Biodiesel Production from Tigernut (*Cyperus esculentus*) Oil and Characterization of its Blend with Petro-diesel

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ABSTRACT (ARIAL, BOLD, 11 FONT, LEFT ALIGNED, CAPS)

This study was carried out to assess the fuel quality of biodiesel produced from tigernut (*Cyperus esculentus*) oil and its blends with petro- diesel. The oil was extracted from the tigernut by solvent extraction method using petroleum ether. The oil was trans-esterified using potassium methoxide at the temperature of 60°C for 60 min at a catalyst concentration of 0.65% and under a constant stirring speed. The crude biodiesel obtained was purified by washing with water and subsequently dried in an oven. The biodiesel was again blended with petro- diesel to obtain various blends of B10, B20, B30 and B40. Oil and biodiesel yields were assessed while physicochemical analysis of the oil, biodiesel and blends were carried out using standard methods for physicochemical parameters including flash point, cloud point and pour point. Results obtained showed that the oil yield from the feedstock was 16%, while the biodiesel yield was 82%. The high and moderate flash points of the biodiesel and blends ranged between 90-178°C, their cloud points ranged between 6.5-13°C while their pour points ranged between -3-(-10)°C. General results of the blends showed that B10 and B20 had performance results closer to petro-diesel and ASTM standards. Therefore, the blends, in addition to being good for biodiesel engines, would also be suited for engines not specifically designed for biodiesel use.

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Keywords: Tigernut, transesterification, biodiesel, biodiesel blends, petro-diesel

1. INTRODUCTION

Currently there is a strong interest in biodiesel, mainly driven by growing volatility in global crude oil markets and concerns over climate change and the desire to address the global

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risk. Biodiesel has proven to be a good substitute for petroleum diesel in motor vehicles and generators, when it meets the international standards such as ASTM for automotive use. Biodiesel is biodegradable, non-toxic and has low emission profiles when compared to fossil fuel and its usage will allow balance between agriculture, economic development and the environment [1]. Biodiesel is produced through a chemical process known as trans-esterification. Transesterification of vegetable oils with low molecular weight simple alcohols (methanol, ethanol, propanol, butanol and amyl alcohol) has been established as the best option to reduce the high viscosity, low volatility, heavy engine deposits and toxic substance formation associated with the direct use of vegetable oils [2, 3]. Tigernut is not really a nut but a small tuber that was discovered some 4000 years ago. It has been cultivated both as livestock feed and for human consumption. It is widely grown in Florida US, Spain, Britain, China, Mali and Ivory Coast. The plant is widely distributed in West Africa where it is cultivated mainly for the edible tubers which it bears underground [4]. In northern Nigeria, the tubers of tigernut can be bought in the market all year round. Table 1 shows the fatty acid composition of tigernut.

Table 1: Fatty acid composition of Tigernut oil

Tigernut oil	**Fatty acid composition (%)**
Oleic	75.72
Linoleic	11.64
Palmitic	10.21
Stearic	1.47
Linolenic	0.64
Arachidic	0.32

Source: Temple [4]

Many research works have explored commercially edible oils like cotton seed oil, sun flower seed oil, soybean oil, peanut oil, coconut oil and palm oil as the feedstock for biodiesel [5, 6], however, availability of these raw materials varies. Although tigernut oil is from an edible feedstock, its use as a potential feedstock for biodiesel production may not likely compete with its use as food since it is not a staple food or widely consumed [7]. Most parts of the tropics are suitable for biofuel crops cultivation including tigernut. This strategy to use crops of relative abundance in a particular region for biofuel production is effectively being employed in USA and Brazil as they are the world largest producers of bioethanol from Sugarcane and other raw materials [8]. Currently, tigernut use in Nigeria is mainly for production of milk juice and as snacks etc. However, the tuber can be used for other numerous purposes aside consumption as food. It has been reported to serve effectively as a supplementary feedstock for biodiesel production [9]. The high fibre content makes it useful for pyrolysis / gasification to biofuels, the moderate starch content also makes it a potential supplementary feedstock for bioethanol production [10]. The wastes emanating from its processing also makes it a veritable feedstock for biogas production under anaerobic digestion [10].

Some studies have been carried out on biodiesel production from tigernut. Barminas *et al*. [11], carried out preliminary studies of transesterification of tigernut *(Cyperus esculentus)* as a source of biofuel. Also, Ugheoke *et al*. [9] studied the optimization of the transesterification process of tigernut oil for biodiesel production, specifically to determine the optimal catalyst concentration level that gives maximum yield of methyl ester (biodiesel) from the oil. Again, Salau *et al.* [12] examined the proximate composition, food functionality and oil characterization of mixed varieties of tigernut rhizome flour and reported oil yields ranging between 25 and 34%.

Most parts of the world use a system known as the 'B' factor to state the amount of biodiesel in any mix [13]. For instance, pure biodiesel is referred to as B100 while B20 is 20% biodiesel and 80% petro-diesel. However, taking U.S as a case study, biodiesel is mostly blended with diesel fuel. Such a blend would have better cold flow properties when compared with neat biodiesel. Consequently, blending biodiesel with petro-diesel may be advantageous for mitigating the poor cold flow properties of biodiesel from many lipid feedstocks. On the other hand, blending at higher ratios may compromise cold flow properties [14]. Again, biodiesel contains no petroleum, but it can be blended with petroleum diesel in any percentage. Biodiesel blends from 2 percent to 20 percent (representing B2 and B20 respectively) can be used in most diesel engines with minor or no modifications. This study therefore aimed to determine the fuel quality of biodiesel produced from methyl esters of tigernut (*Cyperus esculentus*) and its blends with petro-diesel. This was also done in order to determine the most suitable blending ratio for biodiesel produced from tigernut seed oil with petro-diesel and also determine if higher blending ratios differ considerably in quality from lower blending ratios.

2. MATERIALS AND METHODS

2.1. Materials

Fresh tigernut was purchased from a local market in Enugu town of Enugu State, Nigeria. Analytical grade reagents were used for all the analyses carried out without further purification. The petroleum ether was used as procured without further purification. The

Methanol used was a product of Merck, Darmstadt, Germany (99.7% purity), while the potassium hydroxide was a product of Loba Chemie GmbH Switzerland (85% purity). Other materials also used were fractionating column, aluminium foil, 1L beakers, 1.5L biodiesel reactor (fabricated locally), thermo- regulator heater equipped with stirrer (Heizung Chauffage, MGW- LAUDA, D6970, Lauda- Königshofen, Germany), electronic digital weighing balance (Ohaus, Adventurer, model- AR 3130), specific gravity bottle, pH meter (Hanna pH meter model No. 02895), Rotary evaporator, oven (BTOV 1423), Vecstar furnace LF3, Ferranti portable viscometer model VL, Abbe refractometer, semi automatic Cleveland flash point tester and Hewlett Adiabatic Bomb Calorimeter model 1242. The study was carried out in the National Centre for Energy Research and Development, University of Nigeria Nsukka in August, 2010.

2.2. Oil extraction

The tigernut was washed with water to remove sand particles adherent in the tuber and then allowed to dry. The seeds were ground into coarse particle sizes with a mechanical grinder (local mill) and placed in a solar dryer for four days to remove residual moisture. The dried meal was packed in a big fractionating column up to three quarter level and petroleum ether was poured well above the level of the meal in the column. It was closed with aluminium foil and masking tape and then left for a period of 8 h. The mixture of oil and solvent was collected from the bottom of the column with a beaker. This was repeated to extract more the oil from the meal. The oil was recovered using rotary evaporator to distil off the solvent. After the distillation, the oil was left in the open to totally dry up completely.

2.3. Characterization of oil

The oil was characterized for pH using a pH meter, specific gravity using a specific gravity bottle, moisture content by the oven dry method, ash content by heating to dryness in furnace, kinematic viscosity using a viscometer, the acid value, saponification value, Iodine value and Peroxide value by titrimetry, refractive index using Abbe refractometer [15] and percentage free fatty acid (% FFA, as oleic) was determined by multiplying the acid value with the factor 0.503. Thus % FFA = 0.503 x acid value [16].

2.4. Physicochemical analyses

Ash, moisture, fibre and calorific values were determined using AOAC method [17]. Fat, crude nitrogen and protein contents were determined using Soxhlet extraction and micro-Kjedhal methods described in Pearson [18]. Carbohydrate content was determined by difference [19].

2.5. Preparation of Potassium Methoxide

250 ml of methanol was measured into a 500 ml flat bottom flask and covered immediately. 5.8 g of potassium hydroxide was carefully added into the methanol to make a solution which was made airtight. It was shaken and swirled for a few times until the potassium hydroxide was completely dissolved. This gave a catalyst concentration of 0.65%.

2.6. Biodiesel production and purification

The transesterification reaction was carried out in a 1.5L airtight biodiesel reactor vessel fitted with thermo-regulator heater/ stirrer. One litre of tigernut oil was measured into the flask and was heated to a temperature of 60^{o}C. The potassium methoxide was then poured into the flask containing the oil and was immediately covered. The temperature of the system was maintained at 55-60^{o}C for the one hour duration of the reaction. At the end of the reaction, the mixture was transferred into a separatory flask, left for 24 h and then the biodiesel separated from the glycerol by gravity. The biodiesel was purified by washing with water five times to obtain a clear water and neutral pH [20, 21]. The glycerol was not refined further but was kept for other uses.

2.7. Characterization of biodiesel and its blend

The different blends; B10, B20, B30 and B40 were prepared by mixing 10, 20, 30 and 40 ml biodiesel and 90, 80,70,60 ml petro-diesel respectively. They were analyzed in the same way as the tigernut oil for the same parameters and also calorific value using a Bomb calorimeter, flash point using a semi automatic Cleveland flash point tester via the American system of testing materials (ASTM D93), cloud point (ASTM D2500) and pour point (ASTM D97) methods.

3. RESULTS AND DISCUSSION

Table 2 shows the proximate composition of the tigernut tuber and starch after de-oiling of the feedstock, while Table 3 shows the physic-chemical properties of the tigernut oil. The calorific value, carbohydrate content and fat content of the tuber indicate that the feedstock would serve as a good source for biofuels production since the inherent energy content would increase the burning power.

Table 2: Proximate composition of tigernut tuber and starch

Parameters	Tigernut tuber	Tigernut starch
Oil yield (%)	15.91	n/a
Moisture (%)	5.77	8.13
Ash (%)	1.86	6.14
Crude fibre (%)	9.50	7.98
Crude fat (%)	25.70	2.33
Crude nitrogen	1.12	0.52
Crude protein (%)	7.00	3.24
Carbohydrate (%)	65.50	65.18
Calorific value(kcal/g)	524.60	345.88

As shown in Table 2, the oil yield from the tigernut was approximately 16% which is quite low when compared with most oil seed feedstock (Peanut- 50%, Sesame seed- 50%, olive seed- 40% Almond- 50%, castor seed- 50% sunflower seed- 35% etc.) [22]. However, by- product of the tuber after extraction can be put to good use as feedstock for other biofuels production/application. The odour and clear golden yellow colour of the oil are favourable for aesthetic qualities.

Table 3: Physico-chemical properties of tigernut oil

Parameters	Results
Odour	Odourless
Colour	Yellow
Specific gravity	0.91
pH	5.30
Moisture (%)	5.32
Ash content (%)	2.60
Iodine value (g/100g)	143.37
Acid value (mg/KOHg^{-1})	8.97
Saponification value (mg/KOHg^{-1})	161.54
Peroxide value (mEq/Kg)	8.33
Free fatty acid (%)	4.49
Viscosity (mm^2/s)	0.98
Refractive index	0.77

Acid values of the oil and biodiesel were very high and exceeded the ASTM standard of 0.8 mg/KOHg^{-1} (Tables 3 and 4). Vegetable oils containing high free fatty acids have significant effects on the transesterification with methanol using alkaline catalyst. It also interferes with the separation of fatty acid ester and glycerols [23]. This also may have affected the yield of biodiesel which was lower than most high yielding ones (95% and above). For instance, Highina *et al.*, [24] reported 97% biodiesel yield from Jatropha curcas while Meka *et al.* [25] reported 96.85 biodiesel yield from Safflower. Even though the biodiesel yield was lower than more favourable yield of 95% and above, it was however higher than that obtained for peanut oil reported by Itodo *et al.* [26] (50%) and Ibeto *et al.* [8] (79%). The low pH level observed in the oil and the high free fatty acid content (Table 3) shows that it is acidic. This may be responsible for the poor conversion efficiency of the oil to biodiesel. This also suggests that the oil may require the two stage method of either esterification followed by transesterification or saponification followed by transesterification. This will ensure proper pre-treatment of the oil and increase biodiesel yields [27].

The specific gravities of the B100 and the blends were within the range and compared with that of biodiesel from other oil sources. Densities and other gravities are important parameters for diesel fuel injection systems. The values must be maintained within tolerable limits to allow optimal air to fuel injection systems.

Table 4: Physicochemical properties of petro-diesel, biodiesel and biodiesel blends

Parameters	**Petro diesel**	**B100**	**B10**	**B20**	**B30**	**B40**	**ASTM Std**
Biodiesel yield (%)	-	82					≥ 95
Specific gravity	0.85	0.87	0.86	0.86	0.85	0.85	0.875-0.90
Ash content (%)	-	1.13	1.53	1.53	1.20	2.0	0.01 max
Iodine value (g/100g)	-	98.38	90.35	90.35	90.46	90.46	120 max
Acid value (mg/KOHg^{-1})	-	1.122	1.683	1.724	2.805	2.68	0.05 max
Saponification value(mg/KOHg^{-1})	-	108.46	106.74	106.73	104.49	103.37	
Free fatty acid (%)	-	0.56	0.84	0.86	1.40	1.34	
Viscosity (mm^2/s)	5.51	8.08	5.54	5.61	5.84	5.68	1.9-6.0
Refractive index	-	0.77	0.86	0.75	0.77	0.86	
Calorific value (J/g)	22,905	-	15,140.69	26,851.81	32,545.78	19,736.24	
Flash point (^{o}C)	-	178	120	110	92	90	93 min.(US)
Cloud point (^{o}C)	-	13	11	9	6.5	8.0	
Pour point (^{o}C)	-	-3	-7	-9	-10	-9	-10

The ash content of both the B100 and the blends were well above the ASTM standard. This indicates that it may likely have higher mineral contents leading to some level of air pollutants like SO_x and NO_x.

The viscosity of the B100 was slightly higher than the ASTM standard. However, the blends fell within the range required for use in the engines. They also compared well with the petro-diesel whose viscosity was 5.51mm^2/s. However, B10 and B20 had lower values than the other blends. The iodine values were within standard. Higher iodine values indicate high unsaturation in oils and fats. The values obtained in the B100 and blends showed that the unsaturation was taken care of by transesterification. There was no reasonable difference between the values obtained for the different blends. Saponification value is used for checking impurities. The saponification value for the B100 and the blends were lower when compared with that obtained for peanut oil (244.74 and 218.09 mg/KOHg^{-1}) and the Jatropha oil seed which was 193.55mgKOHg^{-1} as reported by Akbar *et al.*, [28]. This indicates that the level of impurities in the tigernut oil biodiesel was very low.

The flash point of the B100 and blends except B30 and B40 were actually within the ASTM 6751 standard specification of 93^{o}C set in order for the biodiesel to be classified as a non-hazardous material for shipping in the United States**.** This indicates that with their use, the fear of fire outbreaks would be eliminated. The value of the flash point for B30 and B40 indicate that they may not be very good blends for use since they did not fall within the ASTM standard range. The cloud point and the pour point of both the B100 and the blends were well within the standard. One of the problems associated with biodiesel is its cold flow properties represented by the pour point. The pour point is the lowest temperature at which frozen oil can flow and is used to specify the cold temperature instability of fuel oil. This shows that biodiesel from tigernut oil would perform very well in very cold and temperate regions. This also indicates that countries in the tropics where cultivation of tigernut thrives, can exploit this outlet into biofuels crop cultivation in order to export the oil to western countries for the purposes of biofuels production. They all also showed good calorific values. This indicates that they would burn with high release of energy.

4. CONCLUSION

The study has shown that tigernut is a good supplementary feedstock for biodiesel production. The results of the blends of the petro-diesel with tigernut biodiesel showed that that B10 and B20 had results closer to B100 and to the ASTM standards and would give better performance than B30 and B40. Therefore, the blends, in addition to being good for biodiesel engines, would also be suited to engines not specifically designed for biodiesel use. This compares very well with biodiesel from other oil sources. Even though tigernut oil is edible oil, the crop is not consumed on a large scale as a staple food and it is not also a major source of edible oil, so it would be advisable if countries in the tropics including Nigeria consider the cultivation of this crop on a large scale. If this is achieved, it would facilitate the use of tigernut for biodiesel production on a large scale and hence alleviate the global concern for food security.

AUTHORS' CONTRIBUTIONS

The first and corresponding author designed the study and wrote the protocol. Author 1 carried out the experimental studies. Author 2' wrote the first draft of the manuscript. 'Author 1' and 2' managed the analyses of the study while Authors 3' and 4' supervised the overall work. All authors approved the final manuscript."

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