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Production and Characterization of Algal Biodiesel from *Spirulina Maxima*

Md. Atiqur Rahman [α] & Kamrun Nahar [σ]

Abstract- Biodiesel is renewable; reduce the greenhouse gas emission and potential as a substitute of fossil fuel. The aim of the current investigation is to produce biodiesel and compare physiochemical properties of spirulina maxima biodiesel (BD) with diesel fuel (DF). Soxlet apparatus was used to extract oil from algal body. Transesterification process was carried out to produce BD by adding potassium hydroxide (KOH) and methanol. Different properties of BD were determined. The physical properties includes density, viscosity, flash point, Higher Calorific Value (HCV), Cetane Number (CN), PH, moisture content, carbon residue, ash content, acid value, etc., and chemical properties include fatty acid composition; fourier transform infra-red (FTIR), elemental analysis. All the fuel properties were ASTM standard and close to those of DF. FTIR results revealed that BD was like diesel like hydrocarbon.

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I. Introduction

In the recent years, energy crisis has become acute around the world due to depletion of petroleum crude and increase global demand for fossil fuel. Due to these problem biodiesel from algae are the most promising source total substitution of fossil fuel due to presence of the hydrocarbon chain similar to DF. Fuels derived from green source for use in diesel engines are known as biodiesel. Biodiesel consists of the methyl ester of the fatty acid component of the triglyceride. Biodiesel has some special advantages when compared to diesel fuels [1].

- Biodiesel can be used in existing engines without any modifications.
- Biodiesel is made entirely from vegetable sources; it does not contain any sulfur, aromatic hydrocarbons, metals or crude oil residues.
- Biodiesel is an oxygenated fuel; emissions of carbon monoxide and soot tend to be reduced compared to conventional diesel fuel.
- Unlike fossil fuels, the use of biodiesel does not contribute to global warming as CO_2 emitted is once again absorbed by the plants grown for vegetable oil/biodiesel production. Thus CO_2 balance is maintained.
- Occupational safety and health administration classify biodiesel as a non-flammable liquid.
- The use of biodiesel can extend the life of diesel engines because it is more lubricating than petroleum diesel fuel.
- Biodiesel is produced from renewable vegetable oils/animal fats and hence improves fuel or energy security and economy independence.

There is a large number of algae like Chlorella sp. Cylindrotheca sp. Nitzschia sp. Schizochytrium sp. spirulina sp., etc grow all over the world. In this study spirulina maxima is used as a sample to analysis the investigation. Extraction of oil from powder algae several processes are existed. All of them solvent extraction by soxhlet apparatus is well known. In the current investigation soxhlet apparatus, method is used to produce algal oil and transesterification process for synthesizing the crude algal oil. A number of studies has evaluated the potential of using crude algal oil in commercial purpose is insufficient. The aim of this study was to evaluate the potential of using BD as an alternative fuel by determining some physicochemical properties and comparing with base DF.

II. Material and Method

a) *Collection and preparation of algal sample*

Spirullina maxima was collected from Science Lab (BCSIR) Dhaka, Bangladesh. The samples were cleaned in fresh water and dried in an oven at 70^{0}C [2].

The dried algal biomass was used for biodiesel production.

b) *Materials*

Methanol (99.9%), Diethyl ether, Methylene chloride, Potassium Hydroxide (KOH) (99.2%), n-Hexane (99%) etc. was purchased from the local market at Dhaka, Bangladesh.

c) *Preparation of biodiesel*

i. *Powdering*

The dried spirullina maxima was powdered by a mechanical crusher. The fine grained algal powder was collected after this process.

ii. *Extraction of algal oil (Soxlet Apparatus Method)*

Soxhlet apparatus is a chemical extraction method. *Spirullima maxima* algal powder (0.5 Kg) was fed to a soxhlet extraction fitted with a round bottom flask with condenser. Oil from algae is extracted through

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separated washing or percolation for 48 hours at 45^{0}C with the solvent system as 15 diethyl ether and 10% methylene chloride in n-hexane [3]. From this process 115ml of crude oil was extracted. Then extracted oil was heated to temperature via the rotary evaporator, so that the diethyl ether present in the crude oil was evaporated.

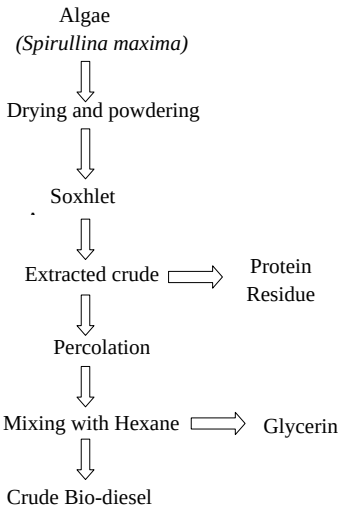

Fig.1: Flow chart of crude biodiesel production from algal powder

iii. *Mixing of Alcohol and Catalyst*

This typical process is mainly done by pouring 5 Liter of crude algal oil into the reactor for preliminary heating to the temperature of about (65-67^{0}C). In a separate container, 19gm of KOH (3.8 gm per liter of oil, got by 3.5 gm stoichiometric equivalent and 0.3 gm. for neutralizing free fatty acid) was dissolved in one liter methanol (200 mL per liter of oil) slowly[4]. This mixture was added continuously to the crude algal oil, and mixing was done properly by using of a stirrer. A typical Biodiesel production setup is shown in fig.4.

iv. *Transesterification Reaction*

Transesterification is the most common method to produce biodiesel. [5] It is the process of reducing viscosity of biodiesel by 75-85% of the original oil value. It is the process of reacting triglyceride with alcohol in presence of a catalyst to produce glyceride and fatty acid ester. Catalyst are usually used to improve the reaction rate, and the yield and alcohol are used to shift the equilibrium to the product side. [6] To complete a transesterification reaction stoichiometric ally a 3:1 molar ratio of alcohol to triglyceride is necessary. However, in actual practice, the ratio needs to be higher to drive the equilibrium to maximum ester yield. [7] Transesterification reaction is given below has been widely used to reduce the high viscosity of triglycerides. [8]

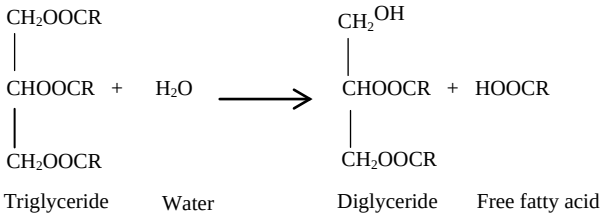

Fig 2 : Transesterification of triglyceride (Overall reaction)

v. *Separation*

After completing the reaction, Glycerin was setting down at the bottom and *spirulina maxima* biodiesel (BD) on the top. Process was continued until separation appears not to be advancing any more. The two product was separated by gravity using settling vessel. The bio diesel is drawn off at the top, and glycerin was drawn off at the bottom of the settling vessel.

vi. *Biodiesel Washing*

Biodiesel was poured off into a separate clean container for washing soap, salt or free fatty acid. Hot water (110^{0}C) was added to the methyl ester. It was stirred lightly and then allowed to settle down. After that the water was drained out from the bottom. The warm water was heated in the main reactor itself. Process was repeated until raising the value of the biodiesel P^H level of 6-7, and no soap bubbles appeared in it.

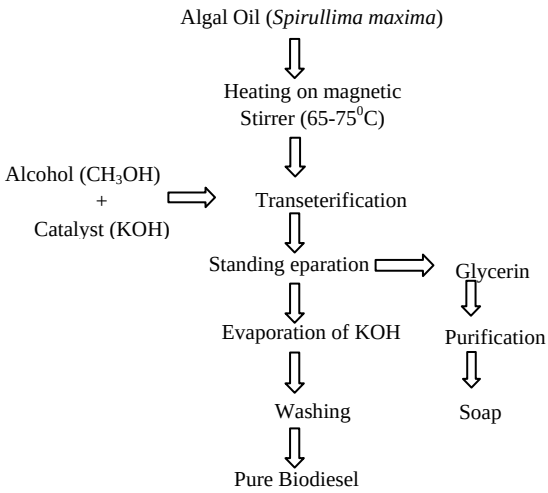

Fig 3 : Flow Chart of Biodiesel Production

The biodiesel was cloudy so it was slowly heated to evaporate out the water. About 3.7 liter of biodiesel were produced for an input quantity of 5 liter of crude oil.

d) Characterization of biodiesel

i. *Physicochemical properties*

Physical properties (Density, Viscosity, Flash point, Pour point, Higher calorific value (HCV), Cetane

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Number (CN), P^H, Moisture content, Carbon residue, Ash content, Acid value, etc.) and chemical properties (Fatty acid composition, FTIR, elemental analysis) were analyzed.

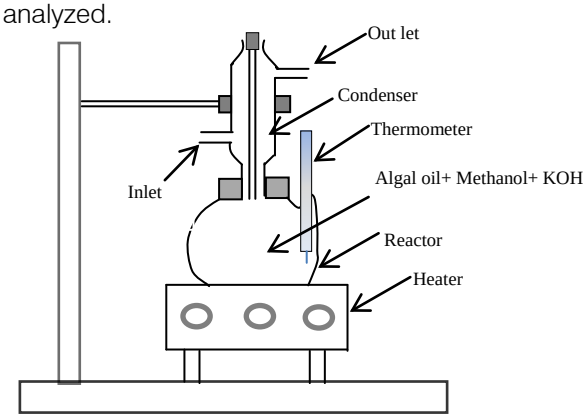

Fig 4 : Biodiesel Production

Fig 5 (a) : Algae

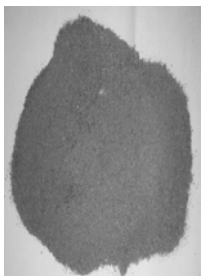

Fig 5 (b) : Grained powder algae

Fig 5 (c) : Extracted crude biodiesel

Fig 5 (d) : Biodiesel

ii. *Fatty Acid Composition*

Fatty Acid Composition was analyzed by Gas Chromatography (GC) (CP-3800 GC varian, USA) equipped with 30 m DB-WAX (J&W, Agilent) capillary column (0.25 mm of internal diameter and 0.25 m of film thickness). Detector (Xame ionization) and Injector (split 1:100) temperatures were kept constant at 250°C. The oven temperature program started at 180°C for 5 min, increased at 4°C min. until 220°C, and kept constant at this temperature for 25 min.

iii. *Fourier Transformed Infra-Red (FTIR) Spectroscopy analysis*

FTIR analysis gives an idea about the suitability of algal oil as Diesel Fuel (DF). It provides the amount of incident spectrum absorbed (percentage) by algal oil which identifies the basic compositional groups along the wave numbers 4000 to 500 per cm.

iv. *Elemental Analysis*

Elemental Analysis of BD in terms of carbon, hydrogen, oxygen, and sulphur (CHNOS) content is important in order to make a necessary material balance of each component. The composition of the algal oil was determined using an elemental analyzer of Model EA1108. Oxygen was calculated by difference. The analysis of the oil was conducted at Analytical Research Division of BCSIR, Dhaka.

III. Result & Discussion

a) Fuel Properties of algal Biodiesel

Biodiesel could be used as an alternative fuel for diesel engine only if its physical and chemical properties confirm to the international standards specification. BD was characterized according to ASTM standard. The physiochemical properties of *spirullina maxima* algal biodiesel is presented in table-1. The physiochemical properties of BD are similar to DF.

b) Fatty Acid Composition

Spirulina maxima biodiesel (150 gm) was taken to determine the fatty acid composition using the borum trioxide method according to ENISO 5509 standard. Fatty Acid composition was calculated as a percentage of the total fatty acids presents in the sample, determined from peak areas. The fatty acids composition of BD is presented in table 2. The BD consisted of carbon chain length between16 to 20. The higher concentration fatty acids were palmitic, Sterice and Linolenic.

c) Elemental analysis

Elemental analysis is shown in table-3. BD has lower carbon content and at the same time having a larger amount of oxygen, which justifies the lower heating value compared to DF.

Table 1 : Property Comparison of BD and DF

Sl No	Properties	ASTM Method	BD	DF
1	Density at 40°C (g/cm^3)	D1298	0.8334	0.86
2	Viscosity (mm^2/s)	D445	4.47	2.83
3	Cetane Number (CN)	D613	55	50
4	HCV (MJ/kg)	D240	38.43	44.5
5	Flash Point (°C)	D93	178	90
6	P^H	-	7	6.54
7	Moisture Content (%)	D6751	0.04	< 0.03
8	Carbon Residue (%)	D524	0.008	0.01
9	Ash Content (%)	D482	Nil	< 0.02
10	Acid value (mgKOH/g)	D975	0.34	0.05

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Table 2 : Fatty acid composition of BD

Fattyacid	Structure	% (w/w)
Palmitic	C16:0	38.85
Palmitoleic	C16:1	9.8
Stearic	C18:0	16.41
Oleic	C 18:1	2.3
Linoleic	C 18:2	7.2
Linolenic	C 18:3	15.1
Arachidic	C 20:1	9.7
Others	-	0.64

Table 3 : Elemental analysis of BD and DF

Properties	BD (%wt)	DF (%wt)
C	67.91	86.18
H	10.69	13.8
O	19.86	< 0.01
N	1.54	-
S	< 0.00	0.034

d) FTIR

The target of the current investigation is to examine suitability of BD as an alternative fuel. Considering the above fact, FTIR analysis of BD was performed. FTIR spectrum of BD and DF was shown in fig 6 (a) and 6 (b).

Neat DF			Neat BD		
Frequency range (cm^{-1})	Bond types	Family	Frequency range (cm^{-1})	Bond types	Family
2923.9-2854.5	C-H stretching	Alkanes	2922.1-2852.7	C-H stretching	Alkanes
1745.3	C=0	Aldehyde/ketone	1737.86	C=O	Aldehyde/ketone
1458.3	C-H bending	Alkanes	1456.26	C-H bending	Alkanes
1377.1	C-X	Fluoride	1066.64	C-O	Alcohol
723.3	=C-H bend	Alkanes	719.45	aromatic C-H	benzene

Table 4 : Frequency comparison table of BD and DF

Table 4 represents the functional group compositional analysis for BD and DF. For DF, the strong absorption frequency 2923.9 cm^{-1}, 2854.5 $cm^{-1}$and 723.3 cm^{-1} represent C-H stretching, which indicate the presence of an alkane and appearance is very strong. The absorbance peaks 1745.3 cm^{-1} and 1377.1 cm^{-1} represented the C=O (Aldehyde/ketone) and C-X (Fluoride) respectively. For BD, strong absorbance peaks 2922.16 cm^{-1}, 2852.72 cm^{-1} and 719.45 cm^{-1} are the C-H stretching, which represent the presence of the alkane group. The absorbance peaks 1737.86 cm^{-1} and 1066.64 cm-1 represent the types of bonds specifically aldehyde/ketones and alcohol respectively. The frequency 719.45 cm^{-1} indicate the presence of benzene. From the FTIR graph, it is seen that major transmittance spectrums peaks both BD and DF are alkanes, and their bond type is very strong.

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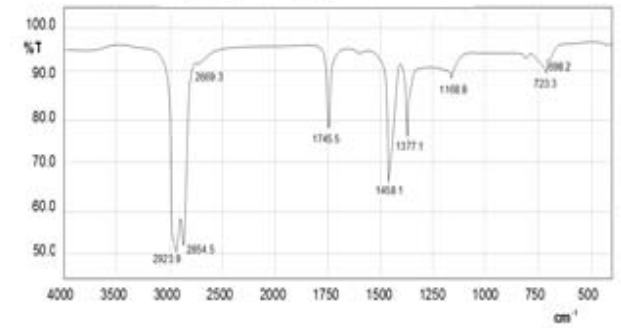
Fig 6(a): FTIR graph for DF



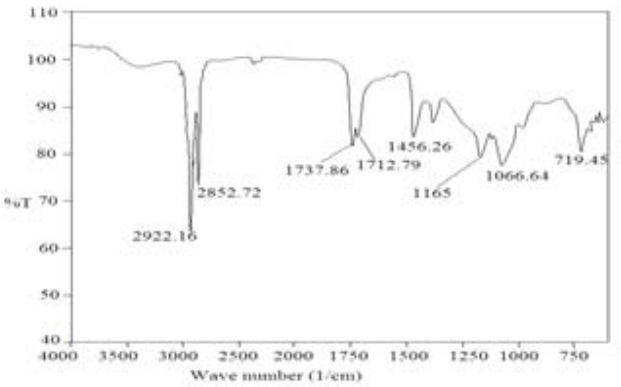


Fig 6(b): FTIR graph for BD

According to the above discussion, clearly both BD and DF are saturated hydrocarbon. The presence of hydrocarbon groups C-H indicates that the liquid has a potential to be used as fuels.[9]

IV. Conclusion

The results of the current investigation are summarized as bellows where pure diesel was used as base fuel for comparing the parameters -

1) The viscosity after the transesterification process was 4.47mm^2/s at 40^{0}C which is 63.3% higher than DF. The viscosity of BD highly decreased after the transesterification process by 70%.
2) The flash point of BD was measured as 178^{0}C. The higher value of flash point decrease of risk of fire and potential safe for storage as compare to DF.
3) CN of BD was found to be 55 whereas DF was 50; higher CN of BD gives higher ignition quality.
4) From FITR graph, the major transmittance spectrums of algal oil peaks was alkanes which indicates that the liquid has a potential to be used as fuels.
5) Palmitic acid percentage by 38.85 is the highest fatty acid composition in BD.
6) Carbon residence of BD is 0.008%, which is suitable for diesel engine from leakage of nozzle, corrosion, cracking of composition.
7) BD has lower carbon content but having a large amount of oxygen compared to DF, which both justify the lower heating value of algal oil.

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