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A breakthrough in Production of Biodiesel from Linseed Oil by Conventional Hotplate Method

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***Abstract*-** Gradual depletion and successive enhancement of demand have led to find a new feedstock for biodiesel production. On this aspect, Biodiesel from linseed oil can play an important role as an alternative source of energy for automobile applications due to favorable climatic and soil conditions for the production of linseed crop. In the present work an attempt has been made to generate biodiesel from linseed oil by hotplate method using base catalyzed trans esterification process which was applied to lower the viscosity at 4.19 cSt of the oil and the reaction was affected by the amount of catalysts, reaction time and temperature, molar ratio of alcohol to oil. This study has revealed the maximum yield of biodiesel up to 94.7% with oil to methanol ratio 1:8, 1 wt% NaOH catalyst. The calorific value of biodiesel was 9543.25 Kcal/Kg. FT-IR and NMR spectrum of linseed oil and its biodiesel samples are analyzed which confirms the conversion of oil to biodiesel and the fuel characteristics have been investigated according to American Society for Testing and Materials and significant improvements were observed and comparable to pure diesel which could be a trustful pathway to commit our present and future fuel demand in the world.

Keywords: Biodiesel; Linseed oil; Conventional heating method; Trans esterification; FTIR.

1. INTRODUCTION

Biodiesel is a green renewable and potentially unlimited source of energy. It is an alternative eco–friendly biofuel for diesel engines produced from renewable biological sources, such as vegetable oils and animal fats [1]. In recent year, biodiesel production processes are based on either conventional or supercritical heating methods. The available methods that are used to produce biodiesel: I) Pyrolysis, II) Micro–emulsions, III) Dilution, and IV) Trans esterification of oils to esters [2]–[6]. The most common way to produce biodiesel is by trans esterification that has proven to be the simplest and the most economical method to produce biodiesel, with similar physical characteristics of fossil diesel and little or no deposit formation when used in diesel engines. Transesterification of oils from linseed oil is that to simply reduce the viscosity of the oils. Transesterification is a process in which an alcohol (methanol or ethanol) in the presence of a catalyst (acid or alkali or enzyme) is used to chemically breakdown of vegetable oils or animal fats into methyl or ethyl esters of the renewable fuel. The transesterification process is consisting of three consecutive and reversible reactions, in which di and monoglycerides are found as intermediates. However, in this process excess amount of alcohol is used to increase the yields of the alkyl esters by shifting the equilibrium towards the formation of esters as well as to allow its phase separation from the glycerol formed as a byproduct. Biodiesel by transesterification of organic feedstock can be obtained by the following methods: (I) conventional heating with acid, base catalysts and co–solvents [7]–[16]; (II) sub– and super–critical methanol conditions with co–solvents and without catalyst [17]–[22]; (III) enzymatic method using lipases [23]–[28]; and (IV) microwave irradiation with acid, base and heterogeneous catalysts [29]–[32].

Linseed is cultivated all over the world in over 2.6 million ha. [35]. There are two types of linseeds, brown and golden seeds. Moderate temperate zone is suitable for growing linseed and it might be an efficient feedstock for biodiesel production with sufficiently high oil content (35–45%) [36] in Bangladesh as well as in the whole world. As an alternative renewable energy, biodiesel from linseed oil has bright future in Bangladesh due to available supply of raw materials. Although biodiesel production from biological source is still an infancy condition in Bangladesh, its prospect is promising. Biodiesel from linseed oil can play an important role in Bangladesh as an alternative diesel fuel. Climatic and soil condition of Bangladesh is suitable for the production of linseed crop. Therefore, linseed was chosen for biodiesel production and collected brown seeds from local region.

In the present study, linseed oil has been taken as a potential and alternative source of energy. The aim of

this work is to extract oil from linseed oil by mechanical press, optimization of reaction parameters of biodiesel production by transesterification method using base catalyst by hot plate method and evaluate the physicochemical properties of this biodiesel.

2. EXPERIMENTAL

2.1. Materials

The chemicals-methanol (99.8%), sodium hydroxide pellets (≥97%), potassium hydroxide pellets (>84%), phenolphthalein solution (pH 8.2-9.8), sulfuric acid (98%), acetone (≥99%), 2-propanol (≥99.0%), oxalic acid (99%) etc. were purchased from Merck chemicals limited, India except methanol (Merck chemicals limited, Germany). All these chemicals were analytical reagent grade and used without further purification. The crude oil sample was extracted from linseed and stored in clean bottles for the biodiesel production.

2.2. Hot plate and magnetic stirrer:

Model: JSHS- 180, manufacturer:JSR, made in Korea. It has heating and stirring control system and used for heating and stirring purposes.

2.3. Seed collection and storage

Brown linseed was collected from our local region of Bangladesh. Sand, gum and various foreign materials were removed from the seeds and dried. The dirt free seeds were stored at room temperature for about 9 months. The samples were analyzed before storage and every three months interval for oil and free fatty acid content during the storage period.

2.4. Extraction of oil

After storage, the rest of the seeds were extracted by mechanical press. A cylinderical, elcetrically operated mechanical press was used where continuous rotation of the press allows raising sufficient pressure for the extraction of oil. Oil drainage nozzles are located at the bottom side of the expeller. At first the linseed were fed into the cylindrical face of the expeller. The handle of the press was moved manually before the switched on. After oil extraction it was kept for couple of days to settle down the mud like particles. At this time, the oil was kept under sunlight to remove bad odor and microorganism if present and at last the oil was filtered at laboratory level. The oil content of linseed was found 37.5 % (w/w).

2.5.METHODS

2.5.1. Analytical traetment of raw oil

Which includes determination of varous properties of raw oil including:

- Determination of FFA value using 2-propanol as a solvent
- Determination of acid value:
- Determination of water content:

2.6.1. Hot plate and magnetic stirrer assisted process:

Almost all biodiesel is produced using base catalyzed trans esterification as it is the most economical process, requiring only low temperatures and pressures while producing almost 98% conversion yield. The reaction was carried out in a 500ml three neck flask under reflux at 60-65˚C with vigorous stirring. The amount of raw material was 50 ml linseed oil and oil to methanol ratio was 1:5, 1:6, 1:8, 1weight % NaOH catalyst was used. At first required amount of catalyst was dissolved in required amount of methanol under magnetic stirring. Then crude linseed oil was slowly added to the mixer and heated at desired temperature. The temparature was monitered by thermometer inserted in the flask. For differrent ratio the reaction time was also different.When the reaction was completed,two visible layer were appeared. After separation the product was washed and dried to measure its kinematic viscosity.

2.6.2.Separation of crude biodiesel:

After completing the biodiesel production reaction, it is essential to separate biodiesel from the unwanted layer of catalyst, glycerine and mineral acids. The phase separation method for all production procedure were same.So, the seperation process is describing combiningly.After cooling the mixed product, it was transferred to the separating funnel.The product was then allowedto settle overnight to produce two distinct liquid phases: crudeester phase at the top and glycerol phase at the bottom.

2.7.4.Washing of crude biodiesel:

Due to improper reaction, very often unreacted triglyceride, diglyceride, mono glyceride and unconverted FFA exists in crude biodiesel. So, Washing is important to get pure biodiesel.At first, hot water was added with thecrude biodiesel and shaken properly. Then the mixer was allowed to stand for phase separation. After settling, a milky water layerwas appeared with impuritieswhich was drained off from the separating funnel. Thus several wash was done until the milky water layer turned into clear. A biker, properly clean with acetone was taken o keep washed biodisel. Then the washed biodiesel was dried in a drier box at 100-105˚C for half an hour. Finally various properties of produced biodiesel were measured.

3. RESULT AND DISCUSSION

3.1.1. Effect of seed storage duration on oil content:

From the results of about 9 months of storage in which the oil analyses of the seed samples were carried out, it

can be inferred that, there is a significant effect of storage time on seed oil content of linseeds (Figure-3.1). The oil content of the seeds varied from maximum of 37.5% before seed storage to minimum (after 9 months of storage) of 30.8%, respectively. The lowest oil percentage was recorded from the seed samples that stored longer.

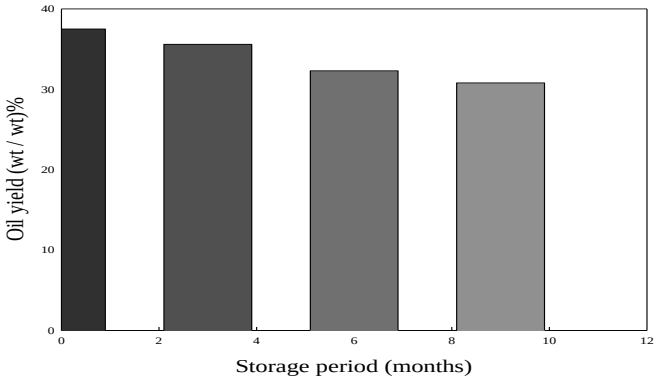

Figure 3.1: Effect of seed storage period on oil yield

As the research does not further on the reason of decreasing oil content, but from the literature I have studied that, high moisture content [37] as well as the higher oxygen availability tends to reduce the oil content with the length of storage time. Also, the metabolism of seeds during storage to provide energy for its physiological activities could be another reason of the seed oil reduction during long storage duration. So, it is expected that one of these factors could be responsible for decreasing oil content of seed.

3.1.2. Effect of seed storage duration on fatty acid content of oil:

In my study, the profile of the fatty acid content of linseeds which was high in linolenic acid was significantly influenced by storage time (Figure-3.2). The free fatty acid content of the seed oil increased linearly over the storage period. The increase ranged from 3.79% to 45.1 % for the time before storage to the last month of storage respectively.

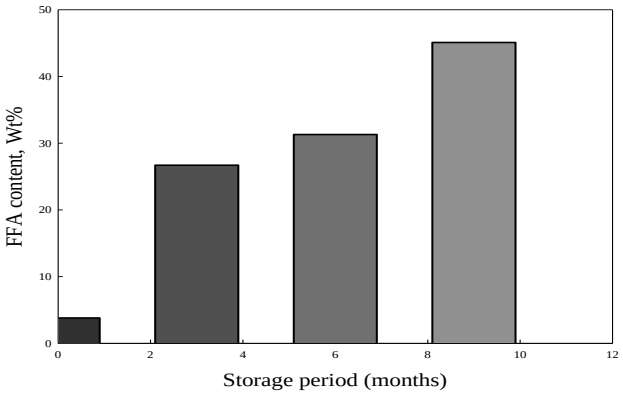

Figure 3.2: Effect of seed storage period on FFA content of oil

Free fatty acids are increased due to the hydrolysis of triglycerides [38] in the presence of moisture and oxygen and the improper handling and storage of the seeds before the oil extraction process [39]. These could affect the FFA content of linseed with the change of seed storage period.

3.1.3. Effect of reaction time:

Base catalyzed transesterification reaction was conducted at optimum molar ratio of oil to methanol, fixed catalyst concentration and optimum temperature.
In hot plate assisted process the molar ratio was (1:8) and reaction temperature (65℃). The sample under different time interval was taken and the results are depicted below.

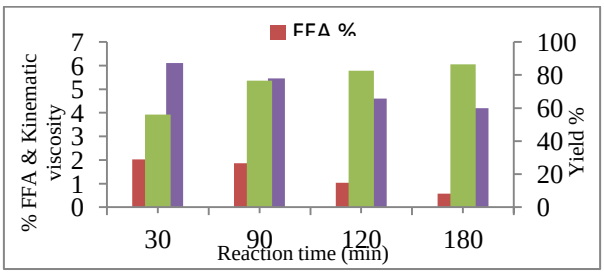

Figure 3.3: Effect of reaction temperature on % FFA, Kinematic viscosity and yield %

In order to achieve perfect contact between the reagents and the oil during transesterification, they must be stirred well at constant rate. In figure-3.3 it was observed that, the % of conversion of oil into methyl ester slightly increased with increased of reaction duration. Results obtained from the experiment concluded that, about 180 min of reaction was sufficient for the completion of the transesterification which reduces the viscosity (4.19 cSt) and % FFA (0.058%). The % of conversion was 86.5%. But with the further increase in time the reaction will move backward.

3.1.4. Effect of reaction temperature:

It was observed that increasing the reaction temperature, had a favorable influence on the yield of ester conversion. In the alkali (NaOH) transesterification reaction, the temperature was maintained at different range. The boiling point of methanol is 64˚ C. But at higher temperature, alcohol will burn and will provide poor yield. As, the temperature increased there was an increased in FFA, % yield and kinematic viscosity.

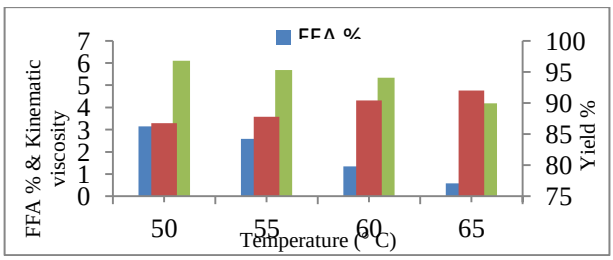

Figure 3.4: Effect of reaction temperature on % FFA, Kinematic viscosity and yield %

From the figure-3.3and 3.4 it was found that, the reaction

was conducted close to the boiling point temperature of methanol at atmospheric pressure. At room temperature the conversion efficiency was noted to be very low (about 10% only) even after 2 h of stirring. With the increased in temperature the conversion occurred at a faster rate. From the above figure it was found that, the optimum temperature for the both process was 65℃.) Researchers achieved better results at temperatures above 50℃ and up to 70℃ while using linseed oil and brassica carinata oil [40]. At 65˚C temperature there was a gradual decreased in % FFA (0.580%), kinematic viscosity (4.19 cSt) and the yield was 92% for hot plate process. But, at further increase in temperature, there will be a negative effect on the conversion [41] with huge loss of methanol.

Table 3.1: Optimum condition for hot plate process in transesterification reaction.

Reaction parameter	Optimum condition
Oil:Methanol	1:8
Catalyst weight	1 %
Temperature	65˚C
Reaction time	180 min

3.4.1.1. Physico-chemical properties of linseed oil methyl ester by hotplate/conventional heating:

The preparation method of linseed biodiesel through base catalyst by hot plate and magnetic stirrer was discussed in the previous chapter. Various properties of produced biodiesel (methyl ester) were measured and the results are given in table 3.2.

Table 3.2: Comparison of the physico-chemical properties of linseed oil methyl ester from hot plate with commercial diesel.

Property	Commercial diesel	Experimental value of produced biodiesel			
		1:4	1:5	1:6	1:8
Pour point,˚ C	-2	-5.8	-5	-7.5	-8
Flash point,˚ C	70	50	52.5	48	43
Fire point,˚ C	-	58	54	50	46.5
Density, gm/cc(15˚ C)	0.8445	0.893	0.880	0.890	0.873
Kinematic viscosity, cSt (40˚ C)	6.06	6.1712	5.852	5.2136	4.191
Calorific value, Kcal/Kg	-	8842.53	9475.83	8641.20	9543.25
Ash content, %	-	0.2505	0.240	0.210	0.100
Moisture content, %	Zero	Nil	Nil	Nil	Nil
Cetane number	51	46	49	52.20	56.03

3.4.1.4 FT-IR and ^{1}H-NMR ^{of} linseed biodiesel:

Fourier transform infrared (FTIR) spectrophotometer was employed to determine the chemical structure of linseed biodiesel and to ensure the conversion of oil into biodiesel. The FT-IR spectra of biodiesel (methyl ester) produced by hot plate assisted process is shown in 3.5.

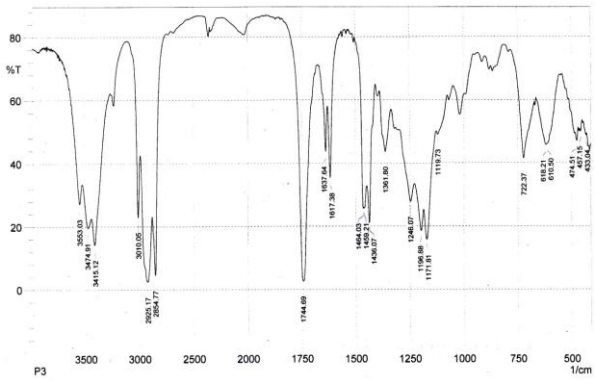

Figure 3.5: FT-IR spectrum of linseed biodiesel by hot plate heating process.

From the figure- 3.5 it was found that, the esters have characteristically strong absorption bands arising from carbonyl (C=O) group around 1744.69 cm^{-1}. The disappearance of peaks at 1068.61cm^{-1} from the spectrum of oil and appearance of new bands in the figure- 3.5 at 1119.73 cm^{-1} indicated the conversion of oil into biodiesel. There was no peak at 1597-1344 cm^{-1}, which indicated that the product was washed sample and also the peak at 1436.07 cm^{-1} in the figure, proved the presence of methyl ester [42].

^{1}H-NMR is a versatile spectroscopy method that has become one of the most powerful techniques to elucidate the structure of chemical compounds.

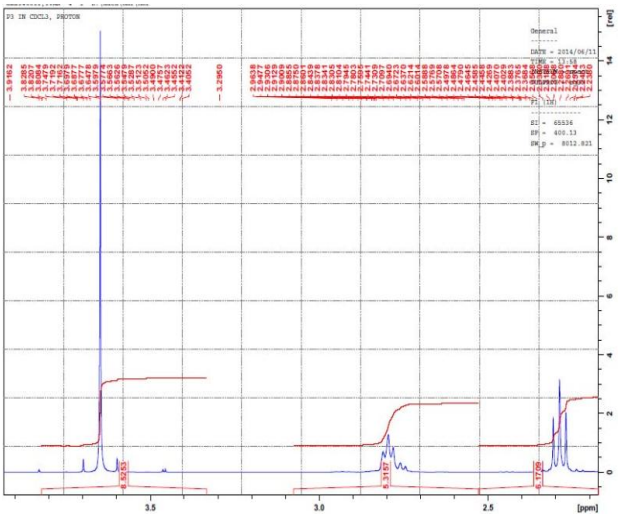

Figure 3.6: H-NMR spectra of linseed biodiesel by

microwave heating process.

Here, ^{1}H-NMR spectroscopy of linseed biodiesel was done to investigate the presence of triglyceride backbone in the sample. In figure-3.6, the protons of the methyl ester moiety and a carbonyl methylene groups are assigned at approximately 3.7 ppm and 2.3 ppm respectively.

4. CONCLUSION

Linseed oil was extracted from linseed quite effectively by mechanical press method and maximum yield of oil was 37.5 wt%. Base catalyzed transesterification method was done using conventional heating method for the production of biodiesel and compared it to commercial diesel which was found to be a promising alternative fuel source. Hot plate heating system with oil to methanol molar ratio 1:8 showed good results point out low kinematic viscosity and high yield. ^{1}H NMR and FTIR spectra of raw oil and biodiesel samples were analyzed which confirmed the conversion of raw oil into biodiesel. Biodiesel properties were evaluated by standard ASTM methods and a significant reduction in viscosity and acid value were found. The properties, such as specific gravity, viscosity, flash point, pour point, cetane number and calorific value were comparable to petro–diesel.

6. ACKOWLEDGEMENT

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8. NOMENCLATURE

Symbol	Meaning	Unit
T	Temperature	(K)
FFA	Free fatty acid content	%
ϑ	kinematic viscosity	mm^2/s