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Development and Characterization of Biodiesel: Analyzing Properties Through GC-MS and Fuel Analyzer

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ABSTRACT
This study examines the production and characterization of biodiesel which is derived from soybean oil through the use of a transesterification process. Biodiesel formed via the chemical reaction of soybean oil and methanol in the presence of sodium hydroxide (NaOH) as a catalyst. The resulting biodiesel samples were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) and Fuel analyzer. The highest yield of methyl ester was obtained by using 60 mL methanol, 250 mL Soyabean oil and 1.4 g NaOH was used as a catalyst. The reaction was carried out at 60 °C for 3h. Among the four primary methyl esters, hexadecenoic acid methyl ester is the dominating ingredient in the combination, with the greatest yield of 11%. The fuel properties of the synthesized biodiesel were evaluated and contrasted with conventional diesel. Notably, the biodiesel exhibited a cetane number of 68.4, considerably higher than that of pure diesel, indicating superior ignition quality. But compared to diesel, biodiesel was shown to have much lower flash and fire points, which could make handling and storage more difficult. These findings show the possibility of soybean oil-derived biodiesel as a viable alternative to traditional diesel, with its higher cetane number enabling increased combustion efficiency. Nevertheless, the decreased flash and fire thresholds demand more work to improve the safety properties of the biodiesel.

Keywords: Biodiesel; Transesterification; GC-MS; Alternative energy; Fuel analyzer.

1. Introduction

Geographically, Bangladesh can produce biodiesel from non-edible materials such as rubber, cotton seed, castor, bahera, neem, karanja, and jatropha. Algal cultivation is the most viable alternative, covering 6.1 million hectares of barren land and water. However, costs must be reduced to make it a viable substitute for diesel [1]. The ASTM definition of biodiesel is a fuel composed of mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats". Fatty acid methyl esters (FAMEs) make up the majority of biodiesel, one of these alternative energy sources. There are four main methods to obtain it: (a) using and combining oils directly, (b) creating oil microemulsions, (c) thermal cracking (vegetable oil pyrolysis), and (d) transesterification (oil alcoholysis). Transesterification of vegetable oils, including soybean, rapeseed, sunflower, palm, coconut, tung, and waste cooking, is the most widely used technique [2]. The chemical process of Transesterification, which creates fatty acid alkyl esters when triglycerides and alcohols react with a catalyst, is how biodiesel is made. Glycerin, sometimes called glycerol, results from transesterification [3]. Enzymes, acids, or alkali can catalyze reactions involving Transesterification. Water and minimal levels of free fatty acids (FFAs) in oil are best suited for alkaline catalysts, whereas a high FFA level is best suited for acid catalysts [4]. Because it is quicker, alkali-catalyzed Transesterification is frequently employed in industry. Numerous investigations have been carried out in lab settings, with molar ratios of 3:1 to 6:1 for alcohol/soybean oil and suggested reaction temperatures close to the boiling point of alcohol [5, 6]. Utilizing biodiesel instead of diesel can improve rural industries' financial standing, increase energy security, and lessen the harmful environmental effects of fossil fuels [7-12]. Because of its high oil content, accessibility, and established agricultural enterprise, soybean oil is commonly used as a feedstock for manufacturing biodiesel. Due to its significant amount of unsaturated fatty acids, it may be transesterified to create biodiesel. Compared to other feedstocks, including palm oil, rapeseed oil, and leftover cooking oil, soybean oil has the advantages of consistent quality, widespread availability, and environmental benefits. It is an essential part of renewable fuel projects and keeps promoting energy security and environmental sustainability [13]. Biodiesel offers improved lubrication, encouraging efficient engine component operation, lowering engine wear, and increasing engine longevity. Sustainable, non-toxic, biodegradable, non-flammable biodiesel is good for the environment. Because of all these characteristics, biodiesel is among the finest substitute fuels for meeting future energy needs [14]. However, there are certain disadvantages to biodiesel. Fuel atomization, combustion, and pumping issues may arise from the increased viscosities of biodiesel fuels, typically between 11 and 17 times greater than those of regular diesel [15]. Fuel injectors at the engine's head and piston may also coke due to using biodiesel. Moreover, operating biodiesel in an unaltered compression ignition engine may result in excessive engine wear [16, 17]. It is possible to produce biodiesel from various oil sources, including soybean, mustard, sesame, moss, jatropha, castor, bahera, neem, and algae in Bangladesh. However, the country only produces enough soybeans to cover 40% of its domestic oil demands. However, 1.7–1.8 million metric tons of soybeans may be produced on 0.7–0.8 million hectares of land in char areas under cultivation. In Bangladesh, mustard plants are also grown in large quantities [18]

Bangladesh's energy sector faces significant pressure due to increasing population growth and reliance on natural gas and petroleum oil. Despite being the twenty-eighth-ranked natural gas producer in 2014, 23.6 billion cubic meters of produced gas are consumed domestically [19, 20] The nation's crude oil reserves are limited, and its natural gas reserves might run out in ten years. To rectify the deficit, the government imports 108187 barrels daily.[28] Fossil fuels provide the bulk of primary energy and contribute to environmental deterioration. Alternative sources like solar, wind, water, geothermal, biomass, and biofuels are being explored, with ethanol and biodiesel being common biofuels. Biodiesel is produced in many countries as an alternative to diesel fuel [21-23]. Petroleum-based fuels are negatively impacting the environment, releasing harmful gases like CO_2, HC, NO_x, and SO_x. As oxygenated fuels, ethanol and biodiesel are essential for lowering emissions of sulfur oxides, particulate matter, unburned hydrocarbons, poly-aromatics, and greenhouse gases. Because of their environmental benefits, they are very appealing substitutes for diesel fuel [24]

With the most excellent methyl ester output of 11% in the RUN-1 sample, this study offers new information on the transesterification method used to produce biodiesel from soybean oil. It suggests that biodiesel may be a better alternative to diesel fuel and provides a detailed analysis of fuel properties, including a cetane number of 68.4.

2. Methodology

This research focuses on the transesterification process used to produce and analyze biodiesel from soybean oil. The essential stages and experimental protocols are listed below to guarantee the experiment's repeatability and the precision of the biodiesel yield and property evaluations.

2.1 Collection and Preparation of Materials

Soybean oil, methanol, and sodium hydroxide (NaOH), which are needed to produce biodiesel, were gathered. Methanol is used to produce methyl esters, while sodium hydroxide catalyzes the transesterification process. The essential apparatus was glassware, an electric balance, measuring cylinders, a separatory funnel, and a hot plate with magnetic stirrer. All equipment used in the experiment was carefully cleaned with distilled water and dried to avoid any contaminants or moisture remained that would impede the transesterification process, possible side reactions that might have a detrimental impact on the yield and quality of biodiesel.

2.2 Transesterification Reaction Setup

Soybean oil was used as a catalyst in a transesterification reaction to react with methanol to produce biodiesel. This stage was essential for forming the byproduct glycerin and fatty acid methyl esters, or biodiesel. To assure precision and consistency in the manufacturing of biodiesel, the systematic process that follows was adhered to:

2.2.1 Preheating the Soybean Oil

Using a magnetic stirrer, the 250 mL of soybean oil was heated to 55 °C to maximize the reaction rate and ensure the oil was liquid for efficient mixing. The temperature was closely regulated to prevent overheating, which could deteriorate the oil.

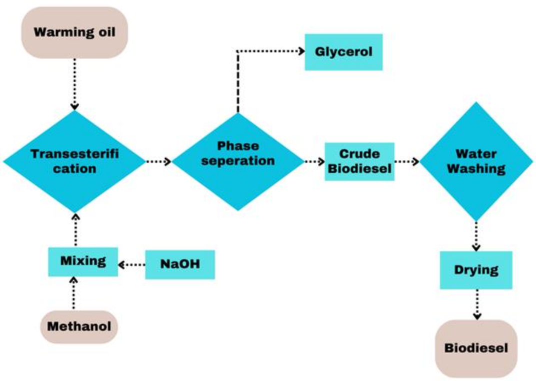


Fig.1. Flow chart of biodiesel production

2.2.2 Preparation of Sodium Methoxide Solution

1.4 g of NaOH and 50 mL of methanol (MeOH) were dissolved in a beaker to create sodium methoxide. The NaOH was thoroughly dissolved by constantly stirring this mixture with the magnetic stirrer. Sodium methoxide was used as the active reagent in the transesterification reaction to convert methyl esters from the oil's triglycerides. This preparatory step was crucial to creating the necessary reaction mixture.

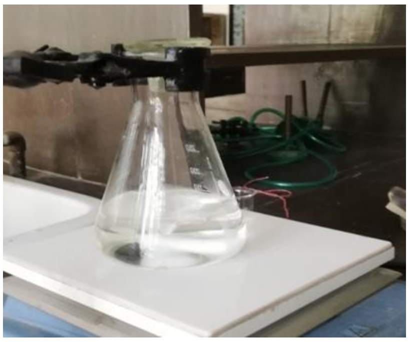


Fig. 2. Mixture of NaOH with methanol

Fig. 3. Warm-up oil

Fig. 4. Mixture of Methanol, NaOH, and vegetable oil

Fig.5. Finished Bio-Diesel production

Fig.6. Final product of Bio-Diesel

2.2.3 Initiation of the Reaction

The produced sodium methoxide solution was then mixed with the heated soybean oil. To ensure the reactants were mixed correctly, the mixture was shaken vigorously for five minutes. The reaction mixture was left to stand for overnight to facilitate full-phase separation. As a result of transesterification reaction, two different layers were formed such as the glycerin phase (lower layer) and the biodiesel phase (upper layer).

2.3 Separation and Analysis of Biodiesel

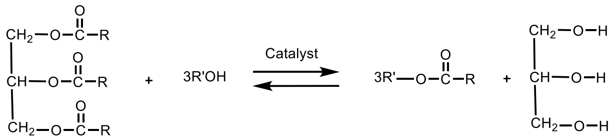
The two layers were evident after the 24-hour response time. A separatory funnel was used to properly separate the biodiesel (upper layer) from the glycerin to ensure that no glycerin remained in the biodiesel, which could impact its quality and quality. A measuring cylinder was used to measure the volume of biodiesel produced, and the percentage yield was computed by considering the starting oil consumption and finally analyzed the fuel qualities of the biodiesel samples thoroughly. Density, viscosity, fire point, and pour point were among the attributes investigated. The methyl ester composition of the fatty acids in the biodiesel samples was also identified and quantified using Gas Chromatography-Mass Spectrometry (GC-MS), with hexadecenoic acid methyl ester being the predominant component. The performance attributes of biodiesel, including the cetane number, flash point, and fire point, were also evaluated using a Fuel Analyzer. By comparing it with conventional diesel, these analyses clarify the combustion efficiency and safety of biodiesel. The RUN-1 sample yielded the highest methyl ester by using 60 mL methanol, 250 mL soybean oil, and 1.4 g NaOH. This outcome showed that temperature and reactant concentration were important reaction parameters affecting biodiesel production. GC-MS analysis revealed the maximum concentration of fatty acid methyl esters was 11% in RUN-1 sample, highlighting the material's potential as a fossil fuel substitute.

3. Chemical Reaction of Biodiesel

In the transesterification process utilized in your study, biodiesel, fat, or vegetable oils rich in triglycerides are mixed with methanol when a catalyst is present. Glycerine and fatty acid methyl esters (FAME) are the results of this process. Since soap synthesis reduces catalyst availability, the optimal efficiency requires a low percentage of free fatty acid (FFA) (less than 2%) and few non-saponifiable components [25]. This is particularly true for oils. It is recommended to use a 6:1 alcohol-to-oil ratio (a 100 % alcohol excess) for transesterification.
The reaction can be simplified as:

Triglyceride + 3 Methanol → 3 Methyl Ester (Biodiesel) + Glycerol.

$$C_3H_5(C_{18}H_{36}O_2)_3 + 3CH_3OH \xrightarrow{NaOH} 3C_{18}H_{36}OCH_3 + C_3H_8O_3$$

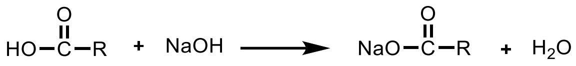


Scheme1: Reaction of Biodiesel Production

Despite its toxicity, methanol is favored because it is less expensive, easier to recover from, and has faster reaction rates. The process in biodiesel produces superior cetane numbers, high lubricity, and an energy content comparable to standard diesel fuels. However, because of solubility problems, a high alcohol percentage could make separating biodiesel from glycerin easier.

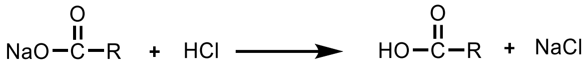
4. Separation of byproducts

The research uses purification and decantation methods to separate and purify the prepared biodiesel byproducts, including glycerin and fatty acid methyl esters (FAME). First, the reaction products, glycerin, and FAME, are separated using decantation, which divides the mixture into two phases based on their densities. Extra purification is necessary because FAME, the top phase, contains contaminants such as glycerides, alcohol, and catalyst residues. Washing the FAME with water and acidified water is part of this purification procedure to get rid of impurities. The FAME is guaranteed to meet biodiesel quality criteria by a final drying step. The article emphasizes how centrifugation can speed up the slow decantation process, which is the bottleneck. Glycerin, a byproduct, usually has little commercial use because of its impurities, which include soaps, free fatty acids, and methanol. Washing and pH changes are used to get rid of these contaminants. Crude glycerin's free fatty acids segregate after settling into different stages. Silicates or ion exchange resins are two more alternative purification methods the study emphasizes to increase product quality.



Fatty acid Soap

Scheme 2: Reaction of Soap Production



Scheme 3: Conversion of Soap into free fatty acid

This research, which goes into great depth about the separation of biodiesel products, emphasizes the vital role of purification in the effectiveness and quality compliance of biodiesel manufacturing by addressing both chemical and mechanical processes in product separation. One important thing is FAME. Significant factors include the challenges of decantation and the need to remove contaminants to meet the biodiesel requirements for clean glycerin. The research analyzes chemical and mechanical processes in product separation to highlight the critical role of purification in biodiesel synthesis's efficacy and quality compliance.

5. Result and Discussion

The transesterification reaction was carried out using vegetable oil, methanol and NaOH as a catalyst. To achieve the highest yield of biodiesel product the reaction is conducted four times, as shown in Table 1. All the products of biodiesel are determined using Gas Chromatography / Mass Spectrometry, presented in **Fig. 7**. Among the four products, the products from RUN-1 was found to have the highest yield of methyl ester. Three hours reaction time was sufficient to have good yield for methyl ester. The reaction temperature was also the most significant variable that affected the transesterification reaction. The molar ratio varied at 3:1 and 1:3 where the temperature was constant at 60 °C.

Table 1 Quantification of Biodiesel

Number of observations	Methanol (ml)	Soybean oil (ml)	NaOH (gm)	Biodiesel (ml)	By-product (ml)
Run-1	60	250	1.4	248	32
Run-2	60	250	1.4	247	70
Run-3	150	50	0.7	175	23
Run-4	300	100	1.4	302	102

GC-MS investigates the methyl ester composition in the production of biodiesel from vegetable oil. The following peaks depict the methyl esters:

Peak -1	Hexadecenoic acid, methyl ester($C_{17}H_{34}O_2$)
Peak-2	9, 12 Octadecadienoic acids, methyl ester ($C_{19}H_{34}O_2$)
Peak -3	9 Octadecenoic acid, methyl ester ($C_{19}H_{36}O_2$)
Peak -4	Octadecanoic acid, methyl ester ($C_{19}H_{38}O_2$)

5.1 GC-MS Spectra for RUN -1

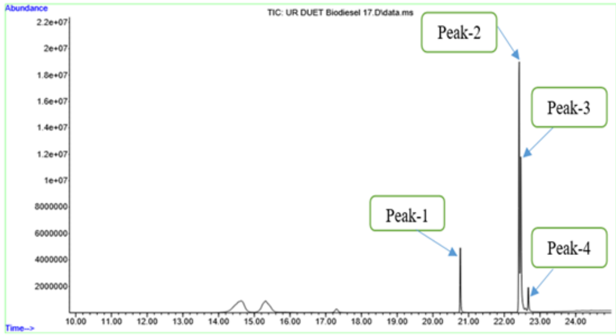


Fig.7. GC Mass Spectra of RUN-1

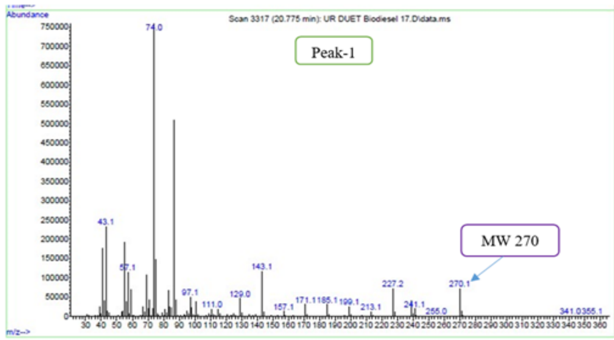


Fig.8. Peak-1

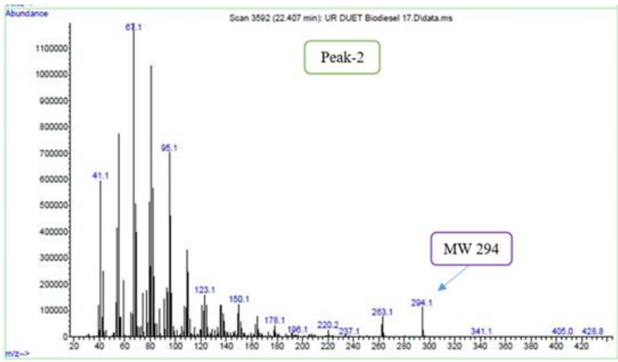


Fig.9. Peak-2

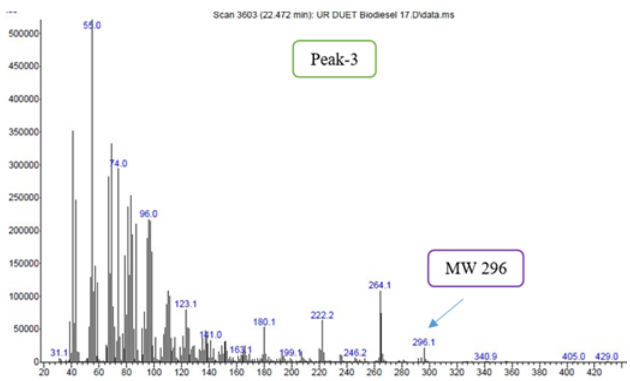


Fig.10. Peak-3

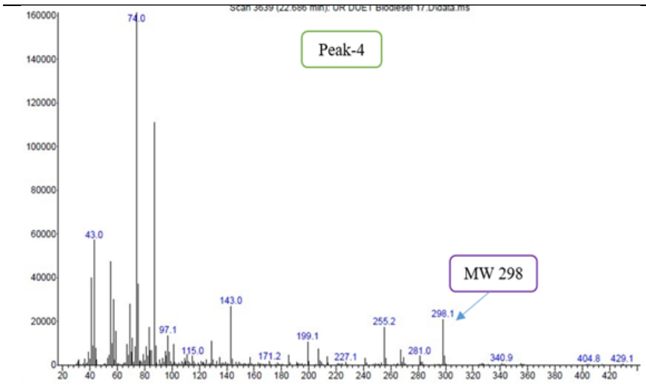


Fig.11. Peak-4

5.2 Analysis of Peak

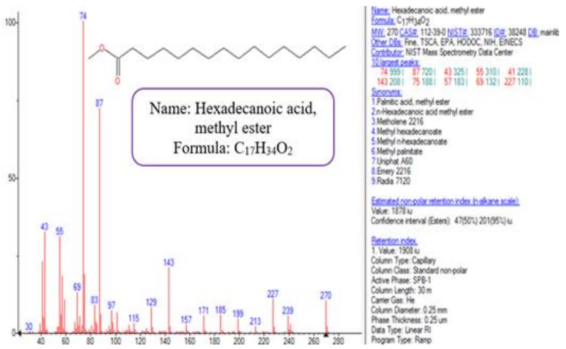


Fig.12. Analysis of Peak-1

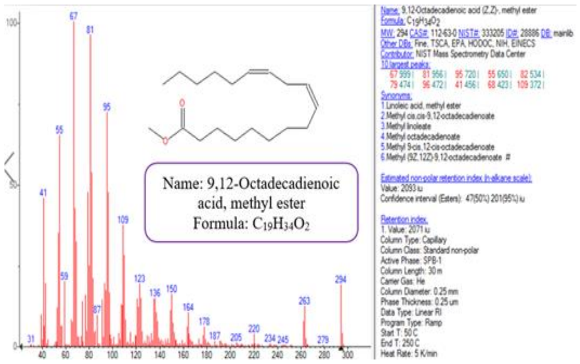


Fig.13. Analysis of Peak-2

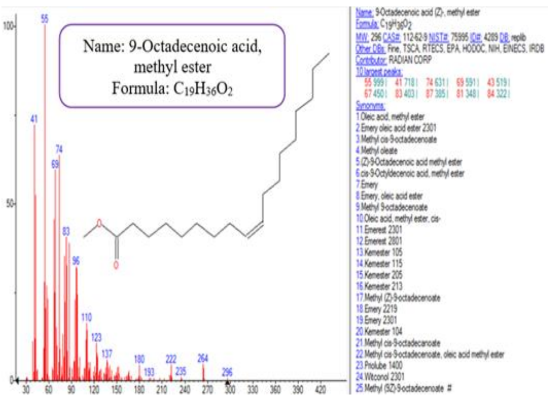


Fig.14. Analysis of Peak-3

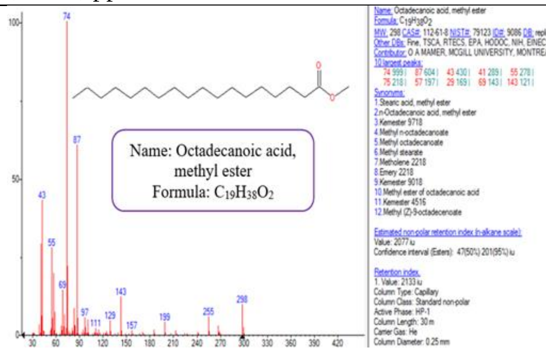


Fig.15. Analysis of Peak-4

5.3 GC-MS Spectra for Different RUN

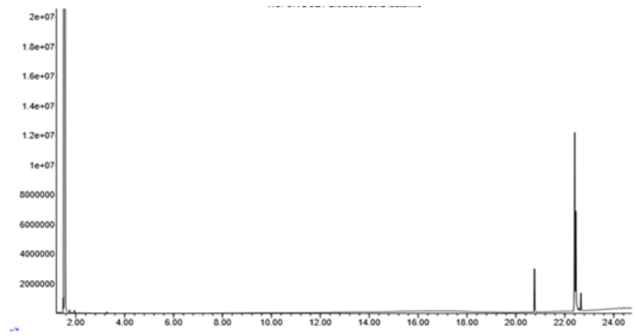


Fig.16. GC-MS Spectra for RUN-2

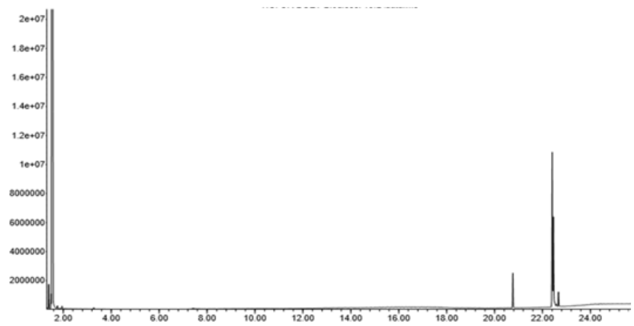


Fig.17. GC-MS Spectra for RUN-3

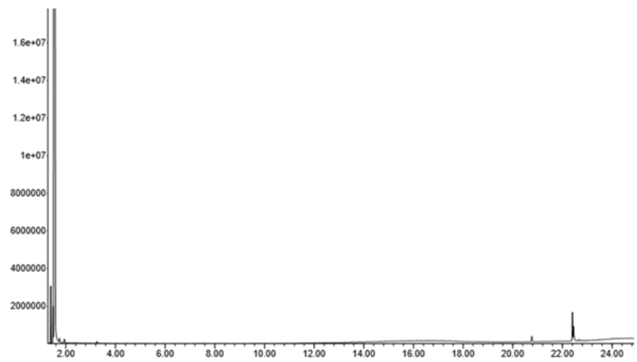


Fig.18. GC-MS Spectra for RUN-4

5.4 Chemical Name and its Molecular Formula

The percentages of methyl esters in the biodiesel were determined using Gas Chromatography/ Mass Spectrometry (GC-MS).

Table 2. The percentages of methyl esters present in the composition of biodiesel produced from vegetable oil.

Peak	Name	Molecular Formula	% of Area
1	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	11%
2	9,12-Octadecadienoic acid (Z, Z), methyl ester	$C_{19}H_{34}O_2$	47%
3	9-Octadecenoic acid (Z), methyl ester	$C_{19}H_{36}O_2$	37%
4	Octadecanoic acid, methyl ester	$C_{19}H_{38}O_2$	5%

From Table 2, we observed that the methyl esters present in the biodiesel are Octadecanoic acid, methyl ester ($C_{19}H_{38}O_2$), and Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$). Their percentages by composition are 5% and 11% respectively. Other esters also initiated in the biodiesel are 9,12-Octadecadienoic acid, methyl ester ($C_{19}H_{34}O_2$) and 9-Octadecenoic acid, methyl ester ($C_{19}H_{36}O_2$) with higher percentages of 47% and 37% that contain some unsaturation. So we can have exposed that Hexadecanoic acid, methyl ester($C_{17}H_{34}O_2$), was the predominant compound in the mixture, having the highest percentage of 11%. The fatty acid methyl ester profile is an essential factor for the determination of the suitability or any other feedstock for use in biodiesel fuel production.

5.5 GC-MS Pure Bio-diesel Vs Experimental GC-MS Spectra

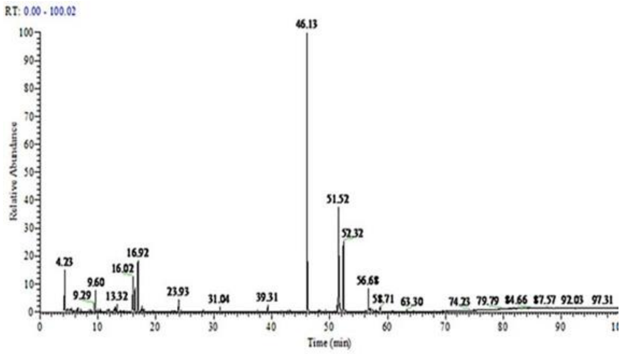


Fig.19. Pure Biodiesel Spectra from literature

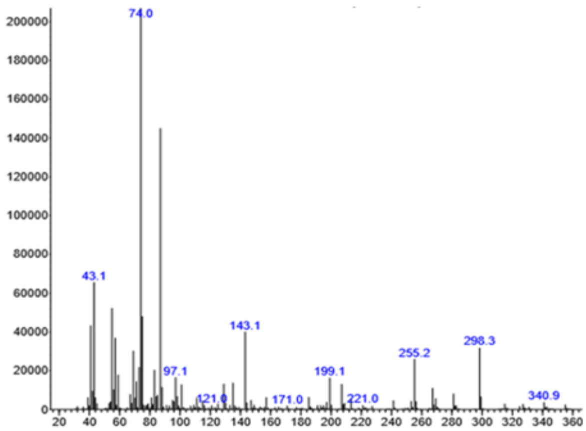


Fig.20. Experimental GC-MS Spectra

5.6 Result from Fuel Analyzer

Temperature 40 °C, Density at 40 °C 869.0 kg/m^3

Table 3. Data from fuel analyzer.

Method	Value	Unit	Av
FAME EN14078	31.57	V%	
FAME ASTM 7-30%	>40.09	V%	
Cetane Improver (EHN)	1904		
Cetane Number	68.4		98.405
Cetane Index	58.2		99.526
Total Aromatics	0	m%	98.747
PNA	0.61	m%	99.507
Total Olefines	0.00	m%	
IBP	217.3	°C	98.064
T10	205.8	°C	98.039
T65	<230	°C	
T85	<260	°C	
T90	>380	°C	96.392
T95	<300	°C	
FBP	356.8	°C	96.322
R250	39	%rec	99.962
R350	>100	%rec	98.383
CFPP	-6.1	°C	99.923
Kinematic Viscosity at 40^{0}C	0.00000341	m^2/s	96.139
Dynamic Viscosity at 40^{0}C	0.00300	Kg/m/s	96.141
T50	328.9	°C	96.345
FAME ASTM 0-7%	>7.02	V%	
Cetane Improver (IPN)	3	ppm	

The most important properties of fuel such as cetane number, cetane index, temperature, density, Kinematic Viscosity at 40 °C, and Dynamic Viscosity at 40 °C, are found in the Table 3. The cetane number and cetane index of the prepared biodiesel were comparatively higher than pure diesel. The temperature was almost identical, and the viscosity at 40 degrees was higher than diesel.

6. Conclusion

Vegetable oil is a reliable source of renewable biodiesel It has the prospective to fill-up partial energy demands in an eco-friendly manner. From the compositions of methyl ester in biodiesel produced from vegetable oil we observed the presence of Octadecanoic acid, methyl ester ($C_{19}H_{38}O_2$), and Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$) with a respective percentages of composition were 5% and 11%. Other esters obtained from the biodiesel were 9,12-Octadecadienoic acid, methyl ester ($C_{19}H_{34}O_2$) and 9-Octadecenoic acid, methyl ester ($C_{19}H_{36}O_2$) having percentages of 47% and 37% respectively. The results conclude that Hexadecanoic acid, methyl ester ($C_{17}H_{34}O_2$), was the major compound in the mixture that have the highest percentage of 11%. As a substitute for gasoline in internal combustion engines, the methyl ester profile of biodiesel demonstrates its viability and reliability.

References

1. Saifullah, A., A. Karim, and M.R. Karim, *Advancement of biodiesel in Bangladesh.* IOSR Journal of Engineering (IOSRJEN), 2016. **6**: p. 59-64.
2. You, Y.-D., et al., *Economic cost analysis of biodiesel production: case in soybean oil.* Energy & Fuels, 2008. **22**(1): p. 182-189.
3. Hoekman, S.K., et al., *Review of biodiesel composition, properties, and specifications.* Renewable and sustainable energy reviews, 2012. **16**(1): p. 143-169.
4. Kulkarni, M.G. and A.K. Dalai, *Waste cooking oil an economical source for biodiesel: a review.*

Industrial & engineering chemistry research, 2006. **45**(9): p. 2901-2913.
5. Usman, A., et al., *Fatty acid methyl esters composition of Trichilia emetica shell oil.* Am Sci Res J Eng Technol Sci, 2016. **21**(1): p. 83-90.
6. Noureddini, H. and D. Zhu, *Kinetics of transesterification of soybean oil.* Journal of the American Oil Chemists' Society, 1997. **74**: p. 1457-1463.
7. Demirbas, A. and A. Biodiesel, *A realistic fuel alternative for diesel engines.* London: Springer-Verlag. ISBN, 2008. **10**: p. 1846289947.
8. Kulišić, B., et al., *Impacts of biodiesel production on Croatian economy.* Energy Policy, 2007. **35**(12): p. 6036-6045.
9. Lee, D.-H., *Algal biodiesel economy and competition among bio-fuels.* Bioresource technology, 2011. **102**(1): p. 43-49.
10. Mukhopadhyay, K., X. Chen, and P. Thomassin, *Economy wide impacts of ethanol and biodiesel policy in Canada: An input–output analysis.* International Journal of Green Energy, 2017. **14**(4): p. 400-415.
11. Naylor, R.L. and M.M. Higgins, *The political economy of biodiesel in an era of low oil prices.* Renewable and Sustainable Energy Reviews, 2017. **77**: p. 695-705.
12. Xing, A., et al., *Life cycle assessment of economy for biodiesel.* Journal of Tsinghua University Science and Technology, 2010. **50**(6): p. 923-927.
13. Borugadda, V.B. and V.V. Goud, *Biodiesel production from renewable feedstocks: Status and opportunities.* Renewable and Sustainable Energy Reviews, 2012. **16**(7): p. 4763-4784.
14. Mahlia, T., et al., *Patent landscape review on biodiesel production: Technology updates.* Renewable and Sustainable Energy Reviews, 2020. **118**: p. 109526.
15. Hassan, M.H. and M.A. Kalam, *An overview of biofuel as a renewable energy source: development and challenges.* Procedia engineering, 2013. **56**: p. 39-53.
16. Kumar, P., M.P. Sharma, and G. Dwivedi, *Impact of ternary blends of biodiesel on diesel engine performance.* Egyptian Journal of Petroleum, 2016. **25**(2): p. 255-261.
17. Dhar, A. and A.K. Agarwal, *Effect of Karanja biodiesel blend on engine wear in a diesel engine.* Fuel, 2014. **134**: p. 81-89.
18. Habibullah, M., et al., *Potential of biodiesel as a renewable energy source in Bangladesh.* Renewable and Sustainable Energy Reviews, 2015. **50**: p. 819-834.
19. Statista. *Production of natural gas worldwide in 2023, by country*. 2024; Available from: https://www.statista.com/statistics/264101/world-natural-gas-production-by-country/.
20. *Gas reserve to last until 2031*. 2015; Available from: https://www.thedailystar.net/news/bangladesh/news/benazirs-immovable-properties-court-appoints-administrators-look-after-them-3628866.
21. Rahman, M.S., et al., *Present situation of renewable energy in Bangladesh: renewable energy resources existing in Bangladesh.* Global Journal of Researches in Engineering Electrical and Electronics Engineering, 2013. **13**(5): p. 1.
22. Reza, S.E., T.A. Nitol, and I. Abd-Al-Fattah, *Present scenario of renewable energy in Bangladesh and a proposed hybrid system to minimize power crisis in remote areas.* International journal of renewable energy research, 2012. **2**(2): p. 280-288.
23. Anam, K. and H. Bustam, *Power crisis & its solution through renewable energy in Bangladesh.* Cyber Journals: Multidisciplinary Journals in Science and Technology, Journal of Selected Areas in Renewable and Sustainable Energy (JRSE), 2011: p. 13-18.
24. Lin, L., et al., *Biodiesel production from crude rice bran oil and properties as fuel.* Applied energy, 2009. **86**(5): p. 681-688.
25. Poth, U., *Drying oils and related products.* Ullmann's Encyclopedia of Industrial Chemistry, 2000.