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Effect of Oxidation Stability on the Fuel and Storage Properties of *Balanites Aegyptiaca* Biodiesel

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ABSTRACT

Biodiesel prepared from Balanites aegyptiaca oil, through transesterifcation process, has promising potentials for use as fuel for diesel engines. However, there is limited information regarding the physical and chemical stability of this biodiesel during storage. In view of the aforementioned, this study was carried out in order to determine the oxidation stability as well as the extent of deterioration of Balanites aegyptiaca biodiesel during storage. Storage stability study was carried-out for three (3) months with the view to investigate the effect of oxidation on the biodiesel. The induction period and storage stability parameters were determined in accordance with official standard methods. The Balanites aegyptiaca biodiesel was found to have an induction period of 66 minutes. The storage stability study shows that: the peroxide value increased from 1.4 mEq/kg to 9.4 mEq/kg; cetane number increased from 50.42 to 52.48; while acid value increased from 0.11 mgKOH/g to 0.36 mgKOH/g. In addition, changes were observed in: saponification value from 215.99 mgKOH/g to 220.19 mgKOH/g, kinematic viscosity from 4.7 mm2/sec to 5.0 mm^2/sec at 40˚C and density from 887 kg/m^3 to 897 kg/m^3 at 15˚C. The iodine value and lower heating value decreases from 68.53 gI$_2$/100g to 55.84 gI$_2$/100g and from 37.5 MJ/kg to 36.2 MJ/kg respectively during the storage period. However, despite the variations observed during the storage, the parameters were still within the ASTM and European specification standards for biodiesel. The result suggests that the Balanites aegyptiaca biodiesel may be stored for up to three months without losing its fuel properties.

Keywords: Balanites aegyptiaca; biodiesel; storage stability parameters; oxidation stability

INTRODUCTION

The major problem that hinders biodiesel commercialization is its poor oxidation stability which plays an important role in determining its quality (Ahmad, 2017). Oxidation stability is the resistance of the fuel to degradation through oxidation with air. Oxidation of the fuel during storage presents an important concern for biodiesel quality which may manifest in terms of deterioration of its kinematic viscosity, acid value and peroxide value (James, 2013). The products of oxidation make biodiesel acidic and facilitate the formation of insoluble gums and sediments, thereby making it unstable during storage. This phenomenon gives rise to the formation of undesirable substances in biodiesel beyond acceptable limits specified by the American Society for Testing and Materials (ASTM D6751) and European Standards (EN 14214) (Musa et al. 2016). Fuel oxidation can contribute to fuel filters plugging which, adversely, affects fuel delivery system of an engine.

Desert date (Balanites aegyptiaca) also called "*Adowa*" (in Yoruba), "*Aduwa*" (in Hausa), "*Tanni*" (in Fulfulde), "*Cungo*" (in Kanuri), is a semi-evergreen plant usually spiny, that grows up to 12 m high in the semi-arid regions of Northern Nigeria. The plant is from Zygophyllaceae family, and has been used as a potential source of medicines, animal feed, soap and fuel wood. Other uses include production of hand tools, praying beads (seibha), saddle and furniture (Donnell 2014). Clement (2010) reported the composition of the plant fruit layers in percentage of the fruit weight for epicarp, mesocarp, endocarp and kernel to be 19.6%, 33.2%, 36.8% and 19.6% respectively. The plant has major biological active components that are responsible for its nutraceutical values

(Azene 2015). The fruit (mesocarp) is edible and contains essential minerals. Beside this, the leaf of the plant is one of the favorite fodders for goats. All parts of the plant have various pharmacological benefits. The fruits and roots of the plant contains diosgenin, which can be used for the production of oral contraceptive and steroids.

Extracts from many parts of this plant have been intensively used in India and Africa for different ethno-botanical uses. As reported by Hena et al. (2017) several parts of the plant have been used traditionally, in Nigeria, for the treatment of several diseases and disorders such as skin diseases, stomach ache, whooping cough, jaundice and as an antidote for snake bite. The kernel cake after extraction of the oil is a source of protein and carbohydrates; making it suitable for the production of feeds for livestock.

Balanites aegyptiaca seeds have been reported to have an oil yield of about 40-50% (Clement 2010). A number of works have revealed that triglycerides shown promise as alternative diesel engine fuel. However, the direct use of vegetable oil is generally considered to be unsatisfactory and impractical for both direct-injection and indirect-type diesel engines (Mogire 2013). The Problems faced in substituting triglycerides for petroluem diesel fuels are mostly associated with high viscosity, low volatility and polyunsaturated character. Blending and heating of vegetable oil may improve volatility and reduce the viscosity of the vegetable oil but its molecular structure remains unchanged, as such its polyunsaturated character remains unchanged as well. The effective way to overcome all the problems associated with the vegetable oil is by conversion of the vegetable oil into fatty acid methyl esters.

The most common way of producing biodiesel is the transesterification of vegetable oil as reported by Zahira et al. (2014). Two other processes are thermal cracking (pyrolysis) and micro-emulsification. The alcoholysis process (transesterification) is the displacement of alcohol by another alcohol from an ester. The process involves reacting triglyceride, such as one of the vegetable oils, with an alcohol in the presence of a catalyst to produce free glycerol and fatty acid esters (Harrison 2011). The reporter added that, in such reaction, triglyceride is allowed to react with excess alcohol such as ethanol or methanol, which takes the place of the ester linked to glycerol, producing glycerol, and the new alcohol's three fatty acid esters.

Biodiesel is produced globally and the source of the oil depends on where the feedstock is grown. In Africa and India, oil producing plants that are resistant to drought and can grow on non-arable land such as Jatropha and Karanja are used (Nada 2011). In Nigerian dryland areas Usman (2019) reported that *Balanites aegyptiaca* biodiesel possessed biodiesel fuel properties as the fuel properties are proper in respect of ASTM D6751 and EN14214 specification standards. As such, it was suggested that *Balanites aegyptiaca* seed could be used as feedstock for biodiesel production and its biodiesel as alternative fuel for internal combustion engines.

The oxidation degradation resistance during storage for biodiesel is an important issue for the sustainability and viability of an alternative fuel. To measure the oxidative stability of fatty acid methyl ester, a standardized test method also known as the Rancimat test has been developed. This accelerated oxidation requires any biodiesel to have an induction period of at least 3 hours at 110 °C. This value has been determined to provide biodiesel with a shelf life of one year when stored under ambient conditions (Dantas et al. 2011). The induction period represents the time during which the oxidation of fatty acids methyl ester (FAME) is being suppressed.

Biodiesel oxidation causes fuel injector deposits, injector coking and corrosion. The by-products of oxidation such as carbonyls and acids, accelerate the degradation process and causes corrosion. Degradation of by-products may cause filter plugging in fuel systems, which may lead to operability problems. Problem in operability usually increases maintenance costs and decreases equipment reliability. Subsequently, problems related to biodiesel exploitation could affect the growth and marketability of biodiesel (Yoon et al. 2007). Therefore, preventing operability problems can potentially improve the viability of biodiesel as a fuel for internal combustion engines.

Gregory (2015) demonstrated that biodiesel made from waste cooking oil shows good biodiesel fuel properties, but requires antioxidants to meet oxidation stability specifications set forth in ASTM D6751 or EN14214. Harrison (2011) also reported that the addition of antioxidant would be sufficient for meeting existing ASTM specifications for biodiesel oxidative stability. The production of biodiesel from cottonseed oil and canola oil were carried out by Hem (2008) using low molecular weight alcohols and potassium hydroxide as catalyst. The FAME produced from cottonseed oil had superior oxidative stability to that produced from canola oil, but haven't satisfied specification standards for oxidation stability.

The ability of liquid fuel to resist change to its chemical and physical characteristics brought about by its interaction with its storage environment is called Storage stability (Robert et al. 2009). These instabilities in fuel may give rise to the formation of some undesirable substances in the biodiesel above specified limits as per global biodiesel specification standards (Kivevele & Zhongjie 2014). A number of studies related to the oxidation and storage stability of biodiesel derived from less common tree-borne non-edible oil seeds under different conditions have been reported in the literature.

Storage stability was first studied by Du Plessia et al. (1985). They monitored the production of peroxides, acids, and aldehydes, as well as increase in kinematic viscosity and decline in Rancimat induction time over a period of 90 days. Ahmad (2017) investigated the oxidation stability of Jatropha biodiesel and its blends; he reported that storage in the presence of oxygen was considered as potential elements affecting oxidative degradation. The author added that the acid value (AV), peroxide value (PV), and viscosity (ν) increased with storage time while the iodine value (IV) on the contrary exhibited a decrease. Another study by Earl and Robert (2014) demonstrated that a decrease in induction time indicated loss of stability (consumption of antioxidant); therefore, recommended that induction time monitoring should be used for predicting quality changes during storage. In addition, Ayoola et al. (2016) investigated the production of biodiesel from waste groundnut oil and reported that vacuum is the most favorable storage medium, compared to freezing and atmospheric conditions. Musa et al. (2016) reported the long-term oxidation stability of biodiesel produced from vegetable oil (peanut, palm kernel and moringa oil). The results from their study hat moringa methyl ester and palm kernel methyl ester blends can be effectively stored under atmospheric conditions and still maintain a shelf life of six (6) months.

Thoyajakhsi (2013) characterized biodiesel produced from *Pongamia pinnata (L.) Pierre*, where the results revealed that most of the quality parameters of biodiesel varied with storage period. The storage period had effect on quality parameters varying marginally up to100 days as compared to control in the case of open and closed high density polyethylene (HDPE) containers. Biodeisel storage in metal containers under open and closed conditions revealed significantly different values from 60th day on wards. Additional work by Thompson (2008) was conducted to determine the extent of deterioration of rape methyl ester (RME) and rape ethyl ester (REE) in storage. The analysis involved triplicate samples of RME and REE stored in steel and glass containers at room temperature (inside) and at the local ambient outdoor temperatures (outside). On the average, the esters increase over time in all properties except its heat of combustion.

From the literature review, it was observed that biodiesel is prone to oxidation; hence storage of biodiesel over a period of time in order to study the effect of oxidation on the biodiesel is an important concern to be addressed.

Therefore, in the current study the storage and oxidation stability of *Balanites aegyptiaca* biodiesel is presented.

METHODOLOGY

DETERMINATION OF OXIDATION STABILITY

The aim of this experiment was to determine the induction period of the biodiesel sample according to European standard (EN14112) official method. 50ml of biodiesel sample was measured. A stream of purified air was passed through the biodiesel sample (50ml) kept at constant temperature of 110˚C in the Rancimat instrument shown in figure 1. The vapor released during the oxidation of biodiesel sample along with air were passed through the flask containing distilled water, which was equipped with an electrode for measuring conductivity. The electrode was connected to a measuring and recording device which indicated the end point of the induction period when the conductivity of water began to increase, which was as a result of dissociation of volatile carboxylic acids produced during the oxidation process. The measurement of this conductivity gives rise to an oxidation curve whose point of inflection is known as the induction period.

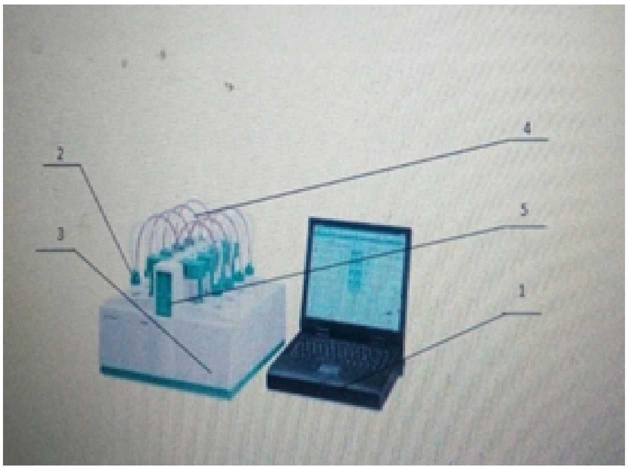

FIGURE1. Rancimat Instrument

DETERMINATION OF STORAGE STABILITY

The aim of this experiment was to study the storage stability of the biodiesel sample and was conducted according to the following procedure. 300 ml of the biodiesel sample was measured and stored in separate plastic sample bottles to assess the level of deterioration of biodiesel during storage at room temperature under normal atmospheric conditions. The storage stability of the B100 was evaluated by replicating the measurement of storage stability parameters namely: peroxide value, acid value, iodine value, calorific value, cetane number, density, saponification number and kinematic viscosity according to AOAC 1990, ASTM D445 and ASTM D7042 standard methods on monthly basis over a period of 3 months. The samples were taken at regular intervals and shaken vigorously for 5 minutes each time the analysis was carried out.

RESULTS AND DISCUSSION

OXIDATION STABILITY OF BALANITES AEGYPTIACA BIODIESEL

TABLE1. Oxidation Stability of Balanites aegyptiaca Biodiesel

Oxidation Stability Parameter	Units	Current Study	ASTM D6751	EN 14214
Induction period	hours	1.6	3.0 min	6.0 min

The oxidative stability of *Balanites aegyptiaca* biodiesel (table 1) shows that the biodiesel is having induction period of 1 hour 6 minutes after 1488 hours (two months two days) of production. This indicated that the biodiesel may not be stored for more than 2880 hours (four months) having not attained both ASTM and EN specification standards. Once this dead time is reached, the level of hydro peroxide increases rapidly, and results in an overall oxidation process. The result is comparable to the oxidative stability of Jatropha biodiesel, having the induction period of 1 hour 4 minutes after 1994 hours (two months three weeks) of production, as reported by Ahmad (2017).

IMPACT OF STORAGE ON STORAGE STABILITY PARAMETERS OF THE BIODIESEL

The variation of peroxide value (figure 2) was found to be increasing with storage period continuously from 1.4 mEq/kg to 9.4 mEq/kg for the methyl ester. Peroxide value measures biodiesel oxidation which takes place at faster rate due to inbuilt oxygen and unsaturated fatty acids. The result is quite impressive when compared with increase from 5 mEq/kg to 9.8 mEq/kg reported by Ahmad (2017) for Jatropha seed methyl ester after three months of storage. Although, peroxide value is not specified in the biodiesel fuel standard, this parameter may influence methyl ester content and cetane number as reported by Nada (2011). Increase in peroxide value may cause an increase in hydro peroxide formation which can give rise to sediments, gum formation and fuel darkness that can lead to fuel filter plugging and deposits formation in engine combustion chamber. This very increase in magnitude for peroxide value throughout the storage period might be due to presence of dissolved oxygen in the biodiesel which facilitated the development of hydro peroxides formation as suggested by Jain (2011).

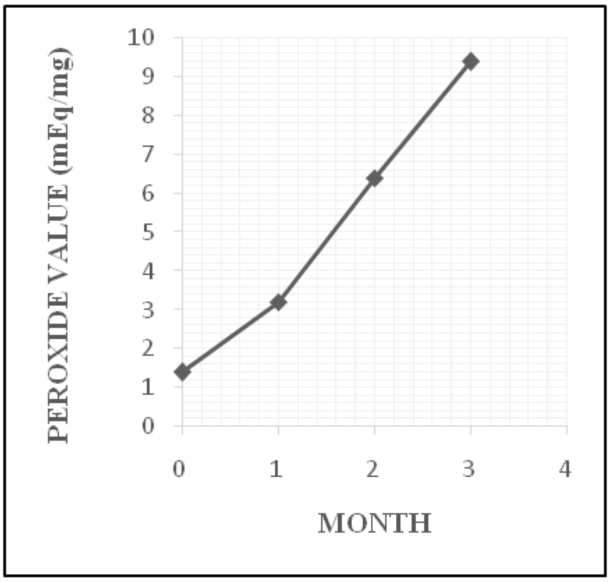

FIGURE 2. Variation of Peroxide Value with time

Acid value is one of the properties that indicate the extent of oxidative deterioration of biodiesel. The current study revealed that acid value increases, with increase in storage period, from 0.11 mgKOH/g to 0.36 mgKOH/g as shown in figure 3 which is in agreement with Musa et al. (2016) who reported for palm kernel (0.3-0.62 mgKOH/g) and moringa (0.22-0.56 mgKOH/g) methyl esters after eight months of storage. It may be interesting to state that even after 3 months storage period, the biodiesel still exhibited an acid value within the ASTM standard. The increase in acid value might be due to the corresponding increase in hydro peroxide which may be further oxidized to the acids during the propagation stage of oxidation (Nada, 2011). This behavior might be due to the presence of the dissolved oxygen remaining in the biodiesel sample that allows the formation of shorter chain fatty acids. High acid value in biodiesel may lead to corrosion in engines (Mogire, 2013).

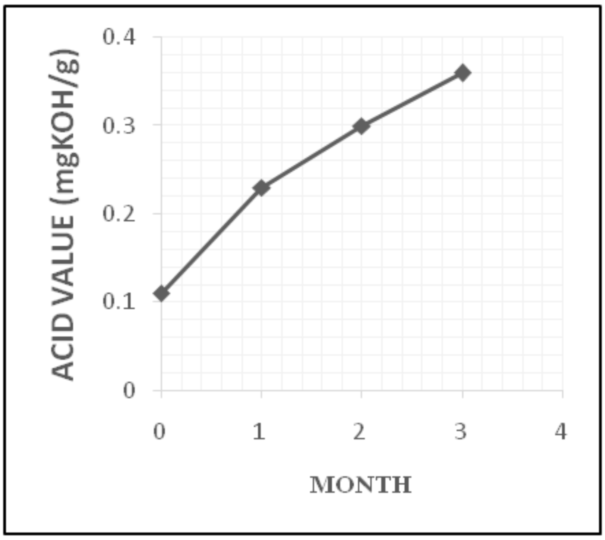

FIGURE 3. Variation of acid value with time

Biodiesel standard specification for density as per ASTM is 875-900 kg/m^3. The density of biodiesel sample determined in the present study as depicted in figure 4 was within the specification limits despite the small increase from 887 kg/m^3 to 897 kg/m^3 during storage, which might be due to moisture absorption and reaction with air (Musa et al. 2016). Oxidation of biodiesel may also form chemical substances like acids, aldehydes or soluble polymers which increases the density of biodiesel, this can give rise to blockage of fuel lines and filters (Fatih et al. 2014).

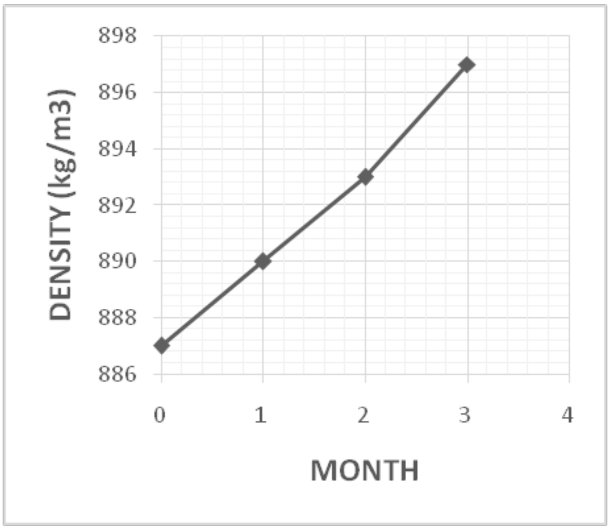

FIGURE 4. Variation of Density with time

The cetane number was found to be increasing with storage period (50.2-52.13) as depicted in figure 5. It is quite impressive to state that despite its increase the biodiesel still exhibited cetane number very close to the Standard. Ahmad (2017) also reported an increasing trend for Jatropha seed methyl ester after four months of storage. The increase in ignition delay quality parameter for Balanites aegyptiaca biodiesel might be due to variations in iodine and saponification number.

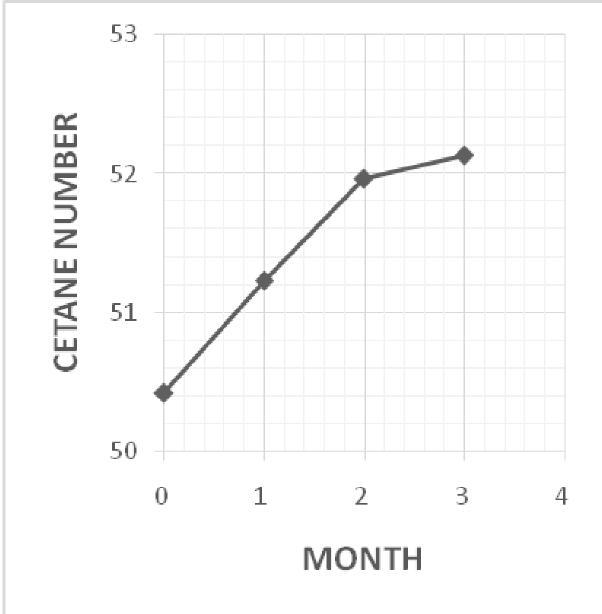

FIGURE 5. Variation of Cetane Number with time

There was a gradual decrease in iodine value of the biodiesel (68.53-55.84 gI2/100g) as depicted in figure 6. Despite the slight deviation, the value remains within the standard specification after three months of storage. Similar results were also reported for Jatropha biodiesel. Nada (2011) also reported decrease in iodine value for *Pongamia pinnata* biodiesel from 89.81 gI_2/100g to 56.48 gI_2/100g after three months of storage. This gradual reduction of double bonds might be due to the oxidation of the biodiesel which can also give rise to sediments and gum formation and fuel darkness that can result to fuel filter plugging and deposits formation in engine combustion chamber.

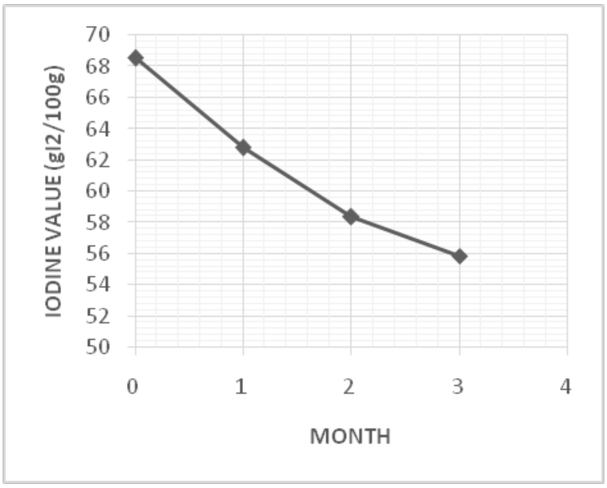

FIGURE 6. Variation of Iodine Value with time

The calorific value decreases with increase in storage period from 37.5 MJ/kg to 36.2 MJ/kg as depicted in figure 7. Similar result was reported for Jatropha biodiesel. This gradual decrease in the lower heating value of the biodiesel shows agreement with 40.6-40.1 MJ/kg and 40.4-39.8 MJ/kg reported by Thompson (1998) for Rapeseed methyl and ethyl esters respectively.

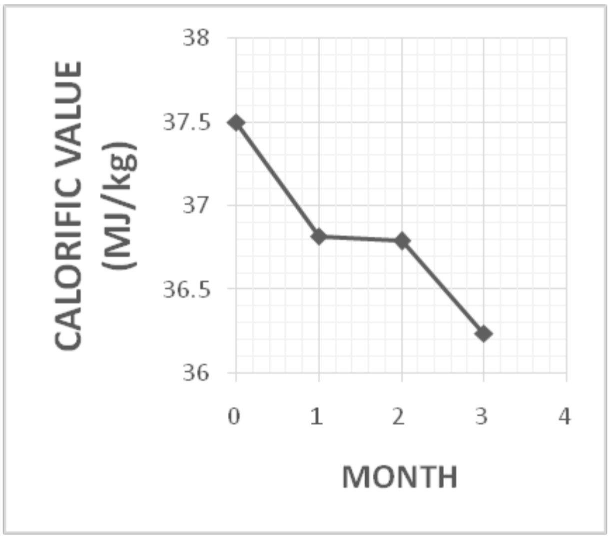

FIGURE 7. Variation of Calorific Value with time

The kinematic viscosity of the biodiesel increases with increase in storage period. Despite its increase from 4.7 mm2/sec to 5.0 mm2/sec during storage as shown in figure 8, the values remain within the standard specification. Ahmad (2017) reported that Jatropha methyl ester maintained a viscosity less than the ASTM D6751 upper limit of 6.0 mm2/sec after the 4 months of storage, while Musa et al. (2016) reported that kinematic viscosity of Peanut, Palm kernel and Moringa Methyl ester exceeded specification standard (6.0 mm^2/sec) after eight months of storage. This gradual increase in flow resistance of the biodiesel samples during storage might be due to the presence of some unsaturated alkyl esters that can simply oxidize to peroxides, acids and alcohols leading to the formation of insoluble gums and sediments as suggested by Fatih et al. (2014). These species with higher molecular weight are characterized with high viscosity and negative effect on the quality of stored biodiesel. This tendency can affect the atomization and spray pattern of fuel leading to incomplete combustion, severe carbon deposits and injector choking which may deteriorate engine performance as reported by Gregory (2015).

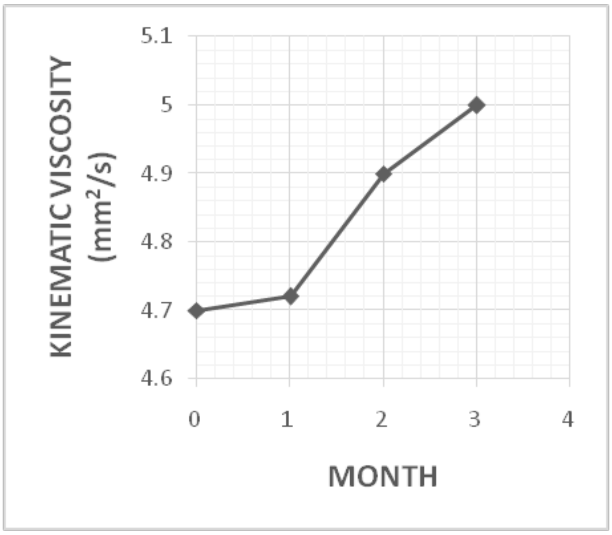

FIGURE 8. Variation of Kinematic Viscosity with time

CONCLUSION

The study revealed that Balanites aegyptiaca biodiesel, having induction period of 1 hour 6 minutes after 1488 hours (two months two days) of production, may not retain its fuel properties for more than 2880 hours (four months). During the storage period of three month, the following fuel properties increased: peroxide value (1.4-9.4 mEq/kg); cetane number (50.2-52.13); acid value (0.11-0.36 mgKOH/g); kinematic viscosity (4.7-5.0 mm^2/sec); and density (887-897 kg/m3). At the same time other properties decreased: iodine value (68.53-55.84 $gI_2/100g$) and calorific value (37.5-36.2 MJ/kg) in storage period. These indicated that the higher the number of months the greater the rate of oxidation. Despite these variations, it may be interesting to note that the biodiesel still exhibited values within the specification standard. Therefore, *Balanites aegyptiaca* biodiesel can be effectively stored under atmospheric conditions and still maintain shelf life of three (3) months. Hence, the safe storage period of *Balanites aegyptiaca* biodiesel could be three (3) months.

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DECLARATION OF COMPETING INTEREST

None

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