12:1 at a catalyst loading of 10% weight of oil. The obtained biodiesel was characterized as an alternative diesel fuel through series of ASTM standard fuel tests. Higher specific gravity, pour point and cloud point were obtained and comparison was made with to petroleum diesel. Specific gravity values were found favorable compared with results for other vegetable oil biodiesel. Results obtained were found to be within limits set by America Standard for testing machine (ASTM) standards for biodiesel.

Keywords: Biodiesel, calcined, transesterification, heterogeneous, reaction, biofuel

1. INTRODUCTION

Population increase is in direct proportion to energy consumption to the energy consumptions. In any nation, energy is the most fundamental requirement for human existence and it is the chief mover of economic growth and plays a vital role in sustaining the modern economy and society (Krishna et al 2013, Ribeiro et al 2011). The world has witnessed many changes in the energy field where many companies around the world created new strategies based on reducing the environmental impact of those energy. According to the International Energy Outlook 2018, the US Energy Information Administration has estimated a significant growth in global energy demands in the next 24 years. The energy consumption was projected to escalate from 549 quadrillion Btu to 815 quadrillion Btu, a 48 % growth between 2012 and 2040. The main contributing factors to high energy consumptions are population size and urbanization rate (Nejat et al. 2015).

The majority of the world's energy need is supplied through petrochemical sources, coal and natural gases, with the exception of hydroelectricity and nuclear energy, are finite and at current usage rates will be consumed shortly (Mahanta and Shrivastava 2004). The impact of fossil fuel-based energy technologies on the environment and global climate has recently been recognized worldwide and is the major driving force in the search for renewable energy sources alternative. Therefore, in order to meet energy needs and keep the global environment as clean as possible, it is crucial to develop green energy technologies that are renewable, sustainable and environmentally benign. Biofuels and biomass-based energy are potential major contributors of energy in the next century.

Approximately, 90% of the biofuel market is dominated by bioethanol and biodiesel. Biofuels are commonly categorized into first, second and third generations. First generation fuels are made from crops such as sugar, starch and animal or oil fat. Most common first-generation biofuels are bioethanol and biodiesel. Among these, biodiesel is produced from oils such as rendered animal fats, rapeseed and palm oil through transesterification reaction. While bioethanol is produced from the fermentation of any feedstock that contains a high content of sugar or starch. Biodiesel, which represents over 80% of biofuels production and accordingly, it is the main alternative to diesel fuel (Mata et al. 2010, Bozbas 2008).

Second generation biofuels are produced from non-food crops like waste, stalks of wheat and corn. Most common second-generation biofuels are biohydrogen, bioethanol, mixed alcohols and wood diesel. Third generation biofuels include Algae fuel; produce 30 times more energy than land crops. These algal biofuel technologies are not yet commercially viable. At present scenario, first and second-generation fuels are the only choices, which are used in gasoline and diesel engines as per classification given with increasing trend of modernization and industrialization, the oil security and its continued availability has become an area of extreme concern for the world. Under such conditions, biofuel energy has an important function in the growth of economy of a country (Rakesh et al 2010).

Non-renewable fossil raw materials (oil, coal and natural gas) are still the most used sources of energy and chemicals, where approximately three quarters are used for generation of power and heat and nearly one quarter is used as fuel for transportation. Only few percent of fossil raw materials are used to produce chemicals and polymer materials (Bhaskar et al 2011). Many important factors like vulnerable dependence of global economy on fossil raw materials, political issues connected with important oil-producing countries, emission of greenhouse gases and climate changes have moved the focus of many governments to explore alternatives of using renewable energy sources (Cherubini and Strømman 2011, Forster and Ramaswamy 2007). During the last few decades, the production and usage of biofuels (biodiesel, biogas and bioethanol fuel) has raised considerable interest as an appropriate means for partial substitution of fossil fuels.

Biofuels derived from plant-based biomass are considered as renewable and represent an environmentally acceptable energy source, thus offering the potential to reduce and replace consumption of fossil fuels to a great extent (Khanal 2008). Biodiesel is a clean-burning alternative fuel that is derived from renewable sources that can be made from vegetable oil, animal fat, and recycled cooking oils. Oils produced from algae, fungi, bacteria, molds, and yeast can also be used to produce biodiesel. According to American Society for Testing and Materials, Biodiesel is a mono-alkyl esters of long chain fatty acids derived from renewable lipids, such as vegetable oils or animal fats, used in compression ignition engines. (ASTM, 2002).

Biodiesel is a type of fuel that is usually produced by transesterification of vegetable oils or animal fats with chemical catalysts (either homogeneous or heterogeneous), especially in the presence of strong acidic or basic solutions, such as hydrochloric acid, sulphuric acid, sodium hydroxide, sodium methoxide and potassium hydroxide. (Diamantopoulos N., Panagiotaras D. and Nikolopoulos D, 2015). Biodiesel is highly biodegradable and has minimal toxicity. It has almost zero emissions of aromatic compounds and other chemical substances that are destructive to the environment. It has a small net contribution of carbon dioxide (CO₂) when the whole life-cycle is considered (including cultivation, production of oil and conversion of oil to biodiesel); and its production can be decentralized so that it could have significant potential for improvement of rural economy (Ahmad et al. 2011; Carraretto et al. 2004; Shemelis and Jorge 2017).

Compared to diesel fuel, biodiesel produces no sulfur, less carbon monoxide, less particulate matters, less smoke and hydrocarbons emission and more oxygen. More free oxygen leads to the complete combustion and reduced emission (Fazal et al 2011; Silitonga et al. 2011; Shemelis and Jorge 2017). Due to the presence of oxygen in its chemical composition, its combustion is more complete, reducing the particulate emissions, carbon monoxide and unburnt hydrocarbons, among other contaminants. Biodiesel is a petroleum diesel substitute and one of the most promising biofuels due to its environmental compatibility and biodegradability (Bateni and Karimi 2016 and Tremblay and Montpetit 2017). It has combustion properties very similar to the Petro-diesel including the energy content and cetane ratings (Ability to start cold) it is comparable to diesel Biodiesel can be used in diesel engines with little or no modification to the engine mechanism, (Makgaba and Daramola 2017). During its production process a by-product glycerin is obtained.

The glycerin after purification can be used in pharmaceutical and cosmetic industry. Transesterification of a vegetable oil was conducted as early as 1853 by scientists E. Duffy and J. Patrick, many years before the first diesel engine became functional (. Rudolf diesels prime model, a single 10 ft. (3 m) iron cylinder with a flywheel at its base, ran on its own power for the first time in Augsburg, Germany on August 10, 1893, declared as International Biodiesel Day. This engine stood as an example of Diesels vision, because it was powered by peanut oil-a biofuel though not biodiesel, since it was not transesterified. He believed that the utilization of biomass fuel was the real future of this engine. During the 1920s, diesel engine manufacturers altered their engines to utilize the lower viscosity of Petro diesel (a fossil fuel), rather than vegetable oil (a biomass fuel). Despite the widespread use of fossil petroleum-derived diesel fuels, interest in vegetable oils as fuels in internal combustion engines is reported in several countries during the 1920s and 1930s and later during World War II.

On August 31, 1937, University of Brussels (Belgium) was granted patent (16) for a Procedure for the transformation of vegetable oils for their uses as fuels and this appears to be the first account of the production of what is known as biodiesel today. More recently, in 1977, Brazilian scientist Expedito Parente invented and submitted for patent, the first industrial process for the production of biodiesel. This process is classified as biodiesel by international norms, conferring a "standardized identity and quality. In 1979, more than a century later after the discovery of the first transesterification of vegetable oil, South Africa initiated the use of transesterified sunflower oil and refined it to diesel fuel standards. An Australian company, Gaskots, obtained the technology from the South African Agricultural Engineers, the company erected the first biodiesel plant in November, 1987 and the first industrial scale plant in April 1989 (with a capacity of 30,000 tons of rapeseed / annum).

There have been several works on biodiesel production using different catalyst mechanisms, however, this research work is centered on the use of heterogeneous catalyst based on calcined cow bone to produce biodiesel. This work seeks to establish an alternative suitable route to produce biodiesel by utilizing waste cow bone thereby solving its disposal problem and contributing to sustainable environment. The factors influencing the production of biodiesel studied in this work are reaction time, temperature, methanol/oil ratio and particle size.

2. MATERIALS AND METHODS

2.1 Materials

Palm Kernel oil: The Palm Kernel oil (PKO) was purchased at Ewo market at Okpella in Edo State, Nigeria.

Cow bones: The bones of white Fulani breed cow were used for the production of the heterogeneous catalyst, it was bought from the abattoir at Ebute area of Ikorodu, in Lagos state, Nigeria.

2.1. Palm Kernel oil processing. Prior to use in the biodiesel process, the purchased palm kernel oil was filtered to remove any entrained solid that may be suspended in the oil.

2.2 Reagents, Glasswares and Equipment

2.2.1 Reagents

- a) Methanol (99.5 % pure with boiling point of 78 °C) manufacture by Sigma Aldrich Inc., Darmstadt, Germany.
- b) Sulphuric acid (98 % analytical grade) manufacture by Thomas Scientific Inc., New Jersey, U.S.A
- c) Sodium hydroxide pellets (98 % analytical grade) manufacture by Ricca Chemical Company, Arlington, U.S.A
- d) Isopropyl Alcohol (99.9 % analytical grade) manufacture by Alliance Chemical, Taxes, U.S
- e) Potassium hydroxide pellets (85%) manufacture by Ricca Chemical Company, Arlington, U.S
- f) Phenolphthalein
- g) Sodium thiosulphate (99.99% with boiling point of 100°C) manufacture by Muby Chemicals, Mumbai, India.
- h) Wij's Reagent (Density of 3.10 g/cm³, Boiling point of 97.4°C and Melting point 27.2 °C) manufacture by Sigma Aldrich Inc., Darmstadt, Germany.
- i) Chloroform (Density:1.49 g/cm³, Boiling Point:61.2 °C, Melting Point: - 63.5 °C) manufacture by Sigma Aldrich Inc., Darmstadt, Germany.
- j) Starch
- k) Potassium Iodide (99.0 % analytical grade) manufacture by GFS Chemicals Inc., Columbus, OH.
- l) Ethanol (95 % analytical grading) manufacture by Sigma Aldrich, Germany
- m) Diethyl ether manufacture by Sigma Aldrich Inc., Darmstadt, Germany.
- n) Hydrochloric acid manufacture by Thomas Scientific Inc., Company, New Jersey, U.S.

All reagent use in the project were of analytical grade and were purchased from FINLAB Laboratories, Ltd., Lagos, Nigeria.

2.2.2 Equipment

- a) Hot plate with magnetic stirrer (C-MAG HS7) with a maximum agitation speed of 300 rpm and maximum temperature (300 °C) manufacture by IKA Laboratory Analytical and Processing Technology, Staufen, Germany.
- b) Thermometer (mercury in glass, range of 0-500)
- c) Hammer mill (Electric motors driving ranging from 2000 to over 500 horsepower (1.5-3.7 MW) manufacture by Broyeur Clero, Germany.
- d) Ball mill manufacture by Shanghai Clirik Machinery Company Ltd, Shanghai, China
- e) Muffle furnace: (volts 230, temperature of 3,000 °C) manufacture by Carbolite-Gero, England.
- f) Viscometer, NDJ-5S digital rotary viscometer
- g) Fourier transform infrared spectrophotometer (FTIR-8400 S) manufacture SHIMADZU, Kyoto, Japan.

2.3 Experimental Procedure

2.3.1 Calcination of the processed cow bone

In the preparation of the catalyst from cow bone the method used by (Akhavue and Ebuzome 2018) was adopted with slight modification. The cow bones obtained were washed thoroughly to remove remnant blood, tissues and other unwanted materials before being dried in an oven at a temperature of 100 °C to remove water. The cow bones were then crushed first using a hammer mill and then a ball mill to further reduce the sizes to about 1mm, 2mm and 4mm..The milled cow bone was calcined in a muffle furnace at a temperature of 900°C for 2 hours and 30 minutes. The calcined bones were then allowed to cool in the furnace before being transferred into the desiccator to avoid contact with air and moisture.

2.3.2 Esterification of the oil

Esterification of the PKO was carried out prior to transesterification reaction because the FFA content of the oil was more than 2%, FFA content of oil greater than 2% reacts with the basic catalyst to produce soap and the yield of the biodiesel becomes low. In the esterification reaction, the procedure used by Akhavue and Ebuzome was adopted with slight modification. The PKO was esterified twice to give FFA value of 1.4025% which was suitable and use for the production biodiesel.

2.3.3 Biodiesel production

Palm kernel oil was transformed to its methyl ester via transesterification. The reaction was carried out in a batch reactor (a 250ml two way round bottom flask). For every run, 25 ml of the palm kernel oil was measured with a measuring cylinder into the reaction flask. The required amount in volume of the methanol solvent was measured with a measuring cylinder and poured into a beaker, the required amount in mass of the catalyst was as well measured from a weighing balance and added to the methanol. The beaker was covered with foil paper and the content vigorously mixed. The prepared methanol/catalyst mixture was thus added to the oil in the glass reactor. All reactants were charged into the reaction flask at room temperature.

The reactor was fitted with a reflux condenser to return most part of the vaporized methanol as reactions took place at temperatures higher than its boiling point. Stirring was initiated at a speed of the magnetic stirrer at 300 rpm. Stirring speed was kept constant for all experiments. The aforementioned variables including methanol-oil molar ratio, reaction time, reaction temperature and catalyst loading were thus varied accordingly based on the experimental design. After the reaction was stopped, the resulting mixture was filtered to remove the catalyst and then poured into a separating funnel to allow to settle.

The catalyst was removed to avoid clogging at the exit of the separating funnel. After settling, the product mixture was separated into two liquid layers: crude methyl ester (biodiesel) layer above and glycerol layer at the bottom since the biodiesel layer is lighter than the glycerol layer. Unreacted methanol was seen above in cases of incomplete reaction or when in true excess. On separation from the glycerol by-product, the biodiesel was washed by adding an equal amount (to the volume of the biodiesel) of warm distilled water to it in the separating funnel, the separating funnel was swirled severally and clamped on a retort stand for some minutes till water layer was separated from diesel layer. The water (now impure) was drained off from the bottom of the funnel. After washing, the biodiesel was dried by heating on a hot plate.

Table 2.1- Levels of the Transesterification Process Variables Chosen for This Study.

Variables	Factors Coding	Unit	Levels		
Reaction Temperature	X1	°C	40 70	50	60
Reaction time	X2	Hour	2	3	4
Oil to methanol ratio	X3		5		
Catalyst particles size	X4	mm	1:6 1:15	1:9	1:12
			1	2	4

2.4 Analytical Tests for PKO and Biodiesel

Standard analytical tests were carried out on both the PKO and the samples of biodiesel produced.

3. RESULTS AND DISCUSSION

3.1 Physio-Chemical Properties of the Palm Kernel Oil

The results of the tests and analysis carried out on the Palm kernel oil are presented in Table 3.1 below.

Table 3.1: Properties of Palm kernel oil

PARAMETER	Palm kernel oil
Acid Value (mg KOH/g oil)	19.26
FFA (mgKOH/g oil)	9.63
Iodine Value (g I ₂ /100 g oil)	64.9
Saponification value (mgKOH/g oil)	245
Peroxide value	2.7
Density (g/cm ³)	0.94
Viscosity (mm ² /s) at 25°C	35.1
Colour	Brown

For suitability of oil for transesterification process (biodiesel production), it is important to bring the FFA of the oil down to below 2% and most preferably 1% in order to ensure that the catalyst does not react with FFA to produce soap and water which will lead to low yield of the biodiesel.

The FFA content of the crude Palm kernel oil was found to be high (9.63%) therefore, the oil was esterified twice to lower the FFA to a value of 1.4025% before proceeding to transesterification.

3.2 Catalyst characterization

3.1 Fourier Transform Infrared (FTIR) Analysis

In this study, the calcined cow bone at 900°C was analyzed using FT-IR. The FT-IR analysis is as shown in figure 4.0. The functional groups in the catalyst were determined using a Fourier transform infrared (FTIR) spectrophotometer (FTIR-8400 S, SHIMADZU). The region of wavelength was within the range of 650 and 4000 cm⁻¹. The spectrum of FTIR analysis of the calcined cow bone is shown in Fig. 2.

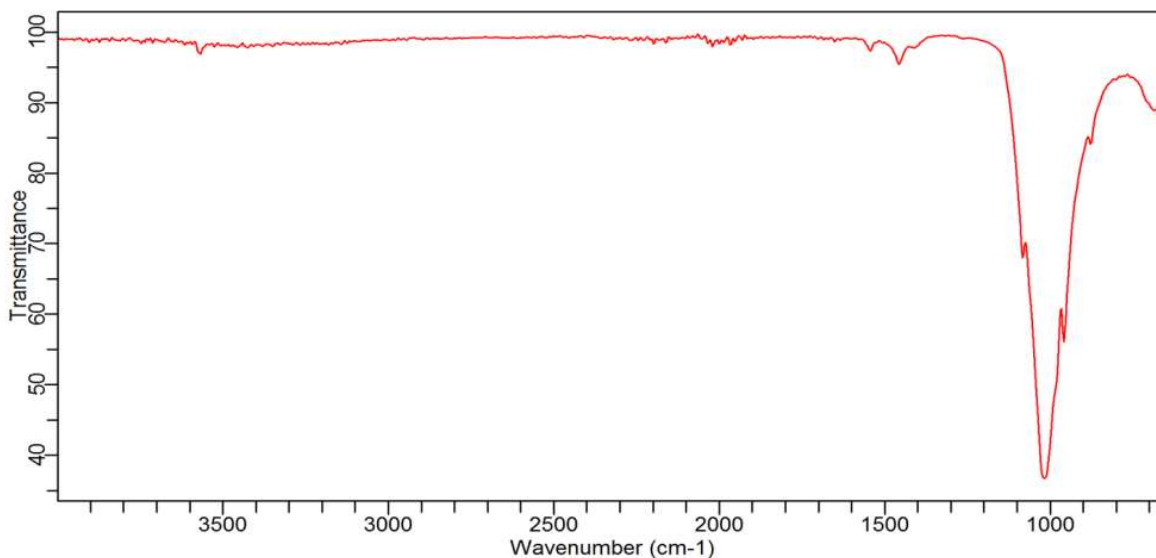


Fig. 3.1: FT-IR spectrum of FTIR analysis of the calcined cow bone at 900°C after the reaction. Based on the area of each peak, major absorption peaks were observed at 3563.97, 2262.54, 2035.09, 1543.10, 1017.60 and 897.55 cm⁻¹. These major absorption bands and their corresponding functional groups are listed in Table 3.2 below.

Table 3.2 Major absorption peaks and their functional groups before Transesterification reaction

Wavelength	Functional groups	Vibration types
3563.97	-OH	Asymmetric stretching
2262.54	-C=O	Stretching Vibration
2035.09	-CH ₂	Symmetric Bending
1543.10	C-O	Stretching Vibration
1457.41	=C-O-C	Symmetric Stretching
897.55	C-H	Plane rocking Vibration

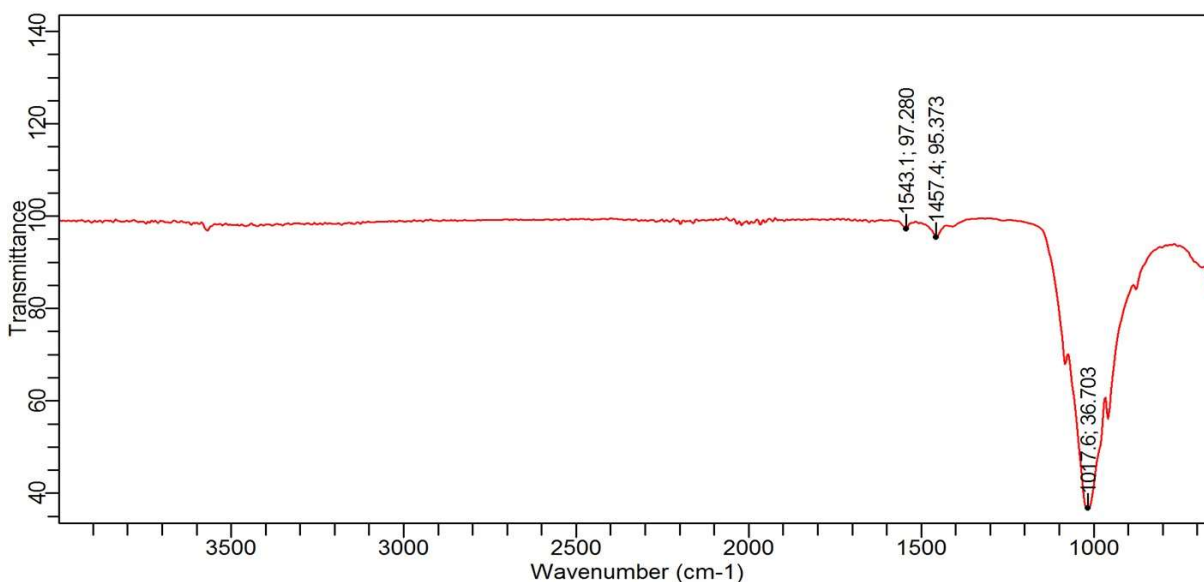


Fig 3.2 FT-IR spectrum of FTIR analysis of the calcined cow bone at 900°C after the reaction

Table 3.3 Major absorption peaks and their functional groups After Transesterification reaction

Wavelength	Functional groups	Vibration types
1543.10	C-O	Stretching Vibration
1457.41	=C-O-C	Symmetric Stretching
1017.64	C-H	Plane rocking Vibration

3.3 Transesterification Process

In this work, the effects of reaction temperature, time, methanol to oil ratio and the heterogenous catalyst particle size on biodiesel yield from palm kernel oil were investigated.

3.3.1 Effect of Oil to Methanol Ratio

The alcohol to oil ratio is one important factor that affects biodiesel production reaction. The stoichiometric ratio for transesterification requires 3 moles of methanol for each mole of oil to yield 3 moles of fatty acid methyl ester and one mole of glycerol. Since the transesterification is a reversible reaction, excess methanol is required to drive the reaction towards the product. Different oil-to-methanol molar ratios varying from 1:6, 1:9, 1:12, and 1:15 were used to investigate the variations of the percentage yield with the molar ratios whilst keeping other factors constant (reaction time, and catalyst dosage etc.). As indicated in figure 4.0 there is a significant increase in the %yield from 1:9 to 1:12 but the increase can be seen to be marginal from 1:12 to 1:15. Further increase in oil-to-methanol ratio after optimal ratio of 1:12 led to a reduction of the biodiesel yield. This is due to excessive methanol beyond the optimal point which does not promote the reaction. The glycerol which is a by-product of the reaction would largely dissolve in the excessive methanol and subsequently inhibit the reaction of methanol to reactants and catalyst, thus interfering with the separation of glycerin, which in turn lowers the conversion by shifting the equilibrium in the reverse direction (Ismail *et.al.*, 2016)

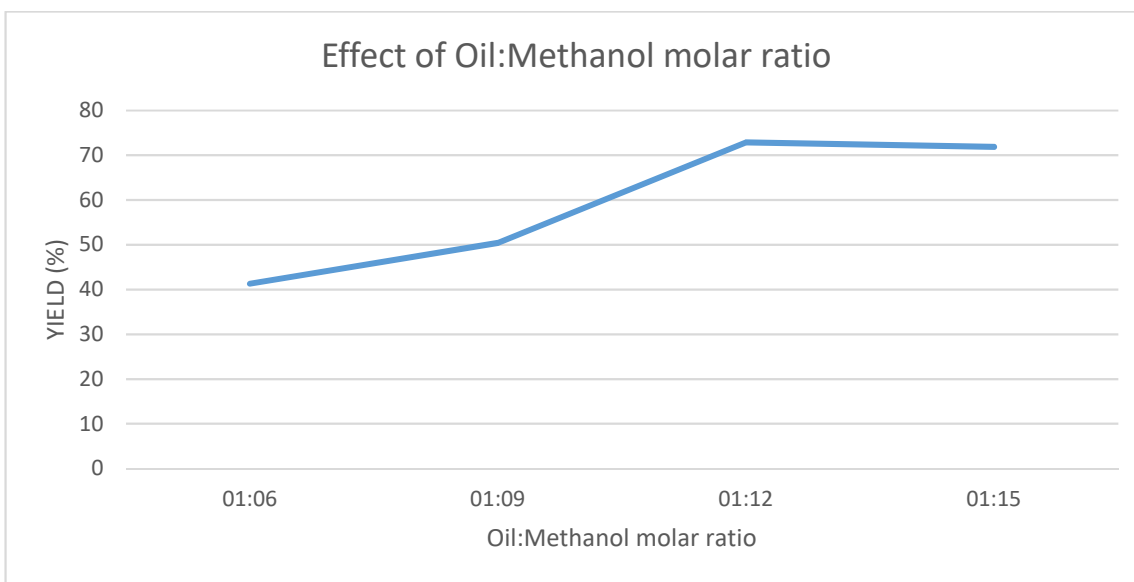


Figure 3.3: Plot of Oil to Methanol Ratio Against Biodiesel Yield

Effect of reaction time on the yield of biodiesel

The reaction time for the production of biodiesel was varied from 2, 3, 4 and 5 hours. The results obtained are as shown in Figure 3.0. It can be deduced that after reaction time of 3 hours, the yield experienced a slight decrease and can be attributed to some esters reverting to form soap with the catalyst. This is in agreement with the work of Norzita Ngadi et al, 2016)..

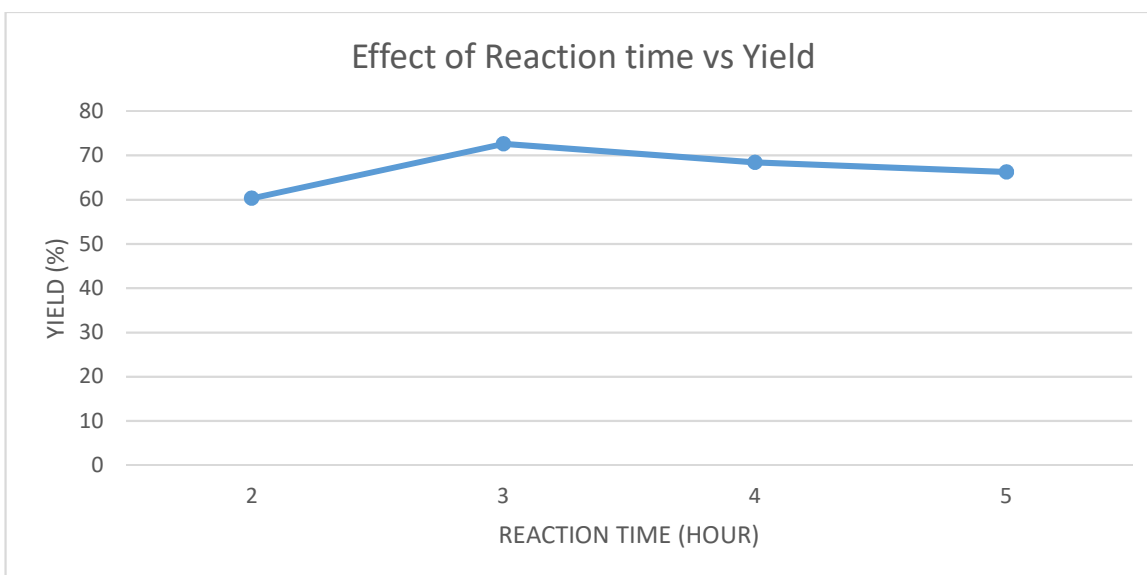


Figure 3.4: Reaction Time vs Yield

3.3.2 Effect of catalyst particle size on Percentage Yield

Different catalyst particle sizes from 1mm, 2mm and 4mm mesh were investigated as per the yield of biodiesel, keeping other variables constant as well. The effect is shown in figure 3.4. At particle sizes of 1mm, 2mm and 4mm, the lower yields can be attributed to the lower surface area of the catalyst to catalyze the transesterification. The lower the particle size, the higher the surface area of the catalyst and the larger the active sites for catalyst activity This is in tandem with the findings of Ismail et.al (2016)

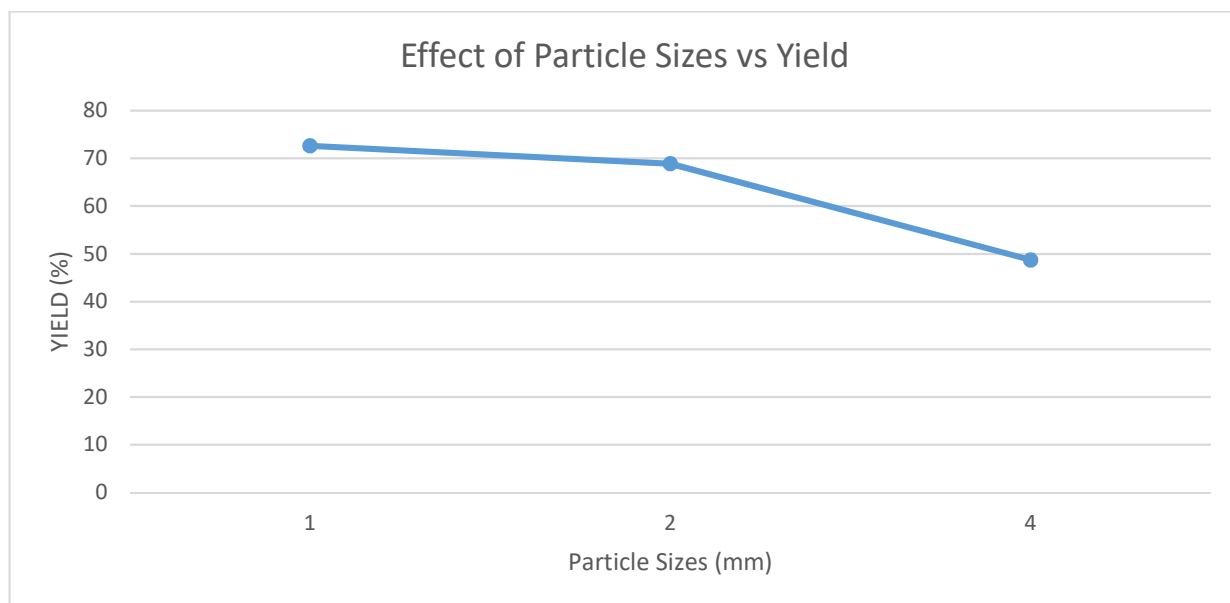


Figure 4.5: Catalyst Particle Size vs Yield

The higher surface area of the catalyst would promote greater chance of collisions between the reactants and the catalyst, thus increasing the rate of transesterification reaction

4.3.3 Effect of oil to reaction temperature

Figure 4.3 shows the biodiesel yield from transesterification of palm kernel oil at different reaction temperature from 40°C to 70°C. The biodiesel yield increases with the reaction temperature until an optimal point of 60°C with a biodiesel yield of 72.6%. Beyond this, the yield was decreased abruptly to 68.3% at 70°C. As the temperature increases and reaches the boiling point of methanol (64.5°C), the methanol will quickly vaporize and form a large number of bubbles, which inhibits the reaction on the two-phase interface and thus decreases the biodiesel yield.

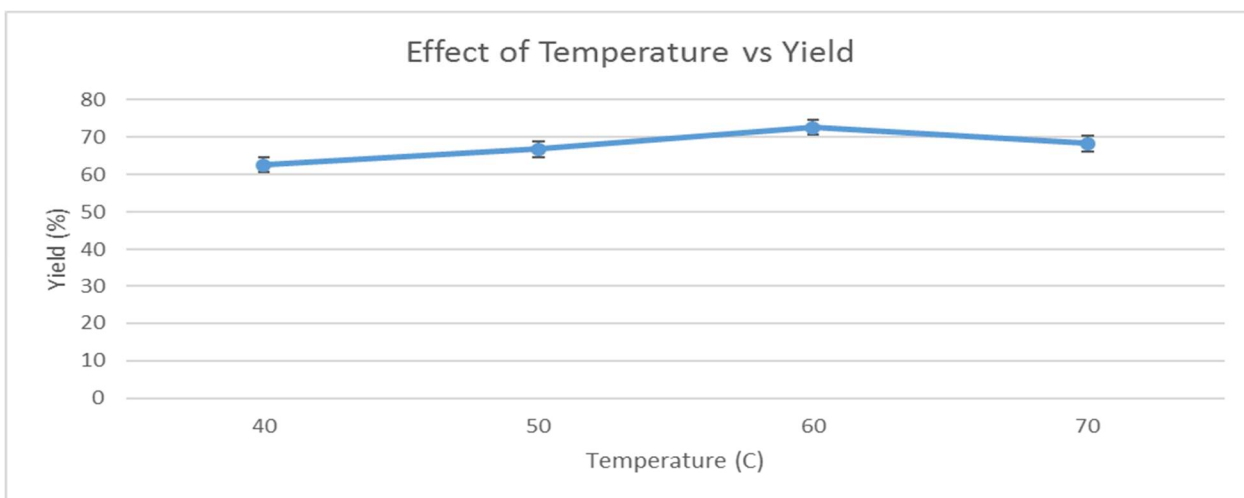


Figure 4.6 Reaction Temperature vs Yield

4.4 Optimal reaction conditions

It can be deduced from the study of the relationship and graphical representation of various reaction condition for biodiesel production from Palm kernel oil using calcined cow bone as heterogeneous catalyst, the optimal conditions are reaction time of 60mins, catalyst particle size of 1mm, methanol to oil ratio of 12:1 at a catalyst loading of 10% weight of oil.

4.5 Properties of biodiesel produced with the optimal reaction parameters

The produced biodiesel using the determined optimal parameters was analyzed and the results stated in Table 4.5 below. As can be seen from the table all the properties of biodiesel obtained at optimized conditions is well within the prescribed limit of ASTM standards.

Table 4.7: Comparison of Properties of Biodiesel Obtain at Optimum Condition with ASTM Standards

Property	Biodiesel from this work	Test Method	Biodiesel standard ASTM D6751	Petro-diesel
Specific gravity	0.87	D1298	0.88	0.84 - 0.86
Kinematic viscosity (mm ² /s)	4.99	D445	1.9-6.0	2.04 - 3.23
Flash point (°C)	160	D93	130- 260	68 - 94
Pour point (°C)	0	D97	-	-15
Cloud point (°C)	4	D2500	15	-12
Saponification value (mgKOH/g oil)	179.60		-	-
Iodine value (g I ₂ /100 g oil)	70.55	D664 11	-	-
Acid value (mgKOH/g oil)	0.56	D664	0.8 max	-
Cetane number	60.82	D613	47-65	41-48

4. CONCLUSION AND RECOMMENDATION

4.1 Conclusion

In conclusion, the calcined cow bone shows good catalytic performance and physicochemical properties as a heterogeneous catalyst for biodiesel production. The optimum parameters of the heterogenous catalyzed transesterification of palm kernel oil were determined as 1:12 oil-to-methanol molar ratio, 1mm catalyst particle size, 60°C reaction temperature, and 3-hour reaction time at 10% wt./wt. of oil catalyst loading.

4.2 Recommendations

This study has demonstrated the possibility of using calcined cow bone as an heterogenous catalyst for the transesterification of palm kernel oil to biodiesel; and it has shown the extent at which the type of feedstock can have an impact on the biodiesel quality; further studies may focus on the use of other feedstocks with lower FFA content and other source of heterogeneous catalyst.

This study presents a biodiesel production method with a potential economic advantage; however, a detailed process cost evaluation was not carried out, future work may look into the quantitative costs of production for the scale up commercial production process.

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