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Base Catalyzed Transesterification of Neem (Azadirachta indica) Seed Oil

Atif A. A. Yassin[1], Anas M. M. Mohammed[2] and Maha A. A. Abdelrahman[2]*
1 Faculty of Engineering and Technology, University of Gezira, P.O. Box (20), Medani, Sudan
2 National Oilseed Processing Research Institute (NOPRI), University of Gezira, P.O. Box (20), Medani, Sudan

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ABSTRACT
Base catalyzed transesterification was used to examine biodiesel synthesis for utilizing Neem (*Azadriachta indica*) seed oil. In addition, physicochemical analysis of the Neem seed oil and biodiesel produced were determined mainly by the American Oil Chemist Society (AOCS) and the American Society for Testing and Materials (ASTM). The experiments were carried out by one-step alkali based catalyzed transesterification method, using low (0.73%) free fatty acids (FFA). High biodiesel yield 92.8 % w/w with 99% total conversion was achieved. It was found that the biodiesel specifications such as; kinematic viscosity (5.43cSt), density (0.8877 g/cm^3) and Flash point (166˚C). These fulfil the requirements of ASTM and the committee of standardization in Europe standard specifications (EN). The results found were highly reliable and showed that the Neem seed oil is attractive as feedstock and has potential for biodiesel production. Therefore, the study recommended that the non-edible oils are economically useful for biodiesel production.

KEYWORDS
Neem seed oil, Transesterification, Biodiesel

1. INTRODUCTION

Energy is fundamental and over dependence on fossil fuels, which is a non-renewable energy resource that represents about 80% of the contribution of the exact demand of world energy, which has been growing in the last decades [1]. The world energy utilization rose about 2.9% in 2018 [1,2]. Moreover, [3] reported that the approximate increased in the energy utilization was 28% between the years 2015 to 2040. However, the limitation of non-renewable energy resources and prospect of many universal environmental considerations are responsible of the searching for alternative sources of energy. Fossil fuel combustion was responsible for 98% of carbon emission; in fact the carbon dioxide emission is considered a major factor affecting the green house [4-8].

The most interesting alternative sources among the renewable resources are vegetable oils. The employment of vegetable oils as biodiesel has many advantages. Vegetable oils can be used without modification in the diesel engine. However; the problems that associated with high viscosity and low stability of unsaturated vegetable oils make the use of vegetable oils directly in diesel engine for long time is practically not possible [9,10].

The biodiesel can be utilized as an alternative in diesel engine [11-13]. Biodiesel can be generated from plant oils or animal fats via transesterification [14-17]. Transesterification reaction changes the physicochemical properties of the vegetable oil to be comparable to petroleum diesel [18]. Biodiesel is a reasonable fuel due to its sustainability, non- flammability can be used directly to diesel engines, free of sulphur, reduce the greenhouse effect and biodegradable [19-25].

Biodiesel produced from animal fats and edible vegetable oils compete with the food demand. High cost of the edible oil and the animal fats demonstrate the difficulty for commercialization [8,26,27]. Thus, the biodiesel production utilizing non-edible vegetable oils eliminates food purpose competition and reduces the cost of production [28,29]. Therefore, utilization of non-edible oils feedstock for biodiesel production gained more interest [30,31].

The Neem tree was brought from India to Sudan in 1921. The Neem tree has been grown successfully in all parts of Sudan. The Neem seed oil contents is ranging between 25-45% [32,33]. This work was subjected to investigate the biodiesel synthesis utilizing Neem seed oil.

2. MATERIALS AND METHODS

Preparation of samples

The Neem tree seeds were collected from Wad Madani town. Seeds were inspected and manually cleaned; then dried for 3 days. The oil was extracted by mechanical extraction in the pilot plant at the

National Oilseed Processing Research Institute (NOPRI), University of Gezira, Sudan. Then oil was filtered and dried.

Biodiesel production

The oil (FFA <2) was subjected to biodiesel production using one step base catalyzed transesterification [34,35]. The optimal reaction parameters: NaOH (0.5%); methanol to oil molar ratio (6:1); temperature (65˚C); 600 rpm were used as described by [36], and then the separated funnel was used overnight to separate the glycerol (bottom layer) from the biodiesel (top layer). Then hot distilled water at 60˚C was used to wash the biodiesel product for many times. The moisture was removed by heating at 110˚C.

Oil analysis

The AOCS official methods were used to determine the Neem seed oil physicochemical characteristics: oil content method No. Aa 4-38, Ab 3-49 and Am 2-93, revised (2000); FFA and acid method No. 3d-63 revised (2003); peroxide value method No. Cd 8-53 revised (2003); Iodine value method No. Cd1-25 revised (2003); saponification value method No. Cd 3-25 revised (2003); unsaponifiable matter method No. Ca 6a-40 (1989); moisture content method No. Ba29-38revised (2003); refractive index method No. Cc 7 - 25 (1997) using an Appe 60 refractometer Cruses, Germany.

Biodiesel specification

The ASTM official methods were used to determine the fuel properties: density method No. D4052-96 (2002) using a digital density meter Model DMA 4500M-Alnamsa; kinematic viscosity method No. D445 (2004) using calibrated viscometer Model TV 4000, France at 40˚C; flash point method No. D93 (2002) using semi-auto maticpetro test close-cup model S⁄N 0526054706, Germany; pour point method No. D97 (2004); ash content method No. D482 (2007); water and sediment content method No. D 2709 (2002) using high speed centrifuge.

3. RESULTS AND DISCUSSION

The Neem seed oil contents are shown in Table (1); its oil content is 26%, which is in the range between 25-45% as reported before [33,37,38]. The FFA is 0.73 %, therefore, the base catalyzed transesterification could be employed directly at this low FFA [34,35]. The acid value is 1.45%, the low acid value leads to high yield of biodiesel produced [39].The peroxide value is 2.83 meq/kg. The Iodine value is 96 mg I2/100g oil, the result is in the range of 82 to 98 mg I2/100g as reported before [7,40,41,42,43]. The saponification value is 199 mg KOH/g oil, the result in the range of 191-202 mg KOH/g oil as was reported [7,41,43,44]. Unsaponifiable matter is 1.35%, the result is lower than 1.84 and 3.17% [44,45]. The moisture content is 0.05%. The Refractive index at 25˚C is 1.4637, which is similar to 1.465 as reported by [44].

The density is 0.9279 g/cm3, the result within the range of 0.86 to 0.965 g/cm3 as was being reported [7,39,42,43,46,47,48,49]. The kinematic viscosity at 40ºC is 48.82 cSt, which is closed to 48.32 cSt as reported by [50], the result is lower than 49.79 cSt [44] and high than the range of 20.5 to 48.5 cSt as was found [42,48,49,51]. The Flash point is 284˚C, which is higher than to 260ºC as reported by [52]. The pour point is -15 C˚, which is in the range of -16 to -9C˚ as was reported [41,53,54].

Table (1): The physicochemical properties of the Neem seed oil

Property	Result
Oil content (%)	26
Free Fatty acids (%)	0.73
Acid value (%)	1.45
Peroxide value (meq/kg)	2.83
Iodine Value (mg I2/100g oil)	96
Saponification value (mg KOH/g oil)	199
Unsaponifacable matter (%)	1.35
Moisture content (%)	0.05
Refractive index at 25 oC	1.4637
Density at 15 ◦C (g/cm3)	0.9279
Kinematic viscosity at 40◦C (cSt)	48.82
Flash point (◦C)	284
Pour point (◦C)	-15

Note: Values in the table are the mean values of three replicates

High biodiesel yield of 92% with 99% total conversion was obtained, which indicated that the base catalyzed transesterification is an efficient method for the biodiesel production from the Neem seed oil (FFA<2). Table (2) represents the biodiesel specifications. The density is reduced from to 0.9279 to 0.8877 g/cm3; decreasing of density mean the reaction was completed and the heavy glycerine was removed. The biodiesel density was similar to the range of EN (0.860-0.900 g/cm3), moreover, this result is similar to 0.8785 kg/m3 and 0.888 kg/m3 as reported by [55,56], respectively. The kinematic viscosity at 40˚C was reduced from 48.82 to 5.43 cSt, since too viscous fuel can affect engines injection system, which leads to power loss. This result is within the range of ASTM (1.9 to 6 cSt) and closed to EN range (3.5 to 5 cSt). Moreover, the kinematic viscosity result was also found in the range of 5.53 to 5.81cSt [7,37,57].

Table (2): Biodiesel specification

Property	Results	Standard	
		ASTM D6751	EN 14214
Density at 15 ˚C (g/cm3)	0.8877	-	0.860-0.900
Kinematic viscosity at 40 ˚C (cSt)	5.43	1.9-6.0	3.5-5.0
Flash point (˚C)	166	130	120
Acid value (%)	0.46	0.8	0.5
Ash content (%)	Nill	<0.02	<0.02
Water and Sediment (%)	Nill	<0.05	<0.05

Note: Values in the table are the mean values of three replicates; ASTM=American society for testing and materials standard; EN=Committee of standardization in Europe standard specifications

The flash point is 166 ˚C which is higher than 130˚C for ASTM and 101˚C minimum for EN specifications. The result is closed to 160˚C as was reported by [58], and within 100 to 170oC as was reported [37,59,60]. High flash point is recommended for safe transportations and confirms the absence of risk of fire during handling or storage [44,61].

The acid value decreased from 1.45% to 0.46% which is less than 0.8% (maximum) and 0.5% (maximum) as recommended by ASTM and EN specifications, respectively. The biodiesel produced was free of ash, water and sediment. The low ash content (such as dirt and extraneous solids) prevents lowering the heating value of biodiesel, combustion deposits, injector tip-plugging and injection system wear [62]. Absence of water and sediment indicates cleanliness of biodiesel, the lower water content prevents the formation of soap through the transesterification reaction. On the other hand, presence of water leads to formation of FFAs and glycerols during transesterification reaction due to triglyceride hydrolysis [63]. Soap formations decrease the methyl ester yield [64]. Overall, the biodiesel produced from Neem seed oil complies with the requirements of ASTM (D6751-09) standard and EN (14214) standard specifications.

4. CONCLUSIONS

The Neem seed oil physicochemical properties showed that the Neem seed oil is promising and attractive as a feedstock source used for synthesis of biodiesel. High yield (92%) with 99% total conversion of biodiesel is produced from the Neem seed oil directly by the base catalyzed transesterification due to low FFA (>2). Moreover, the biodiesel produced agrees with the requirements of ASTM and EN biodiesel standard specifications. Therefore, under appropriate conditions, the conversion of renewable Neem seed oil to biodiesel may be an important supplement for future energy.

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