Corrosion Behaviour of Mild Steel in Biodiesel Prepared from Ghee Butter

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***Abstract*— In this this, biodiesel samples were produced from Ghee Butter (popularly called Mann Shanu in Northern Nigeria) which was obtained from a local market in Yola Northern Nigeria. The Ghee butter was purified in order to get rid of its moisture; it was then reacted with alcohol in esterification reaction to reduce free fatty acids (FFA) and subjected to trans-esterification reaction to produce biodiesel. Two samples (1 and 2) of biodiesel having percentage yields of 41.5% and 39.5% respectively were produced. The physical properties of biodiesel samples produced were compared to those of mineral diesel sample and it was observed that the biodiesel samples exhibited close values to the mineral diesel. Sample 1 exhibited higher viscosity at lower and higher temperatures compared to sample 2. The American petroleum institute (API) gravity for all the samples studied including the mineral diesel was 34.97. The heating values were 37.15, 36.50, and 43.80 MJ/kg for samples 1, 2 and diesel respectively while the cloud points obtained for samples 1, 2 and diesel were 5, 6 and -3^{o}C, respectively. An accelerated corrosion study of mild steel in the prepared biodiesel was investigated for thirty days using the conventional weight loss measurement method. The analysis of the results obtained showed that sample 1 was more aggressive to this material compared to sample 2, while diesel was least corrosive. At lower temperature, sample 2 was more corrosive than sample 1. The results obtained from this study show among other information, that the physical and chemical properties of the prepared biodiesel samples were closely matched with those of mineral diesel and the former can be used as alternative for mineral diesel.**

Keywords — *Corrosion, Mild steel, Biodiesel, Ghee Butter.*

I. INTRODUCTION

The over dependent of the world energy source on fossil fuels, price instability, environmental pollution and fear of extinction of this black gold are some of the reasons why researches have been geared towards alternative sources of energy [1–3]. Renewable energy which can be regenerated once used up is most highly favoured among other sources of alternative energy. Biodiesel is one good source of these highly favoured sources of energy!

Petroleum, for decades, has been the major source of energy used worldwide and our society today depends highly on petroleum and its various products. About 90% of petroleum products are used as energy source for transportation, heating, cooking, electricity generation, as well as feedstock in chemical industries [4]. Despite its uses, petroleum is a non-renewable energy source and causes various environmental problems such as global warming, air/water pollution etc. As demands for energy are increasing and fossil fuels are limited, researches are directed towards alternative renewable fuels.

Renewable energy is energy which comes from natural resources such as hydropower, wind, solar, tides, *biomass* and geothermal [5,6]. Fig. 2 illustrates the global renewable energy consumption.

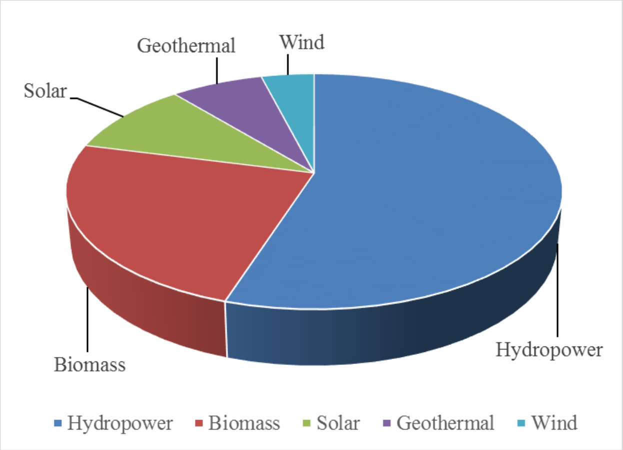

Fig. 1 Global renewable energy consumption

From Fig. 1, hydroelectric power provides over half of the renewable energy consumed globally. This chart does not include the fuel-wood used for energy consumption in developing countries. Bioenergy are usually obtained from *biomass* (organic material from living or recently living organisms – plant and animals). These include wood from trees, charcoal from burned wood, crops, and combustible animal waste products. Bioenergy comes in form of direct combustion, bio-power, and biofuels. Biodiesel, a form of biofuel, can be produced from vegetable oil, used cooking grease, or animal fat, with small amounts of ethanol or wood alcohol (methanol) Biodiesel fuels are attracting increasing attention worldwide as a blending component or a direct replacement for diesel fuel in vehicle engines [6,7].

Some of the advantages of using biodiesel among many are that it is a renewable fuel that can be obtained from vegetable oil and animal tallow; hence, it has low toxicity when compared to diesel obtained from fossil fuels [8–15]. Biodiesel degrades more rapidly than the mineral diesel fuel, minimizing the environmental consequences of biofuel spills [16]. It also has reduced health risk as a result of

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reduced emission of carcinogenic substances and absence of sulfur dioxide. Biodiesel is sustainable; it can be obtained from used cooking oil and residues of fat. This can improve the economy of a country, as well as preserve resources for future generations.

Owing to the advantages of biodiesel, several works had been done on the conversion of various fats and oils to biodiesel [17–20]. However, little or no work had been carried out on the conversion of Ghee butter (locally referred to as Mann Shanu; the fat from raw milk) to biodiesel. This study is therefore aimed at producing biodiesel from Ghee butter using trans-esterification process. The investigation will also evaluate the properties of the biodiesel, such as: specific and API gravity, viscosity, density, pour point, and cloud point and compare these properties to those of standard mineral diesel. Biodiesel, which is hygroscopic in nature (i.e. absorbs water readily from the atmosphere), is used mostly in diesel engines which are basically made of metallic materials. It is therefore worthwhile to study the dissolution of this material in this medium to be able to predict their behaviour and protect their integrity in this medium. This is the motivation behind this study.

II. MATERIALS AND METHODS

The feedstock used in this experiment is Ghee butter (locally referred to as Mann Shanu), obtained from Jimeta Ultra-modern market, Jimeta-Yola, Adamawa state. Some characteristics of the Ghee butter are summarized in Table I.

TABLE I
SOME CHARACTERISTICS OF GHEE BUTTER

Characteristic	Value	Reference
Density (g/cm^3)	0.917	Nil
Specific gravity	0.921	Nil
Pour point (^{o}C)	35	Deshpade & Kulkarni, 2012
Iodine value	36.7	Deshpade & Kulkarni, 2012
Sulfur content (ppm)	18	Visser, 2012

Table II shows the some properties of the reagents used both as reactants and catalysts in this experiment.

TABLE II
CHEMICALS USED IN THIS STUDY

Reagents	Chemical formula	Boiling Point	Purity	Purpose
Methanol	CH_3OH	64.7^{o}C	99.5%	Reagent for first sample
Ethanol	C_2H_5OH	78.37^{o}C	99.5%	Reagent for second sample and washing of mild steel
Sodium hydroxide	NaOH	1388^{o}C	85%	Catalyst
Sulphuric acid	H_2SO_4	337^{o}C	32%	Catalyst neutralizer

About 800 ml of the feedstock (Ghee butter) was measured into a beaker for purification by boiling at 110^{o}C (above the boiling point of water) so that its water-content can be evaporated. The purification process was done in order to liquefy the Ghee butter, as well as remove impurities and moisture from it.

For the first-step esterification, 100 g of the purified Ghee butter was put in a beaker, placed on a heating mantle, and heated to a temperature of 75^{o}C. Then, 0.51 g of concentrated sulphuric acid was mixed with 22.77 g of methanol and heated separately to a temperature of 50^{o}C. The sulphuric acid – methanol mixture was then poured into the beaker containing 100 g of Ghee butter; the mixture was stirred for 60 minutes, while maintaining a temperature of 75^{o}C. After stirring for 60 minutes, the mixture was then transferred to a round bottom flask, which was fitted with a reflux condenser, for the second-step esterification. The same procedure for the first-step esterification was done in the second-step esterification. The reflux condenser, for the second-step esterification, was cooled with tap water; water flow in and out of the condenser was not measured, but was kept constant all through the experiment. Just as in the first-step esterification, the reaction was stirred for 60 minutes; for the second-step esterification, the reaction was then allowed to settle for an additional 4 hours in a separation funnel. After separation, the bottom layer was collected and heated to 110^{o}C for 30 minutes and allowed to cool; this was done in order to evaporate its water-content.

For alkali transesterification reaction, 100 ml of the obtained ester (oil) was heated in a beaker to a temperature of 110^{o}C to evaporate any extra water content. A solution of sodium meth-oxide was prepared in a separate beaker, by dissolving 2 g of NaOH pellets (catalyst concentration of 0.5%) into 300 ml of methanol (ratio of oil to alcohol 1:3). The solution was heated to 60^{o}C and rigorously stirred until the NaOH has dissolved completely. The sodium meth-oxide solution, together with the oil, was then poured into the three-necked round-bottom flask. At a constant temperature of 70^{o}C, the mixture was stirred carefully for 60 minutes using a magnetic stirrer, and was left to settle for 24 hours in a separation funnel. After the mixture had settled for 24 hours, the bottom layer (glycerol and soap) was collected and disposed. Warm water was then used to wash the top layer (biodiesel). This was done in order to remove any excess glycerol that remained in the separation funnel; the biodiesel, mixed with water, was then placed on the heating mantle and allowed to heat until all of the water has evaporated. The same procedure involving purification, two-step esterification, and trans-esterification was repeated in order to produce a second sample of biodiesel however, ethanol was used instead of methanol as in production of sample 1.

The percentage yield of the prepared biodiesel sample was determined using equation (i), to evaluate the production efficiency of biodiesel produced from Ghee butter using two different alcohols.

$$\%\text{Yield} = \frac{\text{Volume of biodiesel produced (actual yield)}}{\text{Volume of Ghee butter used (theoretical yield)}} \times 100 \qquad \text{(i)}$$

The specific gravity, the mass and volume of the biodiesel were measured and recorded. The mass and volume of water was also measured and recorded. Hence, the specific and API gravity values were calculated using the formulas in equations (ii) and (iii) respectively.

$$\text{Specific gravity} = \frac{\text{Density of biodiesel}}{\text{Density of water}} \qquad \text{(ii)}$$

$$\text{API gravity} = \frac{141.5}{\text{Specific gravity}} - 131.5 \qquad \text{(iii)}$$

The viscosity of the biodiesel was measured using a viscometer (size 72980); which is normally used to measure fluids under one flow condition. 15 mL of the biodiesel produced was measured and poured into the viscometer and was then lowered into the immersion bath. The immersion bath was set to a temperature of 30^{o}C and the temperature of the biodiesel was allowed to be uniform with that of the immersion bath. The pipette bulb was then used to pull the biodiesel sample along the viscometer to the mark; at a stable temperature, the bulb was released. When the biodiesel sample got to the first line, the stop watch was started and stopped when the biodiesel got to the second marker; the time taken for the biodiesel to get to the second marker, from the first, was recorded. The same procedure was repeated for temperatures 35, 40, 45 and 50^{o}C.

The viscosity values of the biodiesel samples were calculated using equation (iv):

$$V = Kt \qquad \text{(iv)}$$

where V = kinematic viscosity (mm^2/s) K = calibration constant (for size 72980-viscometer, K = 2.825) and t = average time of flow (s). The density of the prepared biodiesel samples was determined by measuring a certain volume of the biodiesel in a measuring cylinder, and then measuring the corresponding mass using a weighing balance. The density values were calculated using equation (v).

$$\text{Density } (\rho) = \frac{Mass\ (m)}{Volume\ (V)} \qquad \text{(v)}$$

A test-tube fitted with a cork and thermometer was used to determine the pour and cloud points of the biodiesel. Sample of the prepared biodiesel was poured into the test-tube and it was heated in a heating mantle to a desired temperature to obtain clarity and uniform consistency, while the thermometer was used to check and observe the temperature of the biodiesel. The test-tube was then removed after clarity and consistency was observed in the biodiesel and was inserted uprightly in an ice bath. At every 2^{o}C temperature-drop interval, the cloudiness at the bottom of the test-tube was observed. The temperature at which crystals or cloud (cloud point) formed was observed and recorded. After the cloud point was determined, the test-tube was allowed to cool further in the same manner. The temperature of the biodiesel at which it did not flow after about 5 seconds when placed horizontally was recorded as the pour point.

Three sample media were used to study the corrosion behaviour of mild steel (composition: Fe = 98.4%, C = 0.2% and others balance) in biodiesel. These three samples include the two biodiesel samples produced and mineral diesel obtained from Con-oil. All the experiments were carried out in triplicates and the average values were recorded. Cylindrical mild steels of surface areas 2 cm^2 were weighed and recorded and then placed in different beakers, which contained the biodiesel samples and the mineral diesel sample. The beakers containing the biodiesel and diesel samples, with the weighed mild steels, were immersed in water baths at different temperatures, and weight loss measurements were conducted for all the samples in the media using a sensitive electronic weighing machine for thirty days at five-day interval following the procedures and precautions described by Afolabi [21], Bastidas *et al*, [22], Kolman *et al*, [23], and Ashassi-Sorkhabi *et al*, [24].

The weight of the mild steels in each biodiesel/diesel samples before and after being washed with ethanol was measured and recorded and the values were used to calculate the rate of corrosion using equation (vi).

$$\text{Corrosion rate} = \frac{W \times 87.6}{D \times T \times A} \qquad \text{(vi)}$$

where W = weight loss (mg), D = density (g/cm^3) A = exposed surface area (cm^2), T = time of exposure (h) and corrosion rate is in mils per year (mpy; 1 mil = 0.001 in).

III. RESULTS AND DISCUSSION

Table III shows the results obtained from the analyses of the properties of the biodiesel produced from Ghee butter in comparison to mineral diesel. It can be observed from these results that the properties of both samples of biodiesel and mineral diesel are closely rather similar. For instance, the specific gravity values of both samples of biodiesel are 0.85 while this value reads 0.86 for mineral diesel. The API gravity values of all the samples are the same, while the heating of the mineral diesel is slightly higher than those of biodiesel produced from Ghee butter. The pour and cloud points values of the biodiesel samples are relatively higher than those of mineral diesel.

TABLE III
SOME PROPERTIES OF BIODIESEL SAMPLES PRODUCED

Properties	**Biodiesel**		**Standard Diesel Fuel**
	Sample 1	**Sample 2**	
Percentage yield (%)	41.5	39.5	-
Density g/cm^3	0.85	0.85	0.854
Pour Point (^{o}C)	2	2	-6
Cloud Point (^{o}C)	5	6	-3
Specific gravity	0.85	0.85	0.86
Heating value (MJ/Kg)	37.15	36.50	43.8
API Gravity	34.97	34.97	34.97

The heat of combustion which is heating value is a measure of the energy content in fuels. It is an important property of biodiesel that determines the suitability of these materials as alternative to diesel fuel [25]. The heating value of the biodiesel samples 1 and 2 were obtained to be 37.15 MJ/Kg and 36.50 MJ/Kg, respectively. These results showed the effect of the different alcohols used in producing these biodiesel samples. It was shown that ethanol would produce biodiesel of less heating value if Ghee butter is used as the precursor for the biodiesel. Since the esters obtained for both biodiesel samples were denser, the energy content of a full tank biodiesel fuel would be only 4-9% less than diesel fuel.

The viscosity values of both biodiesel samples 1 and 2 at different temperatures are shown on Tables IV. Viscosity can be said to be the most essential property of biodiesel, as it directly affects the operation of fuel injection equipment, most especially at low temperatures. Viscosity affects fuel's fluidity, the higher the temperature the lower the viscosity

(Fig. 2). Biodiesel with low viscosity are easy to pump, atomize, and achieve fine droplets than those with high viscosity. Due to the high viscosity of pure vegetable oil and tallow such as Ghee butter (between 27.2 – 53.6 mm^2/s), they cannot be used directly as diesel for engines. Hence, these oils need to undergo trans-esterification reaction, which was done in this study. The viscosity values of vegetable oil methyl esters decrease sharply following the trans-esterification process [25]. In this study, the kinematic viscosities at 40°C were 5.20 mm^2/s and 5.22 mm^2/s for biodiesel samples 1 and 2, respectively. The kinematic viscosities were observed to decrease with increase in temperature and for both biodiesel samples, the kinematic viscosities ranged from 3.3– 7.2 mm^2/s while lower viscosity values were expectedly observed at higher temperatures.

Table IV
KINEMATIC VISCOSITY FOR SAMPLES 1 AND 2

Temp (°C)	Viscosity mm^2/s Sample 1	Viscosity mm^2/s Sample 2
30	7.15	6.92
35	6.23	6.52
40	5.20	5.22
45	4.45	4.31
50	3.39	3.36

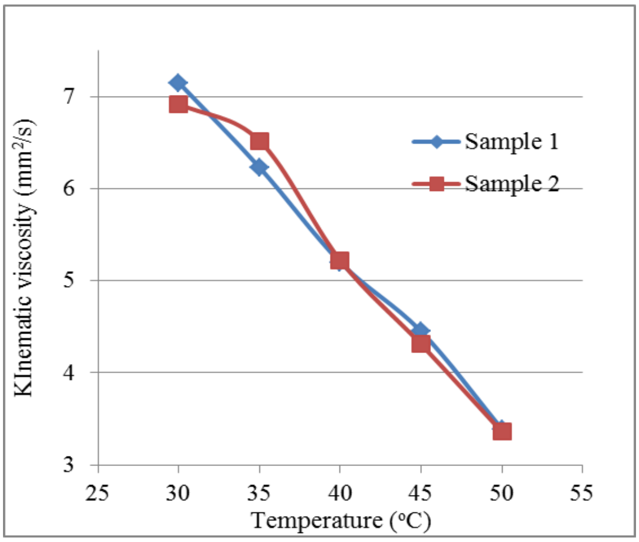

Fig. 2: Kinematic viscosity of biodiesel samples

Most of the currently designed engines in our society today use gasoline or diesel as fuel. The corrosion behaviour of most metallic materials has been reported to be higher in biodiesel than in petro fuel medium [26]. In view of this, the corrosion behaviour of mild steel was investigated in the samples of biodiesel produced from Ghee butter and mineral diesel sample by weight loss measurements at different temperatures in this study. The data obtained were plotted in Figs. 3 and 4. It can be observed from Fig. 3 that the weight losses in the biodiesel samples were quite close to those of the mineral diesel sample. The response of the mild steel is similar in all the media studied. The first twenty days of immersion in the media produced considerable weight loss and thereafter a stable condition occurred which can be attributed to passive film on the surface of the steel samples in these media. Higher weight loss is observed in samples 1 and 2 than the mineral diesel.

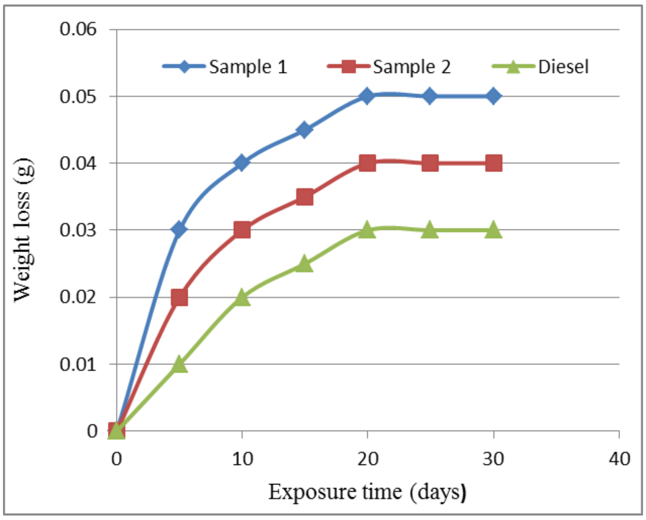

Fig. 3: Weight loss of mild steel in biodiesel/diesel samples at room temperature

As stated by Singh *et al.* [26], the rate of corrosion is influenced by temperature, water content, microbial growth, and type of feedstock used for synthesis of biodiesel. The feedstock used in this study, Ghee butter, contained excess FFAs (which required a two-step esterification reaction). Due to particles of FFAs still present in the feedstock after the two-step esterification, the biodiesel samples were a bit more corrosive than mineral diesel when the rate of corrosion and weight loss was determined.

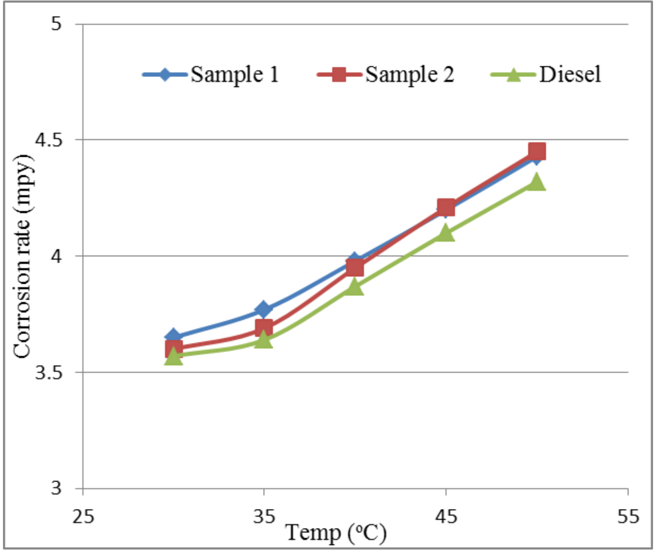

Fig. 4: Corrosion rate of mild steel in biodiesel/diesel samples at different temperatures

The corrosion behaviour of mild steel in these media is proportional to temperature which indicates that the higher the temperature of the environment (biodiesel and mineral diesel samples in this study), the higher the rate of dissolution of mild steel in this study as shown in Fig. 4. The rate of weight loss at lower temperatures was in the range of sample 2 < diesel < sample 1, while at higher temperatures it was diesel < sample 1 < sample 2. These results indicate that the higher the kinematic viscosity, the higher the rate of corrosion. At lower temperatures, sample 1 exhibited higher kinematic viscosity which led to its high aggressiveness at lower temperatures. However, the dissolution of the mild steel in sample 1 decreased at higher temperature as a result of its lower kinematic viscosity. It can also be observed that at temperature of 40°C, oxide films were formed on the surfaces of the mild steels which led to a little increment in the weight of the mild steels. Ethanol was used to wash away these particles, which then reduced the weight of the mild steel a little.

IV. CONCLUSIONS

This study reports the production of biodiesel from Ghee butter (Mann Shanu) by trans-esterification reaction. Two samples of biodiesel produced using ethanol (1) and methanol (2) were characterized and the analyses of the results obtained indicated that the properties of both samples were very similar to those of mineral diesel but the biodiesel sample 1 yielded lower biodiesel, as compared to the sample produced with methanol. The kinematic viscosity at 40°C of samples 1 and 2 were 5.20 and 5.22 mm^2/s, respectively. The corrosion behaviour of mild steel in both biodiesel samples at varying temperatures was as low as that in the mineral diesel. Sample 2 showed lesser dissolution of mild at low temperature while sample 1 produced a better resistant at high temperature. Since almost all the properties of the biodiesel obtained from Ghee butter were closely matched with those of mineral diesel, it can be recommended that the use of biodiesel from Ghee butter can be adopted in the sub-Saharan Africa where this material is abundantly available with limited applications despite its usefulness.

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