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BIODIESEL PRODUCTION FROM SUDANESE *JATROPHA CURCAS* SEED BY THE ALKALI-CATALYZED TRANSESTERIFICATION PROCESS AND ITS ANALYSIS BY GAS CHROMATOGRAPHY

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ABSTRACT

Sudan is highly dependent on diesel. Diesel is used in agriculture machines, transportation, and other applications. This study has been carried out to produced biodiesel from *Jatropha curcas* seed oil. Jatropha was chosen because it is not edible, therefore it will not pose a new problem to humans regarding food competition. This research successfully extracted oil from *Jatropha curcas* seed using n-hexane solvent with yield 39%. The oil than converted to biodiesel by alkaline transesterification process with a conversion yield 92 %. The properties such as flash point, acid value, viscosity, iodine value, density, copper corrosion, cetane number, sulphated ash, pour point, free glycerol, and total glycerol contents were 174 ^{o}C, 0.17 mg KOH/g, 8.912 mm^2/s, 102 g I_2/100 g, 895.8 kg/m^3, class1, 51, 0.0047 %, -3 ^{o}C, 0.01 %,0.03 %, respectively. These quantities obtained were met most of the biodiesel standards that are ASTM D6751, EN14214, and ASTM D975. The GC data showed the fatty acid methyl ester (FAME) has a 12-20 carbon chain and still has monoglycerides, diglycerides, and triglycerides.

Keywords: Biodiesel, FAME, *Jatropha curcas,* Transesterification

1. INTRODUCTION

Alternative diesel fuel biodiesel was made from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and non-toxic, with low emission characteristics, making it environmentally friendly[1]. Free fatty acids, phospholipids, sterols, water, odorants, and other contaminants are common during extraction process to get the crude oil. The crude then will get transesterification process alcohols using alkali or acid catalysts[2].

Jatropha curcas has received increased attention in recent decades as a renewable biofuel source to reduce environmental pollution, desertification, and soil degradation in arid and semiarid regions. Jatropha oil has 5.54 % of moisture, 24.60 % protein, 47.25 % crude fat[3]. Jatropha oil's fatty acid composition differs by region. The primary saturated fatty acids are palmitic acid at 14.1% and stearic acid at 6.7%, while unsaturated fatty acids are oleic acid at 47% and linoleic acid at 31.6%. This composition make it better substitute for biodiesel[3].

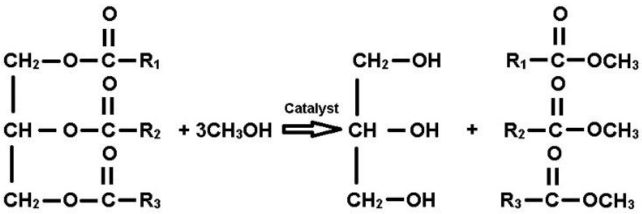

Figure 1 Transesterification of triglyceride with methanol

Figure 1showed the transesterification process to produce a biodiesel (methyl ester). Base-catalyzed transesterification is most preferred used because can occured in room temperature and pressure with high yield[4]. This study was conducted to manufacture biodiesel from *Jatropha curcas* seed oil using KOH as catalyst to produce fatty acid methyl ester (FAME).

Several methods had been developed to extract oil from *Jatropha curcas* seeds, such as mechanical

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pressing[5], and solvent extraction[6], [7]. In this research, the oil from *Jatropha curcas* seeds will be extracted using a hexane solvent extraction method. The biodiesel will characterized, and evaluated for physicochemical properties.

2. METHODS

Materials: Ethanol 99%, *Jatropha curcas* seeds,KOH 97%, distilled water, methanol 99%, toluene 98%, isopropanol, n-hexane 97%.

Instruments: Potentiometric titration, Titrino 885, SVM 3000, Cannon-Fenske routine Viscometer size 75, Cloud and pour point tester - automatic NTE 450, C2000 basic calorimeter, Shimadzu GC-2010.

Extraction procedure: A 100 g of *Jatropha curcas* seeds was extracted with 300 mL of n-hexane in a Soxhlet apparatus about eight hours. At the end of the extraction, the emulsion was cleaned by vacuum filtration to eliminate suspended solids. Then, the organic solvent was removed from the Jatropha oil using a rotary evaporator

Transesterification procedure: A 500 gram of *Jatropha curcas* seed oil was heated to 60 °C in 1 L flask using a heating plate. Then, 200 mL KOH 0.5% (w/v) in methanol is added and the reaction left until 120 minutes. After the reaction was finished, the mix was left for 24 hours to separate the FAME and glycerol. FAME was cleaned using water before the solvent separated with vacuum evaporator. The physicochemical properties of FAME were conducted using EN ISO and ASTM, then compared with ASTM D6751, EN14214, and ASTM D975.

GC was conducted using Shimadzu GC-2010 with flame ionization detector. The SGE HT-5 (25 m x 0.32 um x 0.1 um) was used as column and hydrogen as carrier. The temperature programs are initial 50 ºC, 15 ºC/min up to 180 ºC, 7 ºC/min up to 230 ºC, 20 ºC/min up to 380 ºC[8].

3. RESULTS AND DISCUSSION

The GC-FID chromatogram of *Jatropha curcas* seeds oil FAME showed at Figure 2. From this chromatogram can concluded that *Jatropha curcas* composed by C12-C20 saturated and unsaturated fatty ester. The chromatogram also gives information that palmitic acid and oleic acid are the major composition in *Jatropha curcas* seeds oil. The chromatogram of FAME using EN 14110 standard method (Figure 3) showed the FAME still have glycerol, monoglyceride, diglyceride, and triglyceride as residue.

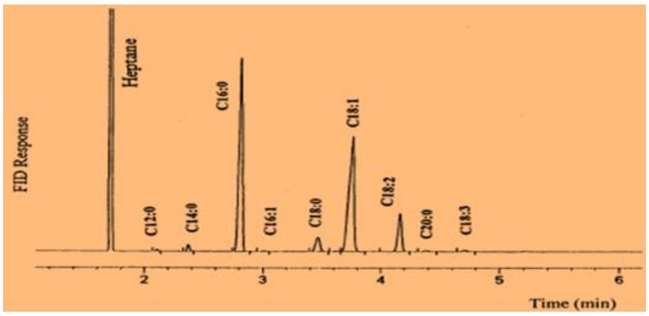

Figure 2. Chromatogram of *Jatropha curcas* FAME composition

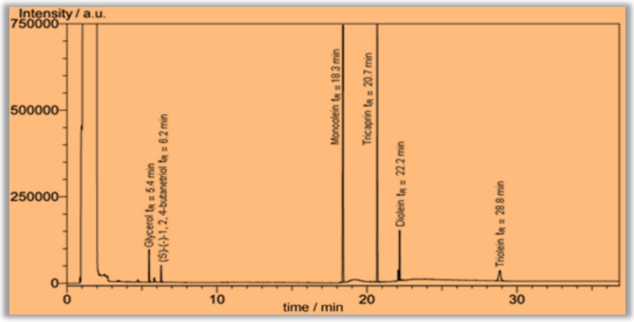

Figure 3. Chromatogram of *Jatropha curcas* FAME residue

The determination of biodiesel fuel quality is an issue of great importance to the successful commercialization of this fuel. ASTM D6751, EN14214, and ASTM D975 were used as FAME standard specification. All the test has done for biodiesel is served in Table 1 and the discussion will do below.

The acid value was calculated using the ASTM D664 standard. The biodiesel had an acid value of 0.17 mg KOH/g. The biodiesel standards ASTM D6751 and EN14214 permitted a maximum acid value of 0.50 mg KOH/g for biodiesel, which the Jatropha FAME met. The free fatty acids who is responsible for acid value can influence low-temperature characteristics and induce corrosion in some metal parts. Copper corrosion test showed the FAME have lower corrosion capability.

The flashpoint observed for *Jatropha* FAME and is 174 °C, which is vital for the handling, storage, and safety of fuels and flammable materials, is within the prescribed limits in American and European biodiesel standards are 130 °C and 101°C, respectively. This data showed that the biodiesel from *Jatropha curcas* seeds oil relatively easy to handle, store, and safe. The *Jatropha* FAME also relatively easy to get because have 90% distillation temperature is 350 ºC. This value meets the ASTM D6751 specification that is maximum 360 ºC.

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Table 1. Properties of FAME from *Jatropha curcas* and its comparison towards diesel and biodiesel specification standards [9]–[36]

Properties of biodiesel	**Unit**	**Diesel**		**Biodiesel Standards**				**Results**
		ASTM D975		**ASTM D6751**		**EN 14214**		
		Method	**Limits**	**Limits**	**Method**	**Limits**	**Method**	
Cetane number	-	ASTM D4737, EN 590	40-55	48-65	ASTM D613	Min 51	EN ISO 5165	51.0
Acid value	mg KOH/g	ASTM D664	0.35	Max 0.5	ASTM D664	Max 0.5	EN 14104	0.17
Kinematic viscosity@40℃	mm^2/s	ASTM D445	2.0-4.5	1.9-6.0	ASTM D445	3.5-5.0	EN ISO 3104	8.912
Sulfated ash	%	ASTM D874		Max 0.02	ASTM D874	Max 0.02	EN ISO 3987	0.0047
Density@15℃	kg/m^3	ASTM D1298	820-860	880	ASTM D1298	860-900	EN ISO 3675	895.8
Water content	% v/v	ASTM D2709	Max 0.05	Max 0.05	ASTM D2709	0.05	EN ISO 12937	0
Copper corrosion	-	ASTM D130	Class 1	No.3	ASTM D130	Class 1	EN ISO 2160	Class 1
Sulfur content	mg/kg	ASTM D5453	Max 0.5	Max 0.05	ASTM D5453	Max 10	EN ISO 20846	0.0063
Iodine value	g I_2/100 g	-	-	-	-	Max 120	EN 14111	102
Flashpoint	°C	ASTM D93	60-80	Min 130	ASTM D93	Min 101	EN ISO 3679	174
Micro Carbon residue	% w/w	ASTM D4530	Max 0.2	Max 0.05	ASTM D4530	Max 0.3	-	0.00
Distillation temperature @90%	℃	ASTM D1160	Max 355	Max 360	ASTM D1160	-	-	350
Pour point	℃	ASTM 97	(-35)-15	(-15)-10	ASTM 97	-	-	-3
Linolenic methyl ester	% w/w	EN 14103	-	-	-	Max 12	EN 14103	0.17
Polyunsaturated methyl esters	% w/w	-	-	-	-	Max 1	EN 14103	0
Ester content	% w/w	-	-	-	Min 96.5	Min 96.5	EN 14103	95
Methanol content	% w/w	-	-	-	-	Max 0.2	EN 14110	0.0012
Alkaline metals (Ca+Mg)	mg/kg	-	-	-	-	Max 5	EN 14538	4.7
Phosphorus content	mg/kg	-	-	-	-	Max 10	EN 14107	<4

Another essential attribute of diesel and its substitution is cetane number. Cetane number used to measuring a diesel combustion quality during compression ignition. The cetane number observed for *Jatropha curcas* FaME is within the allowed limits in American and European biodiesel standards and greater than diesel fuel. Another significant component for optimal engine performance and determining the quality and pricing of biodiesel is density. The density of the biodiesel produced was 895.8 kg/m^3, which is

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well within the ASTM D6751 and EN14214 requirements. The *Jatropha curcas* seeds oil but slightly higher than the density of diesel standard 820-860 kg/m^3. It means the biodiesel will change the machine performance due its density and maybe some machine modification needed.

Fuel with high viscosity will create a bigger drop of fuel in the combustion chamber of an engine, which may not burn completely and make a carbon residue. This residue can cause issues like clogging of engine components. The *Jatropha curcas* biodiesel at 40°C have kinematic viscosity 8.912 mm^2/s. This value higher than of ASTM D6751 and EN14214 that are 1.9-6 mm^2/s and 3.5-5.0 mm^2/s, respectively. This value also higher than the viscosity for diesel standard (ASTMD975) that is 2.0-4.5 mm^2/s. This value means the FAME cannot used alone as a fuel but needs a blending process with a petroleum.

The iodine value is a significant measure for determining the unsaturated free fatty acids. The iodine value of biodiesel is 102 g I_2/100 g, meeting the value of EN14214 biodiesel standards is max 120 g I_2/100 g. Water content can cause corrosion of containers and apparatus, and can cause an emulsion. The water content observed for FAME is 0.05%, that's within the approved limits in ASTM D6751 and EN 14214 biodiesel standards is max 0.05%.

The pour point values of biodiesel manufactured and *Jatropha curcas* seed oil was -3 °C shown in Table 1, due to the greater unsaturated fatty acid content in raw *Jatropha curcas* seeds oil. The result of pour point test was found to be within the specified limits of ASTM D975 diesel standards that is between -35 °C - 15 °C and ASTM D6751 biodiesel standard specifications -15 °C - 10 °C. This value means, the biodiesel from *Jatropha curcas* is suitable not only for the tropical region but also for moderate temperature region.

The inorganic content such as alkaline metals, carbon residue, phosporous, and sulfur can affect machine performance and durability, gas emission, and carbon tax. All these inorganic contents meet the biodiesel specification. Although the FAME meets mayority of spesification standards of ASTM D6751 and EN 14214, the ester content was below the spesification. This research only get 95 %(w/w), not reach the minimum content 96.5 %(w/w). Still need a modification on FAME synthesized process to get optimum FAME product.

4. CONCLUSION

Jatropha curcas seeds oil FAME is a promising feedstock for biofuel in Sudan country. The FAME was synthesized by alkali-catalyzed transesterification with 95% yield. GC-FID and GC-MS chromatograms showed the biodiesel C12-C20 saturated and unsaturated fatty ester, and residue such monoglyceride, diglyceride, triglyceride, and glycerol.

Important fuel properties of biodiesel such as flash point, acid value, viscosity, iodine value, density, copper corrosion, cetane number, and pour point contents are 174 °C, 0.17 mg KOH/g, 8.912 mm^2/s , 102 g I_2/100 g, 895.8 kg/m^3, class 1, 51, and -3 °C , respectively. The biodiesel still has inorganic materials such as alkaline metals, phosphorous, and sulfur.

These quantities obtained were compared with ASTM D6751, EN14214 and ASTM 975 specifications. The properties values of biodiesel produced met most of the biodiesel and fossil diesel standards except the viscosity. The viscosity value is slightly bigger than the required standards. This problem can be easily improved by blending with low viscosity diesel.

REFERENCES

[1] F. Ma and M. A. Hanna, “Biodiesel production: a review1,” *Bioresour. Technol.*, vol. 70, no. 1, pp. 1–15, 1999.

[2] M. Y. Koh and T. I. Tinia, “A review of biodiesel production from Jatropha curcas L. oil,” *Renew. Sustain. Energy Rev.*, vol. 15, no. 5, pp. 2240–2251, 2011.

[3] R. Abdulla, E. S. Chan, and P. Ravindra, “Biodiesel production from Jatropha curcas: A critical review,” *Crit. Rev. Biotechnol.*, vol. 31, no. 1, pp. 53–64, 2011.

[4] S. Sivaprakasam and C. G. Saravanan, “Optimization of the transesterification process for biodiesel production and use of biodiesel in a compression ignition engine,” *Energy and Fuels*, vol. 21, no. 5, pp. 2998–3003, 2007.

[5] Maftuchah, A. Zainudin, A. Winaya, and Y. Rahmadesi, “Biodiesel generated from Jatropha (Jatropha curcas Linn.) seeds selected based on various genotypes crossbred,” *Energy Reports*, vol. 6, pp. 345–350, 2020.

[6] K. Shrestha, J. R. Ghimire, and M. B. Gewali, “Biodiesel Production from Jatropha curcas (L.) Oil of Nepal by Transesterification Process,” *Int. J. Pharm. Chem. Biol. Sci.*, vol. 3, no. 3, pp. 603–609, 2013.

[7] F. Ahmed, S. O. Giwa, M. Ibrahim, and A. Giwa, "Production of biodiesel from Jatropha curcas seed oil using base catalysed transesterification," *Int. J. ChemTech Res.*, vol. 9, no. 6, pp. 322–332, 2016.

[8] A. N. Dias *et al.*, "Evaluation of ASTM D6584 method for biodiesel ethyl esters from sunflower oil and soybean/tallow mixture and for biodiesel methyl esters from tung oil and soybean/tung mixture," *J. Braz. Chem. Soc.*, vol. 25, no. 7, pp. 1161–1165, 2014.

[9] CEN/TC 307, "EN 14107 Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry," 2003.

[10] CEN/TC 307, "EN 14103 Fat and Oil Derivatives - Fatty Acid Methyl Esters (Fame) - Determination of Ester and Linolenic Acid Methyl Ester Contents," 2020.

[11] ISO/TC 28, "ISO 3679 Determination of flash point - Method for flash no-flash and flash point by small scale closed cup tester," 2022.

[12] CEN/TC 307, "EN 14111 Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of iodine value," 2022.

[13] ISO/TC 28, "ISO 20846 Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method," 2019.

[14] ISO/TC 28, "ISO 2160 Petroleum products - Corrosiveness to copper - Copper strip test," 1998.

[15] ISO/TC 28/SC 2, "ISO 12937 Petroleum products - Determination of water - Coulometric Karl Fischer titration method," 2000.

[16] ISO/TC 28/SC 2, "ISO 3675 Crude petroleum and liquid petroleum products - Laboratory determination of density - Hydrometer method," 1998.

[17] ISO/TC 28, "ISO 3987 Petroleum products - Determination of sulfated ash in lubricating oils and additives," 2010.

[18] ISO/TC 28, "ISO 3104 Petroleum products - Transparent and opaque liquids -Determination of kinematic viscosity and calculation of dynamic viscosity," 2020.

[19] CEN/TC 307, "EN 14104 Fat and oil derivates. Fatty acid methyl ester (FAME). Determination of acid value," 2021.

[20] ISO/TC 28, "ISO 5165 Petroleum products - Determination of the ignition quality of diesel fuels - Cetane engine method," 2020.

[21] CEN/TC 19, "EN 14214 Liquid petroleum products - Fatty acid methyl esters (FAME) for use in diesel engines and heating applications - Requirements and test methods," 2019.

[22] ASTM Internasional, "ASTM D97 Standard Test Method for Pour Point of Petroleum Products," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 7.

[23] ASTM Internasional, "ASTM D1160 Standard Test Method for Distillation of Petroleum Products at Reduced Pressure," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 20.

[24] ASTM Internasional, "ASTM D93 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 18.

[25] ASTM Internasional, "ASTM D4530 Standard Test Method for Determination of Carbon Residue (Micro Method)," in *ASTM Volume 05.02: Petroleum Products, Liquid Fuels, And Lubricants (II): D4177 – D6468*, 2021, p. 7.

[26] ASTM Internasional, "ASTM D5453 Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence," in *ASTM Volume 05.02: Petroleum Products, Liquid Fuels, And Lubricants (II): D4177 – D6468*, 2021, p. 12.

[27] ASTM Internasional, "ASTM D130 Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 10.

[28] ASTM Internasional, "ASTM D2709 Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 3.

[29] ASTM Internasional, "ASTM D1298 Standard Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 8.

[30] ASTM Internasional, "ASTM D874 Standard Test Method for Sulfated Ash from Lubricating

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e-ISSN: 2776-4508; p-ISSN: 2776-1711
Publisher : Universitas Pakuan

Oils and Additives," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 5.

[31] ASTM Internasional, "ASTM D445 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 17.

[32] ASTM Internasional, "ASTM D664 Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 11.

[33] ASTM Internasional, "ASTM D613 Standard Test Method for Cetane Number of Diesel Fuel Oil," in *ASTM Volume 05.05: Petroleum Products, Liquid Fuels, And Lubricants (V): D8144 – Latest; Combustion Characteristics; Manufactured Carbon And Graphite Products*, 2021, p. 19.

[34] ASTM Internasional, "ASTM D4737 Standard Test Method for Calculated Cetane Index by Four Variable Equation," in *ASTM Volume 05.02: Petroleum Products, Liquid Fuels, And Lubricants (II): D4177 – D6468*, 2021, p. 5.

[35] ASTM Internasional, "ASTM D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels," in *ASTM Volume 05.03: Petroleum Products, Liquid Fuels, And Lubricants (III): D6469 – D7398*, 2021, p. 11.

[36] ASTM Internasional, "ASTM D975 Standard Specification for Diesel Fuel," in *ASTM Volume 05.01: Petroleum Products, Liquid Fuels, And Lubricants (I): C1234–D3705*, 2021, p. 28.