International Journal of Educational Research and Development
www.multidisciplinaryarticle.net
Online ISSN: 2664-7095, Print ISSN: 2664-7087
Received: 27-01-2024, Accepted: 20-02-2024, Published: 15-03-2024
Volume 6, Issue 1, 2024, Page No. 67-69

Preparation and characterization of biodiesel and biodiesel blends from *Citrullus lanatus* seeds oil

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Abstract
Watermelon seeds oil was converted to biodiesel by using a two-step trans-esterification reaction. and blended with diesel fuel in various proportions. The Physical and chemical properties of biodiesel from watermelon seeds were determined according to ASTM D6751. B20 blend of biodiesel with Fossil Diesel and Biodiesel-diesel-ethanol blends were evaluated according to ASTM D7467. The result shows that the products meet the set standard for biodiesel.

Keywords: Watermelon seeds oil, biodiesel, diesel fuel, transesterification

Introduction
The world as a whole depends on petroleum products as its main source of energy. The over-dependence of countries on petroleum products has led to increasing pollution, depletion of the world's petroleum reserves (gasoline and diesel), and rising fossil fuel prices (Palash, *et al*., 2013) [11]. Researchers are trying to find alternative, renewable, and environmentally friendly sources of energy. Biodiesel is one of the renewable energy sources and is used to reduce dependence on fossil fuels and to protect the environment by reducing emissions of toxic gases. It is biodegradable and nontoxic when compared to petroleum-based diesel (Liaquat, *et al*., 2010) [6] biodiesel is considered an alternative fuel for diesel engines that could partially or completely replace or reduce the use of petroleum-based diesel fuel. It is defined as long-chain mono-alkyl esters derived from vegetable oils and animals (Brown, 1999) [1]. Biodiesel has a relatively high flash point, making it less volatile and safer to transport or handle than petroleum diesel fuel. The cost of feedstock for biodiesel production accounts for a large portion of the direct biodiesel production cost. Therefore, one way to reduce biodiesel production costs is to use cheaper fatty acid-containing feedstock such as animal fats, non-edible oils, waste cooking oils, and by-products of vegetable oil refining. Finding cheaper and non-edible oils for biodiesel production is an important goal (Fadhil, *et al*., 2012) [3].

Transesterification is the most popular method of producing biodiesel. This process involves combining triglycerides (found in oils or fats) with alcohol, facilitated by a catalyst. The reaction of alcohol with fatty acids yields a mono-alkyl ester, commonly known as biodiesel, along with glycerol (Ogunwole, 2012) [9].

Oils of higher free fatty acid content (FFA) cannot be transesterified in the presence of an alkaline base catalyst, as this leads to catalyst deactivation and soap formation. Therefore, the FFA content should be reduced. A two-step transesterification, acid-base transesterification (ABTE) was used to reduce the high FFA content of oils or fats. The oil is pretreated with an acid dissolved in methanol, while the second step is a base-catalyzed transesterification, in which the oil reacts with methanol in the presence of an alkali. (Fadhil, *et al*., 2012) [3]

this paper aims to produce biodiesel from watermelon seeds oil, blend it with diesel fuel in various proportions, and analyze the physicochemical properties.

The watermelon is a member of the Cucurbitaceous family, known as the gourd family, mostly grown for its sweet and juicy fruit. Citrullus (Citrus) is one of the most popular species with high water content as high as 92% of the total weight (Ma & Hanna, 1999) [7]. Watermelon seeds oil could serve as a good plant-derived oil alternative for consumption and medicinal purposes. (Maoto, *et al*., 2019) [8]

Methods
1. Biodiesel production
1.1. Oil Treatment
Watermelon seeds oil was acid-treated with 2% sulfuric acid in 0.60 wt./wt. methanol to reduce the high fatty acid composition. The mixture was stirred vigorously with a magnetic stirrer for 2 hours, then poured into a separating funnel and separated for 24 hours. The upper layer of the treated oil with low fatty acid was transferred to a 500 ml beaker (Kombe, *et al*., 2007) [5]

1.2. Alkaline catalyzed transesterification
500 ml of acid-treated oil were added to a 1 L beaker and warmed to 60 °C, and then 100 ml of freshly prepared methanolic sodium hydroxide were added while stirring at 3000 rpm for a duration of two hours. After agitation, the mixture was transferred into a separating funnel and kept for 24 hours. Upon completion of the separation, two distinct layers formed: a darker-colored layer at the bottom (glycerol) and a layer of transesterified oil (biodiesel) at the top. The glycerin in the lower layer was drained, leaving the biodiesel in the upper layer. The biodiesel was further washed with warm distilled water to eliminate traces of soap and other contaminants (Okullo, Temu and Ntalikwa., 2011) [10].

2. Identification of watermelon seeds biodiesel using Gas Chromatography - mass spectrometry (GC-MS)
Shimadzu GC-MS model (QP2010-Ultra) equipped with a capillary column (Rtx-5ms-30m×0.25 mm×0.25μm) was used to be injected in split mode, helium was used as a carrier gas with a flow rate of 1.61 ml/min, the temperature

program was started from 60°C with a rate of 10°C/min to 300°C. The sample was analyzed in scan mode in the range of m/z 40-550, and the total run time was 24 minutes (Sharmila & Rebecca, 2012) [13]

3. Biodiesel blending
3.1. Diesel-Biodiesel blend
Prior to commencing the blending process, the biodiesel must be devoid of water and sediments. To ensure the uniformity of the blends, the biodiesel and fossil diesel were kept at the same temperature. Volumetrically, the B20 blend, comprising 20% biodiesel and 80% fossil diesel, was prepared in 500 mL batches using graduated cylinders of 250 ml and 50 ml. The blending process involved stirring at a speed of 2500 rpm for 30 minutes to thoroughly homogenize the blends (Jamrozik *et al*., 2017) [4].

3.2. Diesel-Biodiesel-Ethanol blend(D60B20E20)
The Diesel-Biodiesel Ethanol blend was prepared using the same procedure as the biodiesel-diesel blend. The resulting blend was then stored in securely closed bottles to prevent ethanol evaporation and maintain its integrity.

4. Physical and Chemical Properties
The physical and chemical properties of both the produced biodiesel and fossil fuel were characterized according to ASTM D6751 standards. Furthermore, the evaluation of B20 and the Diesel-Biodiesel-Ethanol blend was performed using ASTM D7467 specifications.

Results and discussion
1. Fatty acid profile of Watermelon seeds methyl ester
The fatty acid methyl ester profile is one of the key factors determining the suitability of a feedstock for use in biodiesel production (Sokoto, *et al*., 2011) [14] The total ester content in the biodiesel was found 86.02%. The percentage of saturated fatty acids was 17.11%, and the vast majority of free fatty acids were unsaturated at 68.91%, indicating that biodiesel production was successful. Table 1 shows the details of the methyl esters of the free fatty acids present and their productivity. The profile showed that the 9-octadecynoic acid (C18H32O2) was the predominant compound in the mixture having the highest percentage of 67.48%. A higher percentage of unsaturated FFA improves other fuel properties such as cold flow, kinematic viscosity, and performance at low temperatures (Ramos, *et al*., 2009) [12]

Table 1: Ester content of watermelon seeds Biodiesel

Fatty Acid	Formula	Area%	Structure
Palmitoliec acid	C16H30O2	0.12	C16:1
Pentadecanoic acid,	C15H30O2	16.54	C15:0
margaric acid	C17H34O2	0.11	C17:0
9-Octadecynoic acid	C18H32O2	67.48	C18.:1
cis-11-Eicosenoic acid,	C20H38O2	0.23	C20:1
Docosanoic acid	C22H44O2	0.14	C22:0
Tetracosanoic acid	C24H48O2	0.09	C24:0
linolenic acid	C18H30O2	0.08	C18:3

2. Physical and chemical properties of watermelon seeds biodiesel
Table 2. shows physical and chemical properties of Biodiesel from watermelon seeds oil (B100) and fossil diesel comparing to standard ASTM D6751. The density of watermelon seeds biodiesel was found 0.9113 g/ml which is higher than the density of diesel (0.828). Watermelon seeds biodiesel was found to be less dense than water and can be used as an alternative fuel. The viscosity of watermelon seeds biodiesel and fossil diesel were found 1.68 and 2.71 mm2 /s respectively, which falling in the range of the recommended value for Bio diesel given by ASTM D6751 limits as ranging between 1.9 – 6.0 mm²/s. Viscosity is an important property of any fuel because it indicates the flow ability of a material When oil is converted to biodiesel (Ma & Hanna, 1999) [7]

Flash point is the temperature that indicates the general flammability hazard in the presence of air; higher flash points allow safe handling and storage of biodiesel. The minimum flash points of biodiesel according to ASTM D6751 and EN standards is 130°C and 120°C, respectively (Candeia, *et al*., 2009) [2] The flash point of watermelon seeds biodiesel found 170°C, which met both standards, and the flash point of fossil diesel was found 67.0 °C.

Maximum acid numbers for biodiesel based on ASTM D6751 is 0.5 mg KOH/g, the acid number of watermelon seeds biodiesel was found 0.34 mg KOH/g, indicating the desirable quality of the biodiesel.

Watermelon seeds biodiesel was found to have a water content of 0.036%, which is within the limit for Biodiesel, which is maximum 0.05% according to ASTM D6751.

Table 2: Physical and Chemical properties of watermelon seeds Biodiesel and Diesel

Test	Unit	ASTM D6751 Specification	Fossil Diesel	Biodiesel
Density at 15 °C	g/ml	-	0.8282	0.9113
Density at 20 ºC	g/ml	-	0.8246	0.9079
Pour point	°C	-	-12	3
Cloud point	°C	-	-3	6
Total Acid Number	mg KOH/g	Max. 0.5	0.11	0.34
ASTM Color	-	Max. 3	L1.5	L1.5
Kinematic Viscosity at 40°C	mm²/s	1.9-6.0	2.718	1.68
Flash Point, PMSS	°C	Min. 130	67	170
Water Content	wt. %	Max. 0.05	0.0047	0.036
Micro Carbon Residue	wt. %	Max. 0.05	<0.01	0.22
Conductivity at 20°C	pS/m	Min. 25	70	177
Refractive Index	-	-	1.33	1.33
Total Sulfur	wt. %	Max 0.05	0.0303	0.0152

The pour point and cloud point of watermelon seeds biodiesel were found 3 °C and 6 °C respectively, which were higher than the values of fossil diesel (-12 °C, -3 °C). These properties are related to the use of biodiesel in colder regions.

Carbon residue of watermelon seeds biodiesel was 0.22 wt.%, which is higher than the maximum limit of ASTM D6751 which is 0.05%. While result of fossil diesel found 0.01 wt.%.

The conductivity of watermelon seeds biodiesel and fossil diesel were found 177 pS/m and 70 pS/m respectively. the results met the standard requirement. The refractive index which is a dimensionless number that describes how light passes through the biodiesel was found to be 1.33.

sulfur content was found 0.0152 wt.% and it was below the maximum limit of ASTM D6751 which is 0.05 wt.%. Low sulfur has advantages both for the environment and the engine life.

3. Physical and chemical properties of watermelon seeds biodiesel blends

Table 3 shows the detailed physical and chemical properties of watermelon seeds biodiesel blends compared to ASTM D7467. B20 m*et al*l the requirements of the standard while the Biodiesel-Ethanol blends met most of the standard requirements such as total acid number, kinematic viscosity, and water content. Flash point of the Biodiesel-Ethanol blends had not met the standard requirements. The result shows that the flash point of B20E20 is 12°C, which is lower than the standard requirements due to the presence of residual alcohol in biodiesel. Flash point is important in shipping and safety regulations to define flammable and combustible materials.

Table 3: Physical and Chemical Properties of Watermelon Seeds Biodiesel Blends

Test	Unit	ASTM D7467 Specification	B 20	D60B20E 20
Density at 15 ºC	g/ml	-	0.8448	0.8382
Density at 20ºC	g/ml	-	0.8412	0.8347
Pour point	°C	-	-12	>-12
Cloud point	°C	-	-4	12
Acid Number,	mg KOH/g	Max. 0.3	0.0900	0.0900
ASTM Color	-	-	L1.5	L1.5
Kinematic Viscosity at 40°C	mm²/s	1.9 – 4.1	3.9090	3.0550
Flash Point, PMCC	°C	Min52	73	12
Water Content,	wt. %	0.05	0.0089	0.366
Micro Carbon Residue	wt. %	-	0.0400	-
Conductivity	pS/m	-	130	-
Refractive Index	-	-	1.4612	-
Refractive Index	-	-	1.3915	-
Cold Filter Plugging	°C	-	-8	-5
Total Sulfur	wt. %	Max0.05	0.022	-

Conclusions

- Watermelon seeds biodiesel was synthesized using a two-step transesterification process.
- The biodiesel exhibited a total ester content of 86.02%.
- Saturated fatty acids accounted for 17.11%, with the majority of free fatty acids being unsaturated at 68.91%.
- The produced biodiesel met the recommended standards outlined in ASTM D 6751 for biodiesel fuel.
- The properties of the biodiesel-diesel blend fell within the acceptable range specified by ASTM D7467 standards.
- Biodiesel-diesel-ethanol blends fulfilled the requirements of ASTM D 7467, except the flashpoint**.**

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