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Production of Biodiesel from Watermelon Seed (*Citrullus lanatus*) Oil via Alkali Catalysed Transesterification

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ABSTRACT

This study was focused on the production of biodiesel from watermelon seed oil via alkali catalysed transesterification. The oil was extracted from the seeds using a Soxhlet extractor with petroleum ether and then characterised. The effect of various factor on the transesterification of the extracted oil to produce biodiesel was performed at different conditions: ratio of oil to methanol (1:3, 1:6, 1:8 and 1:10), temperature (50 °C, 55 °C and 60 °C), catalyst concentration (0.5wt%, 1.5wt%, 2.5wt% and 3.5wt%), and stirring speed (250 rpm, 450 rpm, 750 rpm and 1600 rpm) for a total reaction time of 180 minutes. The biodiesel produced was characterized and its properties were compared to ASTM standard values to determine its suitability as a liquid fuel. Results obtained from the characterization of the watermelon seed oil showed that it was a suitable feedstock for biodiesel production as its properties were similar to those specified by the ASTM standards. The optimum conditions for the production of biodiesel were established as oil-methanol ratio (1:6), concentration of catalyst (1.5 wt%), temperature (60 °C), stirring speed (1600 rpm) and methanol volume (430 ml). The biodiesel yield at temperatures of 50 °C, 55 °C and 60°C were 85%, 92% and 95% respectively. The properties of the biodiesel produced were found to be very comparable with those specified by the ASTM standards. The results obtained in this study could serve as a platform for plant wide design and optimization of commercial scale biodiesel plants.

1. INTRODUCTION

With the ever-increasing global population and the attendant increase in the demand for energy sources that are more environmentally friendly, greater attention has been paid to alternative sources of energy such as biodiesel (Gomez *et al*., 2008). This has also been encouraged as a result of the growing concerns of the harmful effect of greenhouse gases (GHG) and other contaminants on the environment as result of the consumption of petroleum-based fuels. Bioenergy sources, particularly biofuels such as biodiesel, bioethanol, biogas etc have been identified as a way of drastically reducing the harmful effects associated with the emission of greenhouse gases (Hajjari et al., 2014). Other than greenhouse gas emissions, consumption of petroleum-based fuels contribute to the production of several air pollutants such as volatile organic compounds (VOCs), oxides of carbon (COx), oxides of sulphur (SOx), nitrogen oxides (NOx), and particulate matter (Klass, 1998).

As a result of the negative effects of harmful gases that come from the combustion of petroleum diesel, biodiesel has been established to be a viable alternative to petroleum-based diesel (Mardhiah et al., 2017; Hama et al., 2018; Sanli et al., 2018). Biodiesel is typically obtained from fat and oil-based feedstocks through a catalyzed transesterification process. Biodiesel as a fuel possesses numerous advantages and these include oxygenating properties, low emission profile, biodegradability, renewability, absence of sulphur, high flash point, inherent lubricity, positive energy balance, eco-friendliness, etc, (Altin et al., 2001; Hajjari et al., 2014).

Watermelon (*Citrullus vulgaris*) is a flowering plant which thrives in conditions of droughts. It is cultivated in large scale and commonly found in the southern part of Africa (Razavi and Milani 2006). The seeds of watermelon which is an otherwise waste material has been established to be rich in oil, hence the investigation into its potential use as a source of biodiesel (Rajeshwer Rao et al., 2012; Albishri et al., 2013). Research is actively investigating biodiesel production from watermelon seed oil to assess its potential. Furthermore, Efavi et al. (2018) compared the physico-chemical properties of watermelon seed oil with those of other vegetable oils and recommended its usage as a feedstock for biodiesel production. Ogunwole (2015) produced biodiesel from watermelon seed oil and characterised the biodiesel to determine its properties. Bhanu Teja and Alagumurthi (2018) investigated the emission characteristics of biodiesel produced from watermelon seed oil in a diesel engine and recorded a reduction in unburned hydrocarbons, CO and NO_X emissions. Other researchers have also investigated watermelon seed oil as a potential feedstock for biodiesel production (Moaddabdoost Baboli and Safe Kordi 2010; Ebiega and Garba, 2011; Aworanti et al., 2013). However, none of these studies have reported on the effect of process variables on the transesterification process.

Therefore, this study will evaluate the production of biodiesel from watermelon seed oil by assessing the effect of process variables such temperature, oil-methanol molar ratio, catalyst load, methanol volume and stirring speed on the biodiesel production process.

2. MATERIALS AND METHODS

2.1. Sample Collection and Preparation

The watermelon fruits were obtained from the refuse dumps in Temboga market in Benin City, Edo State, Nigeria. The seeds were separated from the fruits and dried for 5 days in the sun at ambient temperature for easy dehulling of seeds. The seeds were dehulled after which they were further dried in an electric oven at 60 ^{o}C for 5 hours so as to reduce the moisture content to an appreciable value in order to ease the extraction process. The prepared dehulled seeds were grounded so as to improve the surface area for extraction.

2.2. Extraction of Watermelon Seed Oil

The Soxhlet extraction procedure was adopted for the extraction of the watermelon seed oil and this process was carried out at the National Centre for Energy and Environment, University of Benin. The seeds were blended and weighed and then poured into a tray and the Soxhlet extraction chamber bag was filled with the seeds. The bag was then placed in the extraction chamber with hexane (the solvent used) and the set up was heated using a heating mantle. The apparatus was set up and the heating mantle turned on to begin extraction. Three runs were used per extraction chamber bag filled so as to ensure proper extraction of oil. The residue was discarded each time the required number of runs was completed with the bag refilled with fresh biomass (Ajay et al., 2016).

2.3. Characterisation of Watermelon Seed Oil

The extracted oil was characterised to determine its physico-chemical properties. The properties that were determined include oil yield, peroxide value, acid value, free fatty acid (FFA) value, viscosity, density, saponification value, iodine value and specific gravity according to the methods of AOAC (1990).

2.4. Biodiesel Production

The oil was poured into a glass reactor and then heated to 50 °C. Also, 185 g of methanol was measured into a beaker and 0.5 g NaOH pellets which served as a catalyst was added and it was gently heated in order to dissolve all the sodium hydroxide pellets. The mass of methanol was calculated on a molar ratio basis. The catalyst was allowed to dissolve in the methanol before contact with the oil. The methanol/catalyst mixture was then poured into the reactor and it was immediately sealed to prevent vaporization of the alcohol. The glass reactor was placed on a magnetic stirrer to begin the reaction. Different ratio of oil to methanol (1:3, 1:6, 1:8 and 1:10), temperature (50 °C, 55 °C and 60 °C), catalyst concentration (0.5wt%, 1.5wt%, 2.5wt% and 3.5wt%), stirring speed (250 rpm, 450 rpm, 750 rpm and 1600 rpm) and methanol volume (120 ml, 430 ml and 1196 ml) for a total reaction time of 180 minutes. Biodiesel samples were withdrawn at different intervals of time and they were allowed to separate from glycerol in separating funnels. The yield was measured and recorded. All the biodiesel samples produced were washed with distilled water followed by drying in an evaporator, leaving the clear amber yellow oil which was then characterized (Mazrreku et al., 2016).

2.5. Biodiesel Characterization

The properties of the produced biodiesel were determined using the same procedure as the characterization of the extracted watermelon seed oil. The properties determined include flash point, pour point, cloud point, specific gravity, density, moisture content and sulphated ash.

3. RESULTS AND DISCUSSION

3.1. Characterisation of Watermelon Seed Oil

The watermelon seed oil extracted was characterized to assess its suitability for the production of biodiesel. The results of the characterization of the watermelon oil is presented in Table 1. From Table 1, the yield reported was 49.8%. This yield was close to what was obtained by Adebanjo and Kehinde, (2013) for watermelon seed (41.32%), Jean et al. (2014) for Jathropha seed (48.8%) and Achu et al. (2005) for melon seed oil (44-53%). The peroxide value 7.6 meq/kg reported in Table 1 is moderate and is suitable for biodiesel production. The reason is that peroxide values higher than (9 meq/kg) is an indication of high rancidity which may affect the biodiesel quality to be produced from the oil (Knothe and Dunn, 2001). The peroxide value obtained is slightly lower than the value of 8 meq reported by Musa et al. (2016). The peroxide value is close to the 7.9 meq/kg reported by Mirjana and Milovanovic (2005). The acid value is an important factor in determining the quality of oil to be used for biodiesel. The acid value obtained for the oil was 15.82 mgKOH/g as shown Table 1. The acid value obtained was higher than the acid value 3.91 mgKOH/g reported by Hiba et al. (2015) but was close to 10.4 meq/kg that was reported by Odjobo and Benedict, (2015). Saponification value serves as a measure the degree of unsaturation in a sample of oil. Thus, the higher the saponification value, the more suitable the oil is used for the production of soap rather than for biodiesel production. A saponification value of 189.8 mg/KOH/g was obtained as shown in Table 1.

Table 1: Characterization of the watermelon seed oil

Parameter	Value
Yield (%)	49.80
Specific gravity	0.92
Saponification value (mg/KOH/g oil)	189.80
Peroxide value (meq peroxide/kg oil)	7.60
Iodine value (mg iodine/100g oil)	21.70
Acid value (mg/KOH/g oil)	15.82
Viscosity (mm^2/sec)	28.00

This value is close to that (188 mg/KOH/g) reported by Mirjana and Milovanovic (2005). The kinematic viscosity helps to measure the fluidity of the oil i.e. its resistance to flow. It is also a very important parameter of oils that will be used for biodiesel production. A kinematic viscosity higher than the stipulated limit (35 mm^2/sec) may affect the quality of the biodiesel to be produced from the Watermelon oil. Thus, the kinematic viscosity 2.8 mm^2/sec obtained in this study indicates that the oil is suitable for biodiesel production. The specific gravity of 0.92 was obtained as shown in Table 4.1. Similar values have been reported by other researchers. Rahul et al. (2015) and El-Adawy and Taha (2001) reported specific gravity values of 0.915 and 0.919 respectively for watermelon seed oil. These values are similar to the specific gravity obtained in this study

3.2. Biodiesel Production Profile

The concentration of biodiesel produced was documented as a function of time in the course of the transesterification reaction. This was done at different reaction temperatures and the results are shown in Figures 1 to 3 for temperatures of 50, 55 and 60 °C respectively. From these Figures, it was observed that the triglyceride concentration showed a decreasing trend with respect to time. This is an indication of the consumption of the triglyceride in the course of the transesterification reaction. This point was corroborated by a concomitant increase in the concentration of the methyl ester (biodiesel) produced in the course of reaction. The other intermediate reaction products showed their respective trends as shown in the Figures.

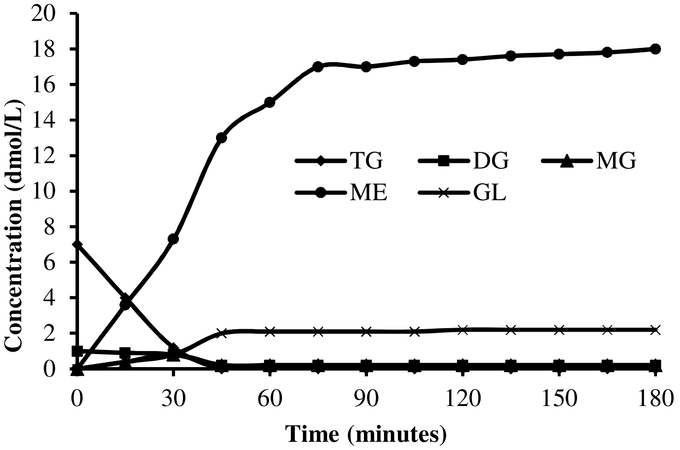

Figure: 1: Concentration of reacting species and products during biodiesel production at 50 °C

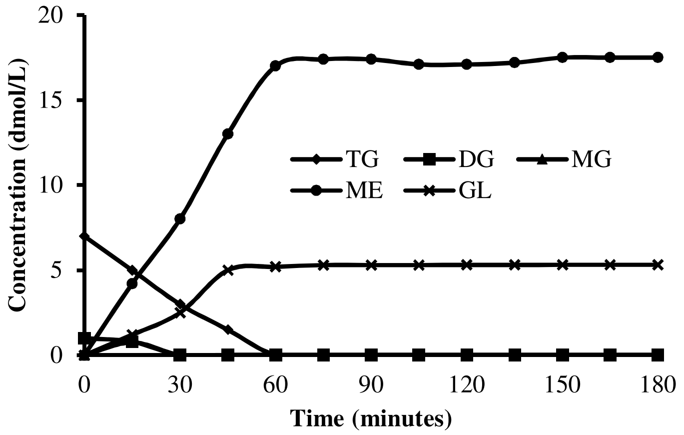

Figure 2: Concentration of reacting species and products during biodiesel production at 55 °C

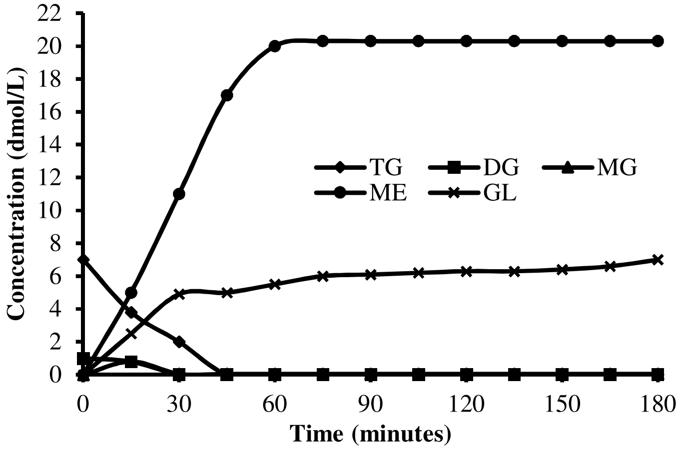

Figure 3: Concentration of reacting species and products during biodiesel production at 60 °C

During the transesterification reaction, as the triglyceride (watermelon seed oil) reacted with the methanol, a reversible reaction occurs with the help of the excess methanol concentration and the NaOH catalyst that was used. From Figure 1, the methyl ester concentration was observed to increase sharply within the first 60 minutes of the reaction to a value of 17 dmol/L. Upon increasing the reaction time to 180 minutes, the amount of methyl ester produced only increased to 18 dmol/L. A similar observation was recorded for Figure 2; however, the maximum methyl ester concentration was 17.5 dmol/L after 180 minutes of reaction. For Figure 3, the methyl ester concentration increased to a peak value of 20 dmol/L after a reaction time of 60 minutes. The value increased to 20.3 dmol/L after 75 minutes and it remained constant till the end of the reaction.

3.3. Effect of Process Variables

3.3.1. Effect of temperature

Temperature affects the kinetic energy of the molecules of the reactants during the transesterification process. Thus, the effect of temperature on the yield of the biodiesel produced was analyzed and the result is as presented in Figure 4.

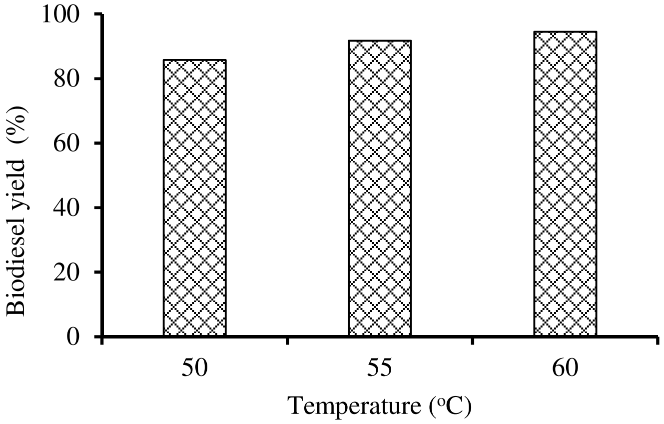

Figure 4: Variation of biodiesel yield with temperature

Figure 4 shows the biodiesel yield obtained at different temperatures (50 °C, 55 °C and 60 °C). Biodiesel yields of 86%, 92% and 95% were observed at temperatures of 50 °C, 55 °C and 60 °C respectively. From the Figure, it can be observed that biodiesel yield increased with temperature. A great caution was taken when choosing the temperature. The experiment was performed by ensuring that the temperature did not exceed 65 °C. The reason for this is due to the fact that methanol boils at 65 °C, thus, if the experiment was performed at a temperature that exceeds its boiling point, the entire methanol may vaporize in the course of the experiment. Observations similar to those presented here were reported by Lotero et al. (2005) and Abbah et al. (2016).

3.3.2. Effect of oil to methanol ratio

The yield of biodiesel is affected by the methanol to oil ratio. The concept of methanol to oil ratio of biodiesel stems from the stoichiometry of the reaction between triglyceride and alcohol. The yield variation with respect to oil to methanol ratio is shown in Figure 5.

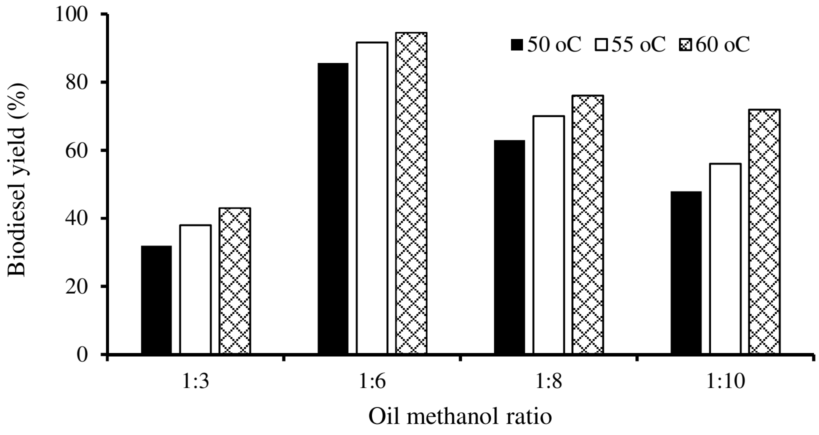

Figure 5: Effect of oil to methanol ratio on biodiesel yield

From this Figure, as the oil to methanol ratio increases, the yield also increases. Maximum biodiesel yield was obtained at an oil to methanol ratio of 1:6. This could be attributed to the fact that the methanolysis of watermelon seed oil requires a higher oil to methanol ratio and this is very important in order to drive the reaction to completion. Increasing the oil to methanol ratio beyond 1:6 does not favour a higher conversion of the watermelon oil to biodiesel. Observation similar to those presented here was reported by Okullo and Temu (2015) and Mu'azu et al. (2015).

3.3.3. Effect of catalyst concentration

Catalyst is required to decrease the activation energy of the reaction but the amount of catalyst to be used also has an effect on the yield of biodiesel. The effect of a catalyst concentration on the biodiesel yield is presented in Figure 6. From this Figure, the respective trend for the biodiesel yield showed that increasing the catalyst concentration increases the yield of the produced biodiesel up to a maximum for a catalyst concentration of 1.5wt% for all the temperatures tested. Increasing the catalyst concentration beyond 1.5wt% did not favour the process as seen in the downward trend observed with respect to biodiesel yield. The reason for this may be due to the formation of soap which lowers the yield of the produced biodiesel (Ebiega and Garba, 2011).

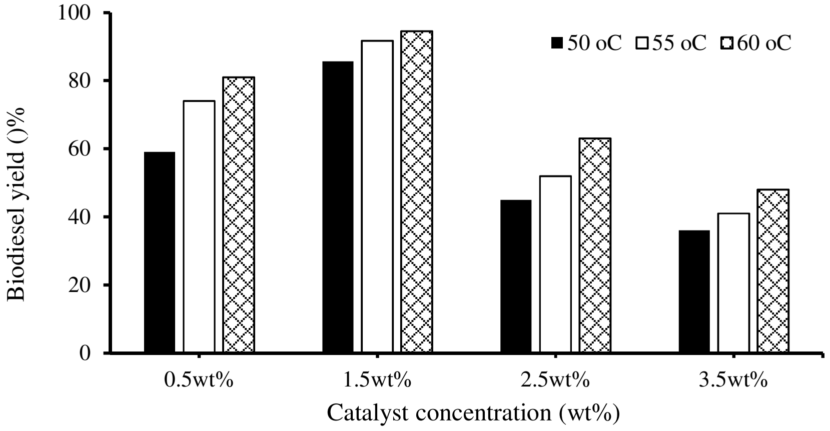

Figure 6: Effect of catalyst concentration on biodiesel yield

3.3.4. Effect of stirring speed

Proper mixing of the watermelon oil with the methoxide solution (methanol and NaOH) is needed to facilitate the attainment of equilibrium conversion. Different stirring speeds were investigated and the results are presented in Figure 7. Biodiesel yield was observed to increase with increase in stirring speed. Lower stirring speeds produced lower biodiesel yields and vice versa for all the temperatures tested. This observation could be attributed to the fact that at the beginning of the reaction, mass transfer controls the reaction due to the poor mixing between the oil and methanol. Thus, a high stirring speed is needed to activate the chemical control phase in order to facilitate the conversion of the reactants to products (Aworanti et al., 2013). Maximum biodiesel yield was obtained at a stirring speed of 750 rpm. Thus, the methanolysis of watermelon oil using sodium hydroxide catalyst does not require a stirring speed as high 1600 rpm to enable it achieve the desired results and this is due to the homogeneous nature of the reaction.

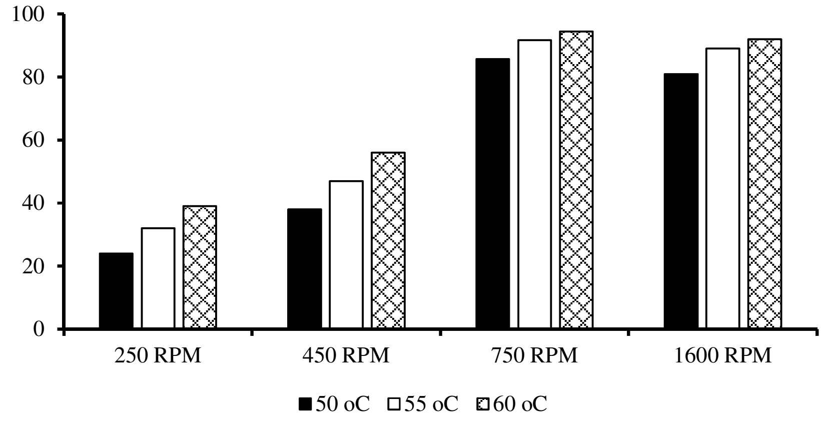

Figure 7: Effect of stirring speed on the biodiesel yield

3.4. Characterization of Produced Biodiesel

A characterization of the produced biodiesel was performed in order to ascertain its quality. Analysis of the characterization results is presented in Table 2. Cloud point measures the lowest temperature for biodiesel utilisation. When the cloud point is below the stipulated limit, biodiesel tend to solidify and form crystals which restrict free movement of engine parts (Alamu et al., 2008). The cloud point -15 °C obtained in this study is within the cloud point (-15 °C to 10 °C) of international standard but it was lower than the cloud point (-3 °C) reported by Ogunwole (2015) and the reason for this difference may be due to the existence of the different fatty acid profile which tend to give different cloud points. In addition, cloud point varies with the types of feedstock, ester composition and the presence of additives (Demirbas, 2008). High cloud point means bad flow cold flow property.

Table 2: Characterized biodiesel properties

Properties	This study	DPR/API Acceptable range/Standard
Flash point (°C)	52	52-96
Pour point (°C)	-18	<0
Kinematic viscosity @40 °C (m^2/s)	5.79	5.5-24.0
Cloud point (°C)	-15	NS
Specific gravity @60/60°F	0.85	NA
Density @60/60°F, (g/ml)	0.92	0.81-0.93
Water content, (%)	<0.01	0.05 max.
Sulphated ash (%)	<0.001	0.02 max.

Kinematic viscosity is a useful parameter that measures the flow resistance of the biodiesel. The viscosity as reported in Table 2 is 5.79 mm^2/s and it is slightly higher than the viscosity 4.84 mm^2/s reported by Alamu et al. (2008). The reason for the difference in viscosity value is because the longer the length of the fatty acid with the saturated methyl esters, the higher the kinematic viscosity (Musa et al., 2016). A very high kinematic will restrain the movement of biodiesel in engine parts causing clogging that will greatly affect the performance of engines. The kinematic viscosity obtained in this study is within the international accepted limit. Flash point helps to determine the lowest temperature at which ignition is produced at atmospheric pressure. The flash point shown in Table 2 is 52 °C and this is within the international limit (52 °C-96 °C).

The flash point is taken into consideration as part of the storage condition of biodiesel because flash point monitors the safe handling and storage of the biodiesel (Canakci and Sanli, 2008). Sulphated ash helps to check the mineral ash residue when the biodiesel burns. Thus, very high sulphated ash content is undesirable for the efficient functioning of engine parts. The sulphated ash recorded in Table 2 is suitable for the biodiesel to be used in engines. The sulphated ash is level was within acceptable standards. Pour point is the temperature at which the biodiesel changes into a semi-solid, thereby losing its flow. The pour point -18 ^{o}C is lower than the pour point -4 ^{o}C obtained by Ogunwole (2015). The pour point obtained in this study is good enough for the biodiesel to be used in colder regions whose temperatures do not go beyond the pour point of the biodiesel. The density as reported in Table 2 is 0.92 g/ml and it is slightly higher than the density value 0.8 g/ml reported by Ogunwole (2015). The density reported in Table 2 is within the acceptable limit (0.81 g/ml -0.93 g/ml). The specific gravity value 0.85 as shown in Table 2 is similar to the specific gravity (0.89) reported by Musa et al. (2016).

4. CONCLUSION

Watermelon seed oil has been shown to be a suitable feedstock for the production of biodiesel. Temperature plays a vital role in the transesterification process as the yield of biodiesel was seen to be significantly influenced by the reaction temperature. A high temperature favoured the biodiesel production process. The transesterification process produces intermediate products such as monoglyceride and diglyceride and the reaction require excess alcohol (preferably, 1:6 with respect to the oil) for the reaction to reach completion. The biodiesel produced has properties comparable to petroleum-based diesel as seen in the similarity in the ASTM standard. Since this study has shown that process variables affect methyl ester biodiesel yield, a complete understanding has been provided and a knowledge gap has been bridged.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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