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Production of Biodiesel from Desert Date Seed Oil

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Abstract : In this work, the oil of desert date (*Balanite aegyptiaca*)seed has been extracted using particle sizes of 0.6mm and 1.8 mm via solvent extraction. The chemical and physical properties of the oil were determined to ascertain its suitability for use in biodiesel production. The results of the analyses of the oil that were obtained to be saponification value, peroxide value, acid value, free fatty acid, specific gravity and moisture contentof 172.5 meqKOH/g, 9.37meq/kg, 0.9256mg/g, 0.47meq/g, 0.9199 and 9.8%, respectivelyhave shown that itis a good raw material for biodiesel production. The production of biodiesel was carried out using the extracted oil through transesterification process by varying catalyst concentration, oil to methanol molar ratio, reaction time and reaction temperature and analysed, and the analysis of the produced biodiesel showed that its cloud point, pour point, flash point and viscosity were 8 ^{o}C, -13^{o}C, 170 ^{o}C and5.2 respectively. The results of the analyses of the produced biodiesel, which were found to compare very well with the standard values, indicated that the liquid produced using the extracted desert date seed oil was biodiesel with a yield of 82%.
Keywords: Biodiesel, date seed oil, solvent oil extraction, transesterification.

1.0 Introduction

As the world population increases, the energy consumption also increases. In any nation, energy is the mostfundamental requirement for human existence and activities [1]. Unfortunately, the non-renewable energy sources that contribute over 86% of the global energy supply are depleting[2].In addition, apart from price hike in the depleting petroleum based products,greenhouse gas emission being emitted as a result of the products contributes significantly to climate change and ozone depletion. This problem has resultedto intense search for alternative feedstock and sustainable technology that can counter the shortcomings of non-renewable energy sources. One of the alternative energy types considered to replace the dwindling conventionaltransportation fuel is biodiesel[3].

Biodiesel, as an alternative fuel, has been currently receiving much attention owing to the limited availability of conventional petroleum diesel and environmental concerns. It can be directly used to replace petroleum diesel without modifying diesel engines since their properties, e.g., specific gravity, cetane number, viscosity, cloud point, and flash point, are similar ([4],[5],[6],[7],[8]). It is a promising alternative or extender to conventional petroleum based diesel fuel. Furthermore, it has a number of advantages such as reducing carbon dioxide emissions by about 78%, nontoxicity and biodegradability. These its benefits have made the fuel a very good environmentally benign one. Furthermore, biodiesel is a renewable energy source [9, 29-37] that has

superior properties than that of petro-diesel fuel ([10], [11]) such as nontoxicity [12]. The research involving the production of fatty acid methyl esters are being embarked on nowadays because it is very important for today's world to identify an alternative to fossil fuel to meet the future demands for energy ([13], [14], [15]), based on the fact that diesel fossil fuel reserves dwindling and at a time will run out [16], especially for use in internal combustion engines, which reduce the peak flame temperature and thereby reduction in various emissions [17], as it (biodiesel) is an alternative fuel that can be prepared from renewable biological sources such as vegetable oils both (edible and nonedible oil) and animal fats ([18], [19]). In other words, biodiesel can be derived from vegetable oil, which is a renewable domestic resource ([20], [21], [5], [6], [7]).

Vegetable oils that have been intensively studied as raw materials for biodieselproduction include soybean oil, palm oil, castor oil, *Parkia biglobbossa* oil, *Jatropha curcas* oil,sunflower oil, coconut oil, rapeseed oil, safflower oil, ground nut oil, neem oil, pea nut oiland cotton seed oil([22],[23],[24],[25],[26]). Other potential vegetable feedstock for biodiesel production includes tobacco (*Nicotianatabacum*), desert date (*Balanites aegyptiaca*), castanhola (*Terminalia catappa*), rubber tree (*Hevea brasiliensis*), tung(*Vernicia fordii*), milkweed (*Asclepias syriaca*), *Zanthoxylum bungeanum*, radish (*Raphanus sativus*), Ethiopian orAbyssinian mustard (*Brassica carinata*), false flax or gold-of-pleasure (*Camelina sativa*), Polanga (*Calophyllum inophyllum*), cardoon (*Cynara cardunculus*), sesame (*Sesamum indicum*), marula (*Sclerocara birrea*), pumpkin(*Cucurbita pepo*), jojoba (*Simmondsia chinensis*) [3]. Among all the mentioned feedstocks for biodiesel production, a very promising one is the desert date tree (*Balaniteaegyptiaca*).

Desert date tree (*Balanite aegyptiaca*) is a deciduous tree which grows naturally in the wild, especially in the Sahel belt of Africa. It can be found in many kinds of habitats, tolerating a wide range of soil types, from sand to heavy clay and climatic moisture levels.The tree is remarkable because it is available during the dry season, when foliage is difficult to obtain and prices of conventional feedstuff are more on the rise, and it is found in many kinds of habitats, as it grows in a variety of soil types.Available reports on the nutritional and anti-nutritional profile of *Balaniteaegyptiaca*seed powder show that the seed powder contains a relatively high amount of protein and lipid. However, in addition to the nutrients, the seed contains high level of anti-nutritional factors; tannins, oxalate and phytic acid[27].Hence, *Balaniteaegyptiaca* seeds as non-edible plant oil is a good source that can be used for biodiesel production.

Therefore, this workis aimed at extracting oil from desert date seed (*Balaniteaegyptiaca*) and producing biodiesel from the extracted oil via transesterification process. In the course of the research, some of the factors (molar ratio, catalyst concentration, reaction time and temperature) affecting biodiesel production were varied and, thereafter, the product obtained was characterized.

2.0 Methodology

2.1 Sample Collection and Preparation

The desert date fruits(Figure 1) used in this work were obtained from Muda Lawal Market located in Bauchi, Bauchi State of Nigeria. After the fruits were obtained, they were screened to remove the defective ones and soaked in a large bowl of water overnight to remove the glycoside pulp from seed coat. The washed seeds (Figure 2) were sundried and their shells were cracked using metal hammer to obtain its kernel. The kernels of the dried seeds were ground using mortar and pestle and, then, separated with the aid of a sieve shaker into two types of particle size, viz. 0.6mm and 1.8 mm.

Figure 1. Raw desert date fruits

Figure 2.Washed desert date seeds

2.2 Oil Extraction

In order to get oil from the prepared seeds of the date fruits, 10g of the ground desert date seed (Figure 3) of 0.6mm particle size was carefully poured into a labelled thimble, after which it was placed in the extraction chamber of a 250 mL Soxhlet apparatus (Figure 4) fitted with a condenser.

Figure 3. Ground desert date seed kernel

The 500 mL distillation flask of the apparatus was containing 250 mL of n-hexane, which was the solvent for the extraction. Using a temperature of 55 ^{o}C and refluxing for a period of 4 hr, desert date seed oil was extracted. The thimble was carefully removed and the collected n-hexane in the top container of the apparatus was drained into another container for reuse. The oil was then dried using atmospheric air so that it would be totally free of n-hexane. The same procedure was repeated for the particle size of 1.8 mm at the same temperature but for 7 hr. The yield of oil extracted was calculated using Equation (1).

$$\%Yield = \frac{Weight\ of\ oil\ extracted}{Weight\ of\ seed\ used} 100\% \quad (1)$$

Figure 4. Experimental set-up for oil extraction

2.3Characterization of the Extracted DesertDate Seed Oil

Colour

The colour of the extracted seed oil was observed visually.

Moisture content

The moisture content of the extracted oil was determined by heating 5 g of the sample placed in a clean dish, which had been previously oven dried, at 105 ^{o}C for about 24 hr in a thermosetting oven. The weights of the dish with its content were measured before and after heating and recorded. The percentage moisture content of the oil was calculated using Equation (2).

$$\%\ Moisture\ content = \frac{Loss\ in\ weight}{Weight\ of\ sample\ before\ drying} 100\% \quad (2)$$

Viscosity

The viscosity of the oil produced was measured using a digital viscometer. To carry out the viscosity measurement, 12mL of desert date seed oil was poured into a 50mL beaker, and a spindle with a number of 7 was immersed into the oil until the level got to the mark on the spindle. Thereafter, it was positioned such that it would not touch the wall of the beaker.The value of the viscosity of the biodiesel was then read from the viscometer and recorded.

Specific gravity

An improvised specific gravity bottle was washed and rinsed with acetone and dried. The bottle was filled with water and its weight, together with the content,was taken. The water was then poured out and the bottle rinsed with acetone and dried. Theoil was then poured into it and the weight of the content in addition to the bottle was taken. The specific gravity of the oil was thus calculated usingEquation (3).

$$Specific\ gravity = \frac{Weight\ of\ oil}{Weight\ of\ equal\ volume\ of\ water} 100\% \quad (3)$$

Saponification value

The method used for the determination of the saponification value was that of British standard institute.For this, 2.0g of the oil was placed in a 250mL conical flask and 25mL of 0.5 Methanolic potassium hydroxide solution was added. A reflux condenser was attached and the flask was refluxed for 30 min on a water bath with continuous swirling until it simmered. The excess potassium hydroxide was titrated against 0.5 M hydrochloric acid using phenolphthalein indicator while still hot. A blank determination was also carried out under the same conditions. The saponification value was calculated using the expression given in Equation (4).

$$Saponification\ value = \frac{(B-R)\times 28.05}{Weight\ of\ oil} \quad (4)$$

Iodine value

In order to determine the iodine value of the extracted desert seed oil,0.1 g of the oil sample was placed in a 250mL conical flask. 1 mL of anhydrous chloroform was added to the flask, followed by 3mL of Hanus solution and flask stopper, andall the components of the contentweremixed and placed in a drawer for exactly 30 min.1 mL of 15% w/v potassium iodide solution was added to the flask to wash down any iodide that may be found on the stopper. The content was then titrated against $0.1NNa_2S_2O_3$ until the solution became light yellow. 2 drops of starch indicator was added and titration continued until the blue colour disappeared. The titre value was recorded. The iodide value was thus calculated using Equation (5).

$$Iodine\ value = \frac{(B-R)\times Normality\ of\ Na_2S_2O_3 \times 12.69}{Weight\ of\ sample} \quad (5)$$

Free fatty acid (FFA)

The free fatty acid content of the oil was measured according to British Standard Institute Method.In this method, 1g of the oil sample was placed in a 250mL conical flask and warmed. 2.5 mL of methanol was added with thorough stirring, followed by two (2) drops of phenolphthalein indicator and a drop of 0.14Npotassium hydroxide solution. The contentcontaining the oil sample wasthen titrated against 0.14N potassium hydroxide solution while shaking vigorously until a permanent light pink colour, which persisted for 1 min, was observed. The end point was recorded, and the FFA value was calculated through the use of Equation (6).

$$\%FFA = \frac{Titre \times N \times 28.2}{Weight\ of\ sample} \quad (6)$$

Acid value

The acid value of the oil was calculated using the already calculated free fatty acid value from the expression given in Equation (7).

$$Acid\ value = \%FFA \times 1.99 \quad (7)$$

Peroxide value

1g of the oil was placed in a 250mL conical flask and 3mL glacial acetic acid/chloroform (3:2v/v) was added. The contents were shaken until everything became homogeneous. After that, 1 mL of saturated potassium iodide solution was added followed by the addition of 0.5mL starch indicator solutionunto the mixture that was then titrated against 0.1 N $Na_2S_2O_3$ until the dark blue colour formed disappeared. Blank determination was also carried out without adding oil. Theperoxide value was calculated using Equation (8).

$$Peroxide\ value = \frac{(S - B) \times Normality\ of\ Na_2S_2O_3}{Weight\ of\ sample} \quad (8)$$

2.4 Transesterification Reaction of the Oil

The transesterification reaction used to convert the extracted oil into biodiesel was carried out in a 250 mL round bottom flask,as a reactor, fitted with a magnetic stirrer. The experimentswereperformed by varying the temperature between 45 and 75^{o}C and varying the catalyst load between 0.3 and 0.5% by weight in order to ascertain their effects on the yield of biodiesel. The progress of the reaction was monitored by withdrawing some samples from the reactor at interval of 30, 45, 60 and75 min.

After each of the experiments, the mixture containing the biodiesel was poured into a separating funnel and allowed to settle for 24 hr leaving the biodiesel at the top while the unreacted methanol and glycerol wereat the bottom of the funnel. The mixture of glycerol and methanol was drained out leaving the biodiesel in the separating funnel to be washed.

In order to wash the biodiesel, water of volume twice that of the biodiesel was measured, poured into a separating funnel and sealed with a tape.The separating funnel was then rolled gently on a table to and fro until the mixture appeared homogeneous. However, care was taken to avoid formation of soap. After that, the separating funnel was restored to its previous position and allowed to stand for an hour until a clear separation of the two liquids was visible. Upon seeing the clear separation between the liquids, the seal was carefully removed from the cover and the waterwas carefully drained out leaving only the biodiesel in the separating funnel.

The yield of the biodiesel obtained from the oil was calculated using the Equation (9).

$$\%Biodiesel\ yield = \frac{Weight\ of\ oil\ biodiesel}{Weight\ of\ oil\ sample\ used} 100\% \quad (9)$$

The biodiesel thus obtained was analysed to investigate its properties so as to be sure that the produced material would be able to serve the purpose it was meant for.

2.5 Determination of Biodiesel Properties

Viscosity

The viscosity of the produced biodiesel was determined using the method described for the viscosity determination of the extracted oil.

Flash point

An improvised method was used for the determination of the flash point of the produced biodiesel. 150mL conical flask was filled to a 6 mL level with biodiesel and was heated at slow constant rate on a hot plate. The lowest temperaturewhen the application of a test flame caused the vapour above the biodiesel sample to be ignited was then taken as the flash point.

Pour point

To determine the pour point of the biodiesel, a cylindrical test tube was filled with the biodiesel to a 5 mL level and held with a wooden clamp bearing a thermometer. The sample was then allowed to cool below 0 ^{o}C in the ice bath after whichit was removed, tilted on the clamp and observed at some intervals. The lowest temperature at which the oil was observed to flow was recorded as its pour point.

Cloud point

The cloud point of the biodiesel was determined by filling a cylindrical test tube with the biodiesel to a 5 mL level and attaching it to a wooden clamp bearing a thermometer. The test tube was placed in an ice bath and inspected at some intervals for cloud formation. The temperature at which a distant cloudiness appeared at the bottom of the test tube was recorded as the cloud point of the biodiesel.

3.0 Results and Discussion

3.1 Properties of desert date seed oil

Shown in Table 1 are the physicochemical properties of the extracted desert date seed oil obtained after it was analysed. As can be observed from the results given in the table, the seed had a low moisture content, which was an indication that the shelf life of the seed is high because there is little or no water for hydrolysis to take place. The average oil content obtained from the desert date seed of 0.6mm was found to be higher than that of 1.8 mm; meaning that the small the particle size the higher the yield of oil obtainable from that seed.

Table 1. Physicochemical properties of desert date seed oil

Property	**Value**
Colour	Pale yellow
Odour	Pleasant
Density (g/cm^3)	0.77
Specific gravity	0.9199
Saponification value (%)	172.5
Peroxide value	9.37
Acid value	0.926
Free fatty acid	0.47
Moisture content (%)	9.8
Oil content for 0.6 (%)	45.2
Oil content for 1.8 mm (%)	41.5
Texture	Viscous

The peroxide value obtained for the oil also revealed that the oil was a non-drying type with a very low degree of unsaturation. Furthermore, it was discovered from the saponification value of the oil that it would be suitable for use in biodiesel production. The acid value is the parameter used to quantify the amount of acid present in a chemical substance, and it should not be more than 1.5 mg/g so as to avoid corrosion of automotive parts. Based on the results obtained, the acid value of the desert date seed oil was within the recommended range for oil to be used for biodiesel production. Moreover, the specific gravity of the extracted oil was estimated to have a value that compared very well with that of petro-diesel. Apart from that, the density of the

oil, given in Table 1,had a value that was also obtained to be close to that of petrol-diesel, which is 0.9 g/m^3. All these facts were found to be indications that the produced biodiesel would be a very good alternative to petro-diesel.

3.2 Effects of Operating Variables on the Transesterification Process

After extracting and characterizing the oil from the desert date seeds, the transesterification process for the production of biodiesel was carried by varying some operating parameters. The parameters varied for the process were molar ratio of oil to alcohol, catalyst concentration and reaction temperature. In addition, while those parameters were varied, the reaction time was kept constant at a particular time. However, four different reaction times were considered for the parameters and the process. Given in Table 2 are the variations applied to each of the parameters. As can be seen from the table, the yield of the biodiesel obtained from each of the reaction times used with the variations in the other parameters are given and observed to vary as the parameters were varied.

Table 2. Results of biodiesel yield with time varying some parameters

Run	Catalyst concentration (wt %)	Molar ratio	Temperature (^{o}C)	Biodiesel yield with			
				30 min	45 min	60 min	75 min
1	0.3	1:3	45	40	45	56	61
2	0.3	1:3	55	75	83	85	91
3	0.3	1:3	65	74	76	80	81
4	0.3	1:3	75	72	75	77	79
5	0.3	1:4	45	80	83	85	89
6	0.3	1:4	55	82	85	86	88
7	0.3	1:4	65	77	78	82	85
8	0.3	1:4	75	69	71	73	74
9	0.3	1:5	45	30	33	38	41
10	0.3	1:5	55	33	39	43	46
11	0.3	1:5	65	35	38	42	45
12	0.3	1:5	75	20	26	28	31
13	0.5	1:3	45	40	45	49	53
14	0.5	1:3	55	38	42	47	51
15	0.5	1:3	65	38	41	45	49
16	0.5	1:3	75	32	38	43	47
17	0.5	1:4	45	25	27	31	34
18	0.5	1:4	55	28	32	35	37
19	0.5	1:4	65	30	32	36	34
20	0.5	1:4	75	29	30	32	33
21	0.5	1:5	45	20	25	29	34
22	0.5	1:5	55	23	27	32	36
23	0.5	1:5	65	22	26	29	31
24	0.5	1:5	75	19	23	27	29

In order to clearly see how the yield of biodiesel was changing for different reaction times and runs (with the other different operating parameters), plots of the yields were developed as shown in Figure 5. According to the figure, the nature of the variations noticed from the plots of the yields of the biodiesel obtained using different reaction times were similar, but with different values, to one another except in few cases.

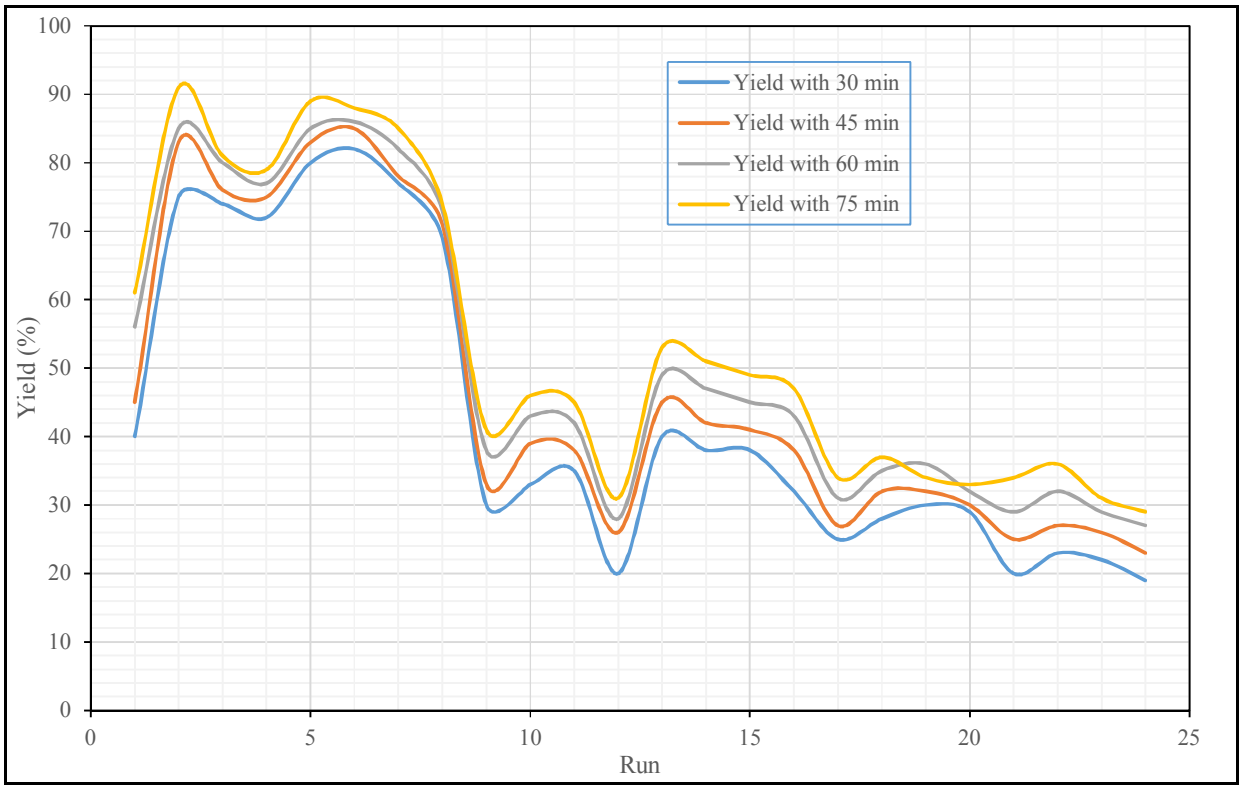

Figure 5. The yield of biodiesel obtained from the different runs at some reaction times

Table 3. Properties of the desert date biodiesel in comparison with some ASTM standards

Parameter	DD Biodiesel	ASTM D6751[28]	ASTM D975[28]
Viscosity @ 40 ^{o}C, mm^2 /s	5.2	1.9-6.0	1.9-4.1
Flash point (^{o}C)	170	130 (min)	60-80
Pour Point (^{o}C)	-13	(-15) - 10	(-35) - 15
Cloud point (^{o}C)	8	-	9

Table 3 shows the properties of the produced biodiesel, and it was deduced from the results that the low pour point (-13 ^{o}C) of the biodiesel could make it usable for engines at cold regions. Besides, the other parameters determined for the biodiesel were found to compare well with the two standards (ASTM D6751 and ASTM D975) considered in this work.

4.0 Conclusion

The results obtained from the preliminary investigation carried out in this work revealed that desert date seed oil was an economically viable oil source because its oil content was found to be high. Also, the oil parameters showed that the oil was composed of moderately long chain fatty acids with a degree of unsaturation,making it a good feedstock for biodiesel production. From the resultsobtained for the flash point test of the produced biodiesel, it was shown that the produced biodiesel hadsome advantages over petrol-diesel because it gave a relative decrease in the emission of greenhouse gases that are associated with petrol-diesel. Furthermore, density and viscosity were adopted as measures of the extent of esters conversion, and the values obtained for the biodiesel were found to compare very well with the ASTM standards. It was also observed from the effect of operating parameters on the transesterification reaction of the desert date seed oil studied that keeping the reaction time constant and varying oil to methanol ratio, catalyst concentration and reaction temperature caused variations to occur in the yield of biodiesel obtained from the oil. Therefore, the production of biodiesel using desert date seed oil has been successfully carried out in this work.

Nomenclature

ASTM American Society for Testing and Materials
B titre values of the blank
B Titre value of blank
DD Desert date
N Normality of base
R titre values of the oil
S Titre value of sample

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