

Biodiesel production from waste cooking sunflower oil and environmental impact analysis

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Abstract

Waste cooking oil offers great potential as a low cost biodiesel feedstock. Several parameters were tested for the optimum production of biodiesel and these included varying the alcohol:oil molar ratios, different catalyst concentrations, temperatures and stirring speed. For the optimum production of biodiesel, the molar ratio of alcohol to oil used was 6:1. The fatty acid methyl esters identified in the biodiesel were methyl palmitate, methyl linoleate, methyl oleate and methyl stearate. The viscosity of the produced biodiesel was within the range of international ASTM standards. Engine exhaust emission tests of biodiesel showed that the carbon monoxide and unburned hydrocarbon emissions were lower than that of petrodiesel. The nitrogenous oxides emission and specific fuel consumption were higher than that of conventional diesel fuel. It can be concluded that biodiesel produced from waste sunflower oil can be considered as a great potential source of commercial biodiesel.

Keywords: Biodiesel; emission; fuel consumption; sunflower oil; viscosity.

1. Introduction

Different types of vegetable oils can be utilized for the production of biodiesel, such as sunflower, palm, soybean, coconut, rapeseed, safflower and cottonseed oils (Saifuddin *et al.*, 2014a; Lisboa *et al.*, 2014; Andreani & Rocha, 2012; Somporn & Shabbir, 2009; Steven & Lee, 2010). All these sources are considered to have the potential resources as raw materials for the production of biodiesel. It can be also derived from renewable biomass feedstock, such as algae and animal fats, biodegradable (Wyatt *et al.*, 2010; Steven & Lee, 2010; Hossain & Boyce, 2009; Hossain *et al.*, 2008; Saifuddin *et al.*, 2014b). There are no restrictions to the use of other types of vegetable oils.

Within several methods to produce biodiesel, transesterification is one of the most used ways to produce biodiesel (Lisboa *et al.*, 2014). Transesterification is the process of reacting a triglyceride molecule with an excess of alcohol in the presence or absence of a catalyst to produce fatty acid methyl esters and glycerol (Demirbas, 2005). The purpose of the transesterification process of vegetable oils to their methyl esters is to get the lower viscosity of the oil (Sheinbaum-Pardo *et al.*, 2013). Since, viscosity is the most vital characteristics to assess the biodiesel quality and its effects on the operational injecting equipment. Consequently, fuel consumption

and gas emission rate are important as well. The increase in viscosity makes incomplete combustion and poor atomization of the fuel spray due to low fluidity of the fuel (Encinar *et al.*, 2007). Lower viscosity biofuel can be generated by using transesterification reaction of triacylglycerides oil with an alcohol in the presence of an alkali or acid catalyst (Nezihe & Aysegul, 2007).

Biodiesel fuel is an environmental friendly fuel that can be used in diesel engines with little or without any significant modifications (Xiangmei *et al.*, 2008; Hossain *et al.*, 2010). It also releases less exhaust gases compared to petrol diesel fuel or petrodiesel (Zhang *et al.*, 2003). Biodiesel is also non-flammable, non-toxic and non-explosive. It has a high flash point (with a flash point of 423 K as compared to 337 K for petrodiesel), heat content and excellent lubricity (Demirbas, 2005). Studies have shown that biodiesel reduces the emission of carbon monoxide, sulphur, polyaromatics, unburned hydrocarbon, smoke, noise and particulate emissions, compared to regular diesel fuel (Demirbas, 2005). Therefore, the objectives of this study were to produce biodiesel from waste cooking sunflower oil, to evaluate the different variables affecting the alkaline methanolysis of waste oil and to determine the characteristics of the biodiesel; for example, to identify methyl esters present in the biodiesel, the exhaust emissions and fuel consumption by engines.

2. Materials and methods

2.1. Transesterification reaction and purification of the methyl esters

The waste sunflower cooking oil was filtered to remove food residues and solid precipitates. The transesterification reactions were performed. The cooking oil was first heated to 60°C in an incubator to liquidify the oil and to remove water from the waste sunflower oil. Methanol and dissolved catalyst were then added to 56 ml of waste sunflower oil and the mixture was stirred vigorously. The reaction mixture was stirred for particular reaction time in an incubator shaker at room temperature. At this stage the waste sunflower oil is converted to its corresponding esters.

Once the reaction is complete, two major products exist: glycerin and biodiesel. Each has a substantial amount of excess methanol that was used in the reaction. Excess methanol was used in the biodiesel formation to ensure the total conversion of the oil to its esters. The reaction mixture was poured into a beaker and left under a fan for about 20 minutes to dry. Excess methanol in the mixture is then removed via evaporation. Phase separation occurs at this stage. The reaction mixture was then poured into a separating funnel. The glycerin layer is much denser than biodiesel layer. As a result, the two layers will be separated by gravity forces, where glycerin is at the bottom. Glycerin can be drawn off the bottom of the separating funnel, while the methyl esters (biodiesel) form the upper layer (Figure 1).

Once the glycerin and biodiesel layers have been separated, the glycerin layer was removed and the mixture was neutralized with an acid. Once separated from the glycerin, the biodiesel is purified by washing gently with warm distilled water to remove residual catalyst or soaps. This procedure is continued until the methyl ester layer becomes clear. It is then dried by using anhydrous sodium sulphate to remove all the water and sent to storage. Then the finished biodiesel will be analyzed to assess the ASTM specifications.

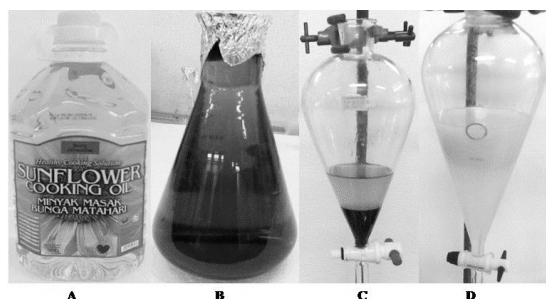


Fig. 1. A) Straight sunflower oil, B) Filtered waste sunflower cooking oil, C) Phase separation, upper is biodiesel and D) Formation of emulsion.

2.2. Optimizing the conditions for biodiesel production

Methanol was used for the transesterification reaction to produce biodiesel from waste sunflower oil. Five different molar ratios of oil to alcohol, 4:1, 3:1, 1:3, 1:4 and 1:6 were used in this reaction. Sodium hydroxide was used in the experiments as a catalyst. Four different percentages of catalysts by weight of oil were used to produce biodiesel. These were 0.05 g, 1.00 g, 1.50 g and 2.00 g of NaOH. The temperature range was between 25 - 62.5° C and the reaction time range was between 0.5 - 8.5 hours. Lastly the stirring speed was varied between 50 - 300 rpm.

2.3. Biodiesel analysis

2.3.1. Gas chromatography-mass spectrometry (GC-MS) analysis

The specific component of methyl esters in the biodiesel were determined using an Agilent 6890 Gas Chromatography installed with a mass spectrometry detector. A capillary column, (length: 30 m, film thickness: 0.25m and ID: 0.25 mm) was used. Helium was used as the carrier gas.

2.3.2. Characterization of the biodiesel

The biodiesel produced under optimum conditions was sent to the Engine Tribology Laboratory of Department of Mechanical Engineering to determine the properties. Several properties of the biodiesel were determined (Figure 2):

- 1) Kinematic viscosity
- 2) Total acid number (TAN) and total base number (TBN)
- 3) Multi element concentration

2.3.3. Engine exhaust emission test

A volume of 2.50 liters of biodiesel was required to perform the engine emission test. The BOSCH gas analyzer model EET 008.36 was used to measure carbon monoxide (CO) and unburned hydrocarbon (HC). Whereas, the Bacharach model CA300NSX analyzer was used to measure NO_x concentration. The measurement of CO and NO_x were according to SAE J117 June'95 standards and HC to SAE J215 March'95 standards. Fuel consumption of the biodiesel was also measured and compared with the fuel consumption of normal petrodiesel. The model of the engine was YANMAR TF120-M. This engine is horizontal, water-cooled and single cylinder.

The accumulation of deposit was carried out for 8 hours at 2000 rpm constant engine speed and 15 Nm load for each test fuel.



Fig. 2. Viscosity, TAN and multi element analyzer.

3. Results and discussion

With regard to catalyst concentration, the optimum value for alkaline transesterification is 2% (Figure 3). As the catalyst concentration increases, the biodiesel yield increases as well until a maximum concentration of 1% (Bhatti *et al.*, 2008). When it exceeded 1%, a decrease was observed. But the maximum biodiesel has been achieved by 2% NaOH. When the catalyst concentration was below 2%, emulsion or soap (due to saponification reaction with sodium hydroxide) has occurred and hence the viscosity of the whole solution increased and finally resulted in the formation of a gel. These explanations show that a catalyst concentration of 2% is the optimum for transesterification (Yaakob *et al.*, 2013).

Figure 4 shows that maximum biodiesel yield is achieved using a molar ratio of 1:6 for oil:methanol. Theoretically, an oil /methanol ratio of 1:3 is enough to form methyl esters from the reaction of methanol with triglycerides. However, the reaction might be in equilibrium or backward. The yield of methyl esters will increase, as the methanol:oil ratio increases. However, their relationships are not directly proportional, but rather hyperbolic. According to many studies, a ratio of 1:6 is sufficient for the high yield (almost complete) of methyl esters (Guan *et al.*, 2008; Hossain & Boyce, 2009; Hossain *et al.*, 2010). With a higher oil:alcohol ratio, only a slight improvement in methyl esters yield can be obtained. For more economically viable, production of biodiesel, a maximum oil:methanol ratio of 1:6 was used and it contributed to maximum conversion rate in this experiment.

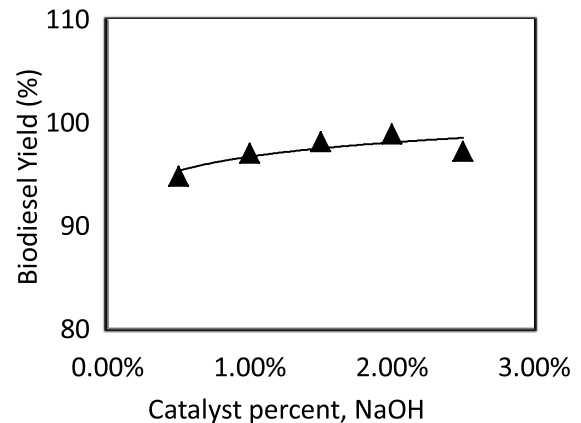


Fig. 3. Optimization of alkaline transesterification of waste palm oil.

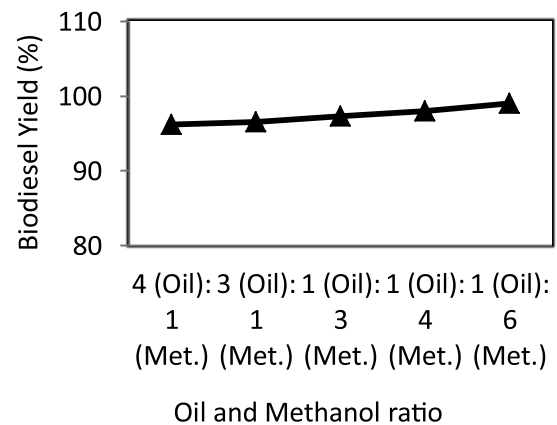


Fig. 4. Optimization of bioethanol yield by different oil and methanol ratio.

3.1. Biodiesel analysis

3.1.1. Gas chromatography-mass spectrometry (GC-MS) analysis for chemical composition

The spectrum in the GC-MS record shows how a compound is broken up into fragments by means of energy (usually a beam of electrons) (Woods & Fryer, 2007). The way a compound splits into fragments is characteristic of its structure. The fatty acid methyl ester (FAMES) profile in the produced biodiesel is shown in Figure 5.

It can be seen that there are a total of 4 compounds that are present in the biodiesel sample. Details of these compounds are shown in the library search report (Table 1) containing the retention time, area (%), possible identities, and the qualities of the compounds. Each of the peaks of solution has its unique retention time, which is important in determining the chemical compounds in the biodiesel sample (Wisniewski *et al.*, 2010).

The FAMES are separated according to carbon number (the number of carbon atoms in the fatty acid chain, not

including the methyl ester carbon) and according to the degree of unsaturation. The position of the double bond(s) and their configuration (cis or trans) are also important parameters and adds additional information to the characterization of the biodiesel. A good separation was obtained (Figure 6), the yields of isolated FAMES are methyl palmitate, methyl linoleate, methyl oleate and methyl stearate, which eluted at 20.577, 23.792, 23.898 and 24.330 minutes, respectively. This separation is sufficient for most classical oil characterization. The biodiesel consists mainly of 9-octadecenoic acid

(z)-, methyl ester (methyl oleate) (i.e. C18:1) and 9, 12-octadecadienoic acid (z, z)-, methyl ester (methyl linoleate) (i.e. C18:2). The biodiesel produced composed mainly of 18 carbon fatty acids. Besides linear saturated fatty acids, di-unsaturated and polyunsaturated fatty acids were seen (Lina *et al.*, 2006). The mass spectrum of the highest peak indicated that the compound present was linoleic acid methyl esters. The results shown in Figure 6 were similar to the reported fatty acid profile of sunflower oil as mentioned in Table 1.

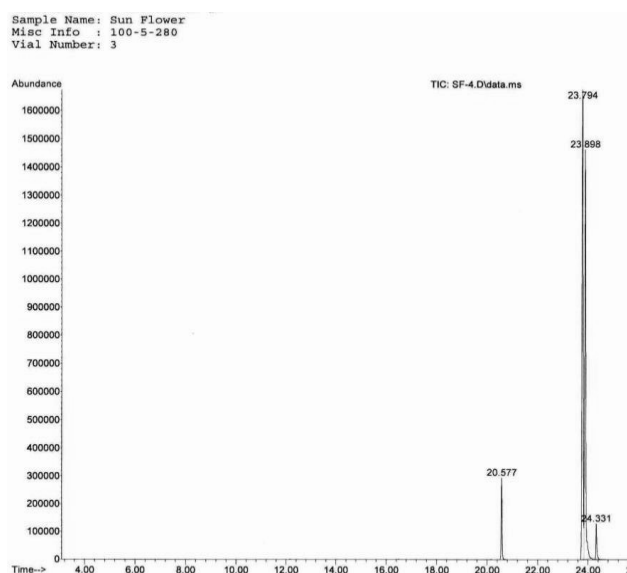


Fig. 5. Identification of each peak.

Table 1. GC/MS library search report for sunflower biodiesel

Product Me-ester	Retention Time min.	% Peak Area	% Purity
Hexadecanoic acid	20.577	6.43	98
Octadecadienoic acid	23.792	48.22	99
Octadecenoic acid	23.988	42.14	99
Octadecanoic acid	24.330	3.21	98

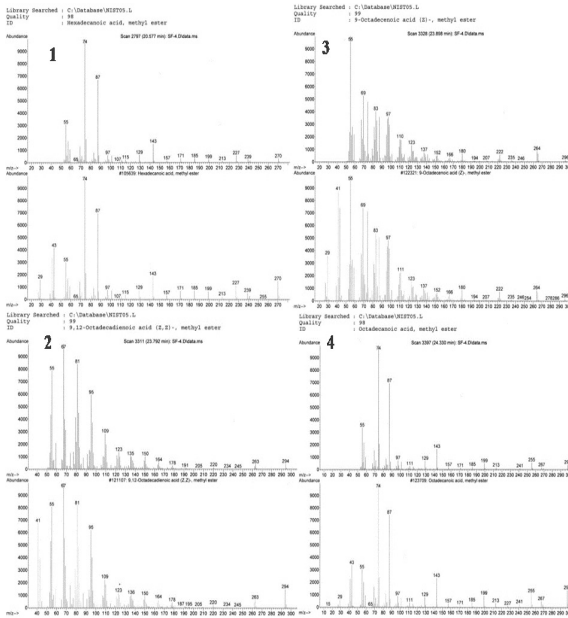


Fig. 6. Mass spectrum of each peak and identification of corresponding components. Whereas, 1) Hexadecanoic acid, 2) 9,12-octadecadienoic acid (z, z)-, 3) 9-octadecenoic acid (z)-, and 4) Octadecanoic acid, methyl ester peaks.

3.2. Characterization of the biodiesel

3.2.1. Kinematic viscosity

From Table 2, the viscosity of the biodiesel produced from transesterification of waste sunflower oil was 4.74 cSt. This kinematic viscosity value at 40°C is limited between 1.9 to 6.0 cSt. Therefore, the waste sunflower oil-based biodiesel met the specification of the ASTM standards. The viscosity value of waste cooking oil is decreased after transesterification and prevented operational problems such as engine deposits (Hossain & Buyce, 2009; Demirbas, 2005; Encinar *et al.*, 2007). The viscosity values of vegetable oils vary between 23.2 and 53.0 cSt, 9 to 17 times greater than petroleum diesel fuel (Mohammed, 2009; Demirbas, 2005). The total viscosity of biodiesel depends on each component inside the solution also and its chain length of either the fatty acid or alcohol moiety in a fatty acid methyl (Knothe & Steidley, 2005). Therefore, these chemical compositions of sunflower biodiesel are suitable for maintaining a desired viscosity as per ASTM.

3.2.2. Total acid number (TAN) and total base number (TBN)

If the free fatty acid (especially unsaturated fatty acids) content of the biodiesel is higher, then the oxidation stability of biodiesel will be less (Ma & Hanna, 1999). Free fatty acid can lead to corrosion of the engine due

to its acidity and may be an indication of the presence of water in the fuel (Haseeb *et al.*, 2010). The total base number (TBN) determines how effective the control of acids formed will be, during the combustion process. The ASTM standard approved a maximum acid value for biodiesel of less than 0.8 mg KOH/g and 10-15 mg KOH/g of total base number for diesel engine operations. However, the TBN values of the biodiesel produced in this study were not detectable but TAN is 1.4 (Table 3). This may be due to the sample being less reactive or there was excessive base value. Waste cooking oils are usually high in TAN value due to the presence of high free fatty acid content (Leung *et al.*, 2006; Ma & Hanna, 1999; Hossain & Buyce, 2009).

Table 2. Viscosity of the biodiesel and the ASTM standard.

Characteristics	Value (cSt)	ASTM standard (cSt)
Viscosity (at 40°C)	4.74	1.9 – 6.0

Table 3. Total Acid Number (TAN) and Total Base Number (TBN) of the biodiesel produced and their respective ASTM standard.

Characteristics	Value, mg KOH/g	ASTM standard mg KOH/g
TAN	1.4	<0.8
TBN	No	10 – 15

3.2.3. Multi elements concentration

Metal elements such as phosphorus, magnesium, and calcium present in the biodiesel can form ash deposits in fuel injection system and poison the catalytic converters of the engines and reduce its ability to decrease the exhaust emission (Korn *et al.*, 2007). An ASTM D6751 standard limit for sodium, magnesium and calcium concentrations is less than 5 ppm to prevent engine damage. The magnesium values belong to the limits of ASTM D6751 (Figure 7). Sodium hydroxide was utilized as catalysts in this experiment and anhydrous sodium sulfate was used as drying agent in the biodiesel production. That is why NaOH value is little higher than ASTM. Phosphorus level was found to be very high (20.5) in the biodiesel produced from waste cooking sunflower oil. High level of phosphorus may be due to incomplete refining of the vegetable oil and from proteins encountered in the rendering process. Phospholipids and phosphorus sources may be impeded by phase separation method during the transesterification process due to their emulsifying properties and residual phosphorus can also be removed by distillation process.

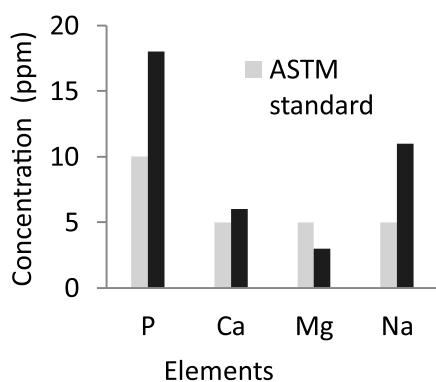


Fig. 7. Elements concentration of the produced biodiesel and their respective ASTM standard.

3.3. Engine exhaust emission test

3.3.1. Carbon monoxide (CO), Hydrocarbon (HC) and Nitrogenous oxide (NO_x) emission

The CO emission of the produced biodiesel was much lower than diesel, with readings of 0.025 and 0.028 %, respectively (Table 4). The produced biodiesel is 10.71 % lower in carbon monoxide emission, compared to diesel. Biodiesel itself has about 11% oxygen content in it and this facilitates complete combustion. In addition, the diesel engine used in this experiment provided more air at high speed, which increases the turbulence intensity in the combustion chamber and can affect the air-fuel mixing process. This probably led to a more complete combustion and thus the lower CO emission is obtained at high speed (Pugazhivadivu & Jeyachandran, 2005).

It was found that the biodiesel produced showed lower HC emission, 22.75 ppm, compared to diesel, 25.33 ppm. Its emission level was 10.19% lower than diesel fuel. In this current experiment, the significant reduction of the unburned hydrocarbons is probably due to complete combustion of the sunflower biodiesel (Ramadhas *et al.*, 2005).

The produced biodiesel showed a higher NO_x emission, of 548.67 ppm, compared to diesel (515.40 ppm). The NO_x emission of the biodiesel was 6.46%, which is slightly higher than diesel. This increase is probably due to the higher combustion temperatures and the higher oxygen content of the biodiesel, which will lead to better oxidation of the nitrogen, thus increasing the nitrogenous oxide emission (Lei *et al.*, 2010; Pugazhivadivu & Jeyachandran, 2005).

3.3.2. Specific fuel consumption (SFC)

It can be seen that the waste sunflower based biodiesel showed slightly higher fuel consumption (0.612 ml/sec) than that of diesel (0.588 ml/sec). At the 15 Nm load condition, the fuel consumption of biodiesel was more than 4.08 % than that of diesel. This is probably due to the oxygen content and consequently, the lower calorific value of the waste sunflower based biodiesel compared to petrodiesel (Ramadhas *et al.*, 2005).

Table 4. Fuel consumption and greenhouse gas emission analysis.

Sample	Load (Nm)	Speed (rpm)	Fuel consumption, ml/sec	HC, ppm	CO, Vol. %	NO _x , ppm
Biodiesel	15	2000	0.612	22.75	0.025	548.67
Diesel	15	2000	0.588	25.33	0.028	515.40

4. Conclusion

In conclusion, four methyl ester peaks were observed in the chromatogram and a library search report from the GC-MS showed that the fatty acid methyl esters found in the biodiesel were methyl palmitate, methyl linoleate, methyl oleate and methyl stearate. The viscosity and magnesium content of produced biodiesel met the ASTM standard specifications and it had a lower emission of carbon monoxide and unburned hydrocarbon, compared to conventional diesel. However, there was a slight increase

in nitrogenous oxide emission and fuel consumption vis-à-vis petro-diesel. The phosphorus content was also higher than that specified by the ASTM standards. Thus, waste cooking sunflower oil is recommended as a potential feedstock for biodiesel production.

5. Acknowledgement

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إنتاج وقود الديزل الحيوي من نفايات زيت الطبخ المستخرج من بذور عباد الشمس و تحليل الأثر البيئي

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خلاصة

تمثل متبقيات زيت الطبخ قيمة كبيرة كمصدر لوقود ديزيل حيوي منخفض التكلفة. تم اختبار العديد من العوامل المؤثرة للوصول إلى الإنتاج الأمثل من وقود الديزل الحيوي وشملت هذه العوامل التغير في محتوى الكحول والنسبة المولية للزيت والتركيزات المختلفة للعامل المساعد والحرارة وسرعة الخلط. وجد أن النسبة المولية من الكحول إلى الزيت المؤدية إلى أفضل إنتاج هي 6:1. كانت الإسترات الميثيلية للأحماض الدهنية التي تم تحديدها في وقود الديزل الحيوي هي بالميتات الميثيل، لينولييت الميثيل، أولييت الميثيل وستيرات الميثيل. كانت لزوجة الوقود الديزل الحيوي المنتج في نطاق معايير ASTM الدولية. وأظهرت اختبارات عادم المحرك من وقود الديزل الحيوي أن انبعاثات أول أكسيد الكربون والهيدروكربونات غير المحترقة أقل من تلك في حالة الديزل البترولي. كما كانت انبعاثات الأكاسيد النتروجينية واستهلاك الوقود أعلى من وقود الديزل التقليدي. ويمكن أن نخلص إلى أن وقود الديزل الحيوي المستخرج من نفايات زيت عباد الشمس يمكن أن يكون مصدرا هاما من وقود الديزل الحيوي التجاري.