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Production of biodiesel from neem seed oil

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Abstract

Neem oil was extracted from neem seeds by mechanical extraction method. Yield of oil was 21.32%. The physicochemical properties of the extracted oil were studied in detail. The oil corresponds to diesel except acid value (14.21%) and sulphur content. Acid esterification was performed to reduce the acid value which was followed by transesterification to produce biodiesel. The conditions of the transesterification of the oil were optimized and were found to be 20% methanol and 1.0% NaOH at 60 °C for 90 min. The optimum yield of biodiesel was 98 %. Finally, the performance study in a diesel engine was conducted with diesel and biodiesel blends. The brake thermal efficiency for 5% blend of biodiesel was 16.67% for brake power of 0.79 KW.

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Introduction

Energy has been the synonym of economic development of any country in the current world. But the current fossil fuel based economy suffers from the issue of sustainability because of environmental impacts, economic dependence and energy security. The production and consumption of fossil fuels have raised the concentration of CO_2 in the atmosphere to cause a severe impact on the environment (Westermann *et al.*, 2007). At present 86% of the world energy consumption and almost 100% of the energy needed in the transportation sector are met by fossil fuels (Dorian *et al.*, 2006). One-fifth of the global CO_2 emissions are created by transport sector (Goldemberg, 2008), which accounts for 60% of global oil consumption (Anonymous, 2008). Around the world there were about 806 million cars and light trucks on the road in 2007 (Banik *et al.,* 2015). These numbers are projected to increase by 1.3 billion by 2030 and to 2 billion vehicles by 2050 (Banik *et al.,* 2015). These growths will affect the stability of ecosystems and global climate as well as global oil reserves (Balat, 2009). The biofuels of recent days can resolve the issues of sustainable development, energy security and reduction of green house gas emissions (Sylvester *et al.*, 2013). As an alternative to petroleum based transportation fuels, biodiesel is a suitable renewable substitute for petroleum based diesel. Biodiesel is a methyl or ethyl ester made from renewable biological sources such as vegetable oils (both edible and non edible), recycled waste vegetable oils and animal fats (Wilson, 2010). Biodiesel reduces net carbon-dioxide emissions by 78% on a lifecycle basis when compared to conventional diesel fuel (Carvalho *et al.*, 2011). Emissions such as total hydro carbons and CO are usually found to be significantly low with biodiesel as compared to petroleum diesel. This may be due to more complete combustion caused by the increased oxygen content in the flame coming from the biodiesel molecule (Hemant *et al.,* 2011). The annual demand of diesel fuel in Bangladesh is increasing tremendously.

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To meet the growing demand, we are totally dependent on foreign countries and large amount of foreign currencies are spent to import the diesel fuel. If biodiesel is used to some extent in place of diesel fuel, a vast amount of foreign currencies will be saved. Hence, we pointed our eyes on the production of biodiesel from non edible neem seed oil. Literature shows that the yield of oil from neem seed is about 15.4%-24.5% (Kaura *et al.*, 1998). Neem plant is available in many parts of the world including Bangladesh and is very cheap compared to other sources. The non-edible renewable neem seed oil can play a vital role as a substitute to diesel fuel. The climate and soil condition of Bangladesh is also suitable for the cultivation of this plant. If the developed process is scaled up to commercial levels, then excellent business opportunity will be offered by the biodiesel and this could be a major step towards the creation of an eco friendly transportation fuel. By increasing neem tree plantation in Bangladesh, we can meet our demand. Finally, the by-product (glycerin) of trans esterification can also be used in the soap industry. The present investigation includes: preparation of biodiesel from neem seed oil, optimization of different parameters for maximum biodiesel production, determination of properties of neem seed oil and prepared biodiesel, comparison of the fuel properties of conventional diesel with prepared biodiesel and performance study of the biodiesel in diesel engine.

Materials and methods

Oil extraction from Neem seeds

To prepare the seeds for oil extraction, seeds were heated in full sunlight on a black plastic sheet for several hours. The seeds were heated, but not burnt. This process breaks down the cells that contain the oil, allowing the oil to flow out more easily. The heat also liquefies the oil, which improves the extraction process. Oil is then extracted by mechanical extraction method. Conversion of oil from seed is 21.20%. After extraction, fuel properties of crude neem seed oil were determined.

Production of biodiesel

The acid value of the reaction mixture was determined by a standard acid base titration method using a standard solution of 1.0 M KOH solution. Prior to transesterification, acid esterification of the oil was required to reduce the free fatty acid (14.32%). The methanol and catalyst (conc. H_2SO_4) mixture was then charged into a two-necked closed reaction vessel and the raw oil was added. The reaction time and temperature was 1 hr and 60 ^{o}C respectively. The optimum condition for reducing the free fatty acid of neem oil below 2% (1.6%) was obtained by 0.58:1 methanol to oil ratio, 0.75% v/v sulphuric acid to oil volume. After acid esterification, trans esterification was done with NaOH catalyst in a two -neck round bottom flask equipped with condenser and magnetic stirrer. The reaction time was varied from 1 hr to 2 hrs and temperature was around 60 ˚C. After completion of trans esterification, methyl ester was separated from mixture of methyl ester and glycerin. The mixture was taken in a separating funnel and left for 16 hours. The mixture was separated in two layers, biodiesel as the top layer and glycerin as bottom layer. Glycerin layer was withdrawn and required product was obtained. Washing of biodiesel was necessary to remove the soluble components. Hot water was sprayed on top of biodiesel. Then it was allowed to settle down. The product was dried using a vacuum evaporator at 80 ^{0}C and pressure was 180 atm. After drying the pure product became clear.

Characterization of crude neem oil and produced biodiesel

All the parameters for fuel properties were estimated by standard methods such as density at 15 ^{0}C by IP 131/57 method, colour index by ASTM and DIN 51900 method, kinematic viscosity by viscometer 73/53 method, pour point by ASTM D 97-57, IP 15/55 method, flash point by ASTM D 93-62 method, sulphur content by IP 61/59 method, water content by IP 74/57 method, carbon residue by ASTM D 189-65 method, ash content by IP 4/58 method, acid value by IP 1/58 method, calorific value by bomb calorimeter IP 12/58, cetane number by ASTM-D 613-86, fire point by IP 35/42 and cloud point by ASTM-D 2500 methods.

Results and discussion

Physicochemical properties of neem seed oil

After extraction of oil from neem seeds, the physicochemical properties of the oil were determined according to the standard procedure. Table I. shows the properties of the neem seed oil.

FTIR analysis of crude neem oil

To determine the functional group of extracted neem seed oil, FTIR spectroscopic analysis was employed (Fig. 1.). Appropriate quantities of KBr and neem seed oil (in the ratio of 100:0.1) were mixed by grinding in an agate mortar and pellets were made with about 100 mg mixture. FTIR spectra were recorded with FTIR 8400S Shimadzu spectrophotometer in the range of 4000-400 cm^{-1}. Resolution was kept at 2 cm^{-1} and the no. of scans were 30. The major peaks are in the region of 1743.65 cm^{-1} and 2924.09 cm^{-1}. FTIR spectroscopic analysis shows that the main functional groups of neem oil are carboxylic acid (C=O) appears as main

Table I. Physicochemical properties of neem oil

Name of analysis	Standard method	Result
Density at 15˚C, g/cc	IP-160/57	0.93
Kinematic viscosity 40˚C, cSt	ASTM-D 445-65	40.7512
Kinematic viscosity 100˚C, cSt	ASTM-D 445-65	8.608
Acid value, mg KOH/g	ASTM-D 664	28.64
Carbon residue %	ASTM-D 189-65	4.3
Ash content, % (w/w)	ASTM-D 482-63	0.12
Water content,%(v/v)	IP-74/57	Nil
Flash point,˚C	ASTM-D 93-62	54
Fire point ,˚C	ASTM-D 92	60
Pour point,˚C	ASTM-D 97-57	-12
Cloud point,˚C	ASTM-D 2500	-8
Color index	ASTM-D 1500	4.7
API gravity	ASTM-D 613-86	20.65
Calorific Value, MJ/kg	ASTM-D 240	39.501
Sulfur content,%	ASTM-D 129-64	0.18
Cetane number	ASTM-D 613-86	-

Fig. 1. FTIR analysis of crude neem oil

peak in 1743.65 cm^{-1} region and alkanes (C-H) that appears in 1462.04-2924.09 cm^{-1} (Table II)

Table II. Analysis of peak obtained in FTIR of neem seed oil

Wave number cm^{-1}	Transmittance (%)	Functional group
723.31	57.952	C-H aromatic bending vibration
1165	48.382	C-O stretching vibration of alcohol
1377.17	54.698	C-O-H bending vibration of alcohol
1462.04	49.045	C-H bending vibration of alkanes ($-CH_2-$)
1645.28	57.567	C-H bending vibration of alkenes ($=CH_2$)
1743.65	43.259	C=O stretching vibration of esters
2924.09	41.875	C-H stretching vibration of alkanes

Process variables in trans esterification

Effect of temperature on yield of biodiesel

For the optimization of temperature, the percentage of catalyst and methanol was considered constant and the temperature was varied as shown in Fig. 2. The graph shows that as the temperature increases, production of biodiesel increases. Maximum (98%) production of biodiesel occurs at

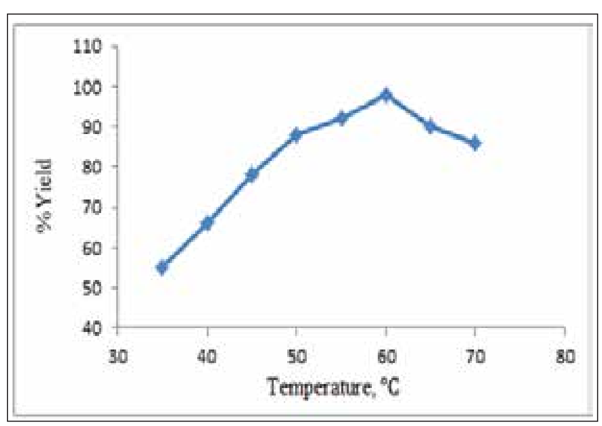

Fig. 2. Effect of temperature on the yield of biodiesel

60°C which is near boiling temperature of methanol. Further increase in temperature causes reduction of biodiesel production since boiling of methanol commenced and biodiesel separated from the system.

Effect of variation of methanol on the yield of biodiesel

The effect of change of molar ratio of methanol to oil was optimized. The amount of the catalyst NaOH was kept constant at 1 % of the oil. The temperature was fixed at 60^0 C.

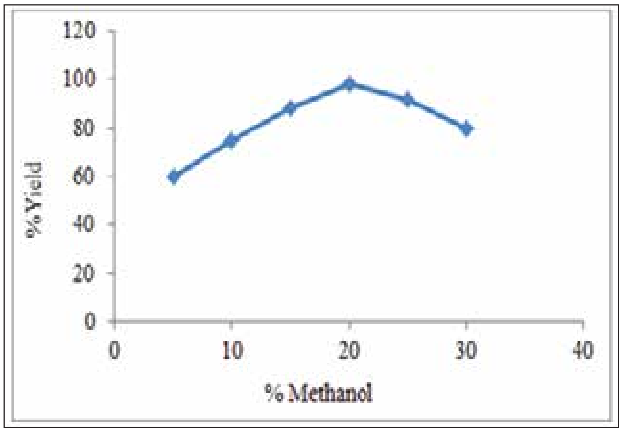

Fig. 3. Effect of methanol on the yield of biodiesel

Under this condition the percentage of methanol was varied to get maximum yield of bio- diesel (shown in Fig. 3). As the percentage of methanol increases, production of biodiesel increases. Maximum (98%) production of biodiesel occurs for (20%) of methanol. Further increase in percentage of methanol causes reduction of biodiesel production.

Effect of variation of catalyst on yield of biodiesel

To optimize the amount of NaOH, the percentage of methanol was maintained constant at 20 % on the wt % of oil

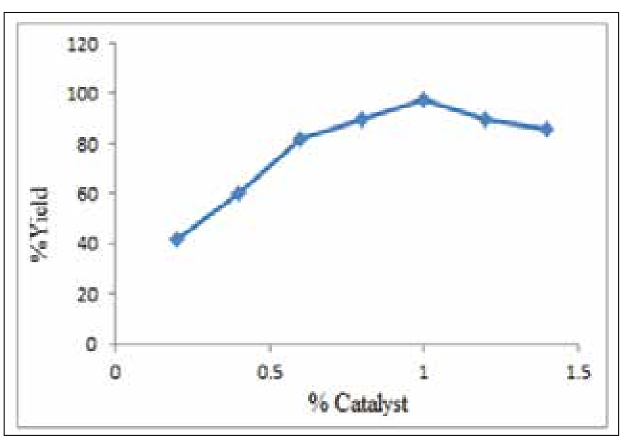

Fig. 4. Effect of variation of catalyst on the yield of biodiesel

taken. The temperature was also kept constant. In this condition, the reaction were carried out with NaOH catalyst at concentrations 0.20, 0.40, 0.60, 0.80, 1.00, 1.20 and 1.40% in order to determine optimum condition for the biodiesel production from the oil. The maximum yield of biodiesel was found to be at 1.00 % NaOH concentration as shown in Fig. 4.

Effect of reaction time on yield of biodiesel

All variables except reaction time were kept constant in their optimum value. The tran sesterification reaction was continued for different period of time such as 30, 60, 90 and 120 min respectively to yield the product. The result of this optimization is shown in (Fig. 5). It is found that with the increase of duration of reaction, the yield of biodiesel was enhanced. Maximum yield (about 98 %) was obtained in 90 mins.

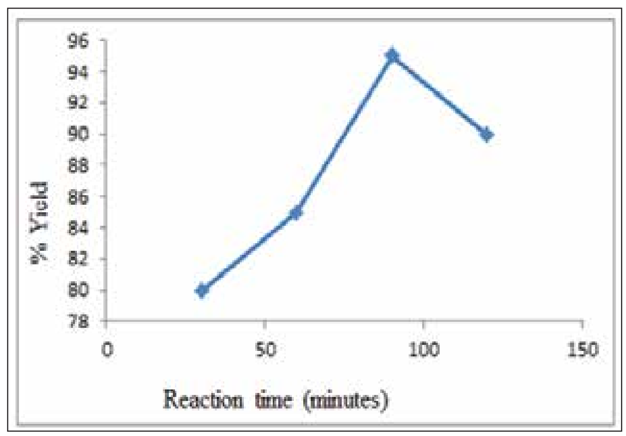

Fig. 5. Effect of reaction time on the yield of biodiesel

Analysis of peak obtained in FTIR spectra of biodiesel

To determine the functional group of neem seed oil biodiesel, FTIR spectroscopic analysis was performed following the procedure identical to that for the oil. FTIR spectrum shown in Fig. 6 reveals that the major peaks are in the region of 1460.11 cm^{-1} and 2926.01 cm^{-1} and the main functional groups of neem biodiesel are carboxylic acid (C=O) showing bands as the main peak in 1743.65 cm^{-1} region and alkanes (C-H) in the region of 1460.11-2854.65 and 2926.01 cm^{-1} (Table III). The main functional groups of produced bio-diesel are carboxylic acid (C=O) showing the main peak in 1743.65 cm^{-1} region and alkanes (C-H) in the 1460.11-2926.01 range (Table III).

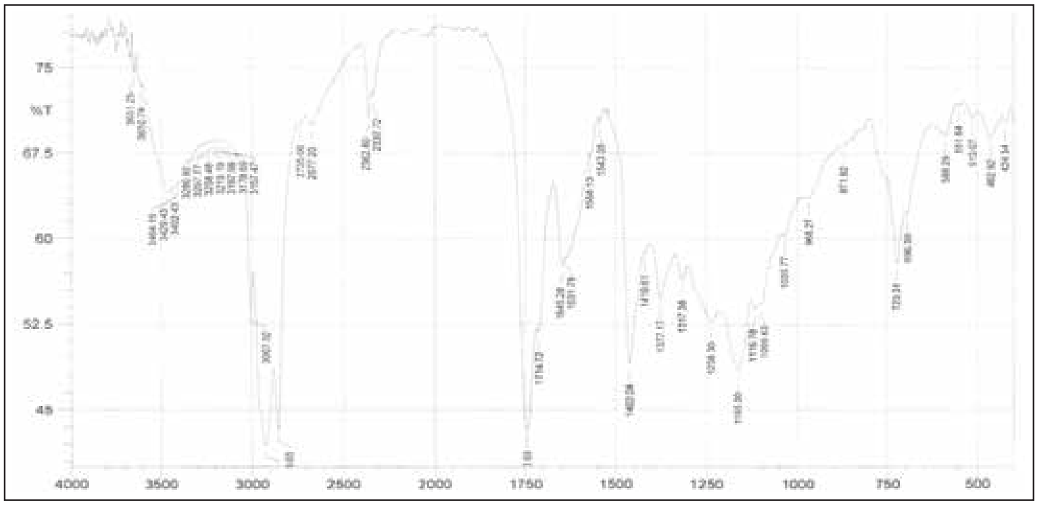

Fig. 6. FTIR analysis of biodiesel

Table III. Analysis of bands obtained in FTIR spectra of biodiesel

Wave number cm^{-1}	Transmittance (%)	Functional group
723.31	74.217	C-H aromatic bending vibration
1170.79	60.595	C=O stretching vibration of alcohol
1365.6	70.53	C-O-H bending vibration of alcohol
1460.11	61.932	C-H bending vibration of alkanes (-CH_2-)
2854.65	48.664	C-H stretching vibration of alkanes (-CH_2-)
1743.65	47.451	C=O stretching vibration of esters
2926.01	44.32	C-H stretching vibration of alkanes
3454.51	73.753	O-H (hydrogen bonded)

Optimum condition of biodiesel production from neem oil

The optimum conditions for biodiesel production from neem seed oil can be summarized as follows: The overall trans esterification reaction requires about 20% of methanol on the basis oil taken; catalyst (NaOH) with a concentration is 1.0% of the oil and a reaction time of 90 mins. at a temperature of 60˚C with moderate stirring rate. The optimum yield is 98 %.

Characteristics of neem biodiesel

The biodiesel obtained was then characterized by the standard methods. Table IV describes the fuel characteristics of biodiesel from neem seed oil and also gives a comparison of obtained biodiesel with conventional diesel fuel.

Engine performance study

Produced biodiesel (Neem Methyl Ester: NME) was blended with diesel in different proportions and engine performance study was done. Engine specification was such as Engine type: 4- stroke CI engine, number of cylinder: one and compression ratio: 16.5.

Speed optimization

Fig. 7 illustrates the variation of brake thermal efficiency with engine speed at load 55.6 N with diesel. From Fig.7 it is apparent that the brake thermal efficiency of engine increases with increase in engine speed. After reaching the maximum value, the efficiency of the engine also decreases. This is due to the fact that, initially with the increase of engine speed the torque produced by the engine increases, hence the efficiency also increases. But at higher rpm (>900) more amount of fuel is injected into the engine cylinder per cycle and due to higher engine speed these fuel doesn't get sufficient time to burn completely which reduce the efficiency of the engine. Hence the optimum speed was 900 rpm.

Table -IV Comparison of produced biodiesel with standard and commercial diesel

Parameter	Standard method	Biodiesel standard (ASTM)	Neem biodiesel	Commercial diesel (ASTM)
Density at 15˚C, gm/cc	IP -160/57	0.88	0.875	0.8445
Kinematic Viscosity at 40˚ C,cSt	ASTM -D 445 -65	1.9 -6.0	6.17	2.71
Acid value, mg KOH/g	ASTM -D 664	0.80 max	0.8716	0.34
Carbon residue % (w/w)	ASTM -D 189 -65	0.05 max	0.75	0.05
Ash content, % (w/w)	ASTM -D 482 -63	0.02 max	0.006	0.02
Water content,%	IP -74/57	0.05	Nil	Nil
Flash point,˚C	ASTM -D 93 -62	100 -170	70	65
Pour point ,˚C	ASTM -D 97 -57	-15 to -16	-16	-20
Cloud point,˚C	ASTM -D 2500	-3 to -12	-12	-12
API gravity			30.21	34.52
Calorific Value, MJ/kg	ASTM -D 240	38.586	40.2	44.5
Sulfur content,%	ASTM -D 129 -64		0.07	0.905
Cetane number	ASTM -D 613 -86	48 -60	53	51

Load optimization

The engine was run at the fixed rpm (900) and brake power was varied from 0.44 KW to 1.05 KW. Fig.8. illustrates variation of brake thermal efficiency of engine with respect to brake power. The efficiency of the engine increases with the increase in brake power. The maximum brake thermal efficiency of diesel fuel is 20.38% at brake power 0.79 KW. Higher brake thermal efficiency is due to better mixing of fuel with air which results in better combustion. At higher brake power (> 0.79 KW) more amount of fuel is injected into the engine cylinder which is not completely burnt. It causes higher BSFC and low brake thermal efficiency. Hence optimum engine load (brake power) was 0.79 KW.

Brake thermal efficiency

The variation of brake thermal efficiency with engine load using all types of fuels/blends shown in Fig. 9. Brake power was varied from 0.44 KW to 1.05 KW. Here DF= Diesel Fuel

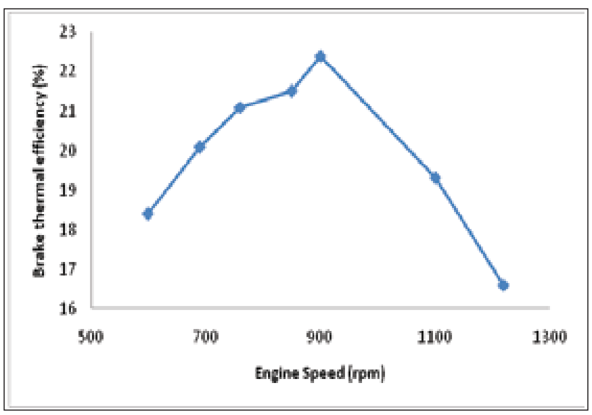

Fig. 7. Variation of brake thermal efficiency with engine speed (at load 55.6N)

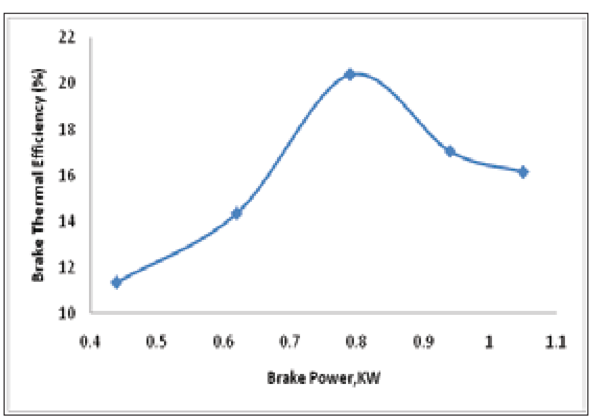

Fig. 8 .Variation of brake thermal efficiency with brake power (900 rpm)

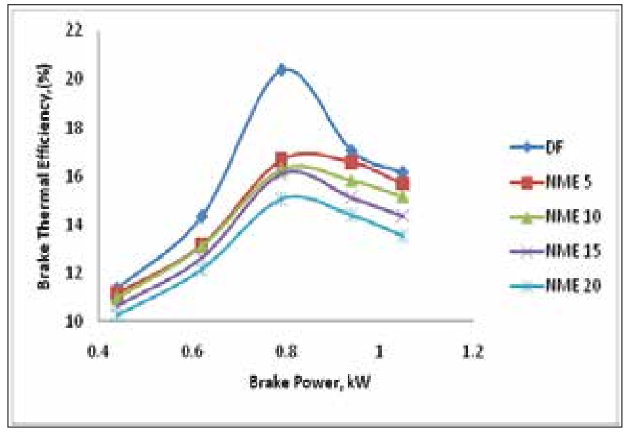

Fig. 9. Variation of brake thermal efficiency with brake power of different NME blends

and NME= Neem Methyl Ester. The brake thermal efficiency was calculated by the ratio of brake power to input power. The brake thermal efficiency increases with the engine load (brake power) for all fuels/ blends. The maximum value of brake thermal efficiencies with DF, NME5, NME10, NME15 and NME20 were found to be 20.38 %, 16.67, 16.28 %, 16.10 % and15.06 % respectively at brake power 0.79 KW

Brake specific fuel consumption (BSFC)

Fig. 10. compares the fuel consumption of diesel/ NME at various brake power in the range of 0.44 KW-1.02 KW. The Fig. shows that BSFC decreases with the increase of brake power up to brake power 0.79 KW. Then the BSFC increases with the increase of brake power. The value of BFSC with

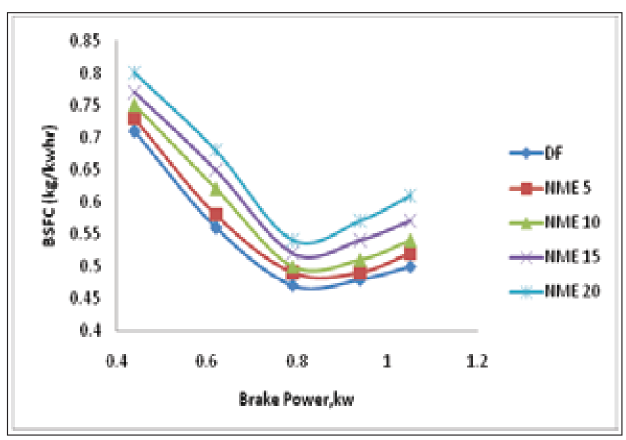

Fig. 10. Variation of brake specific fuel consumption with brake power of different NME blends

DF, NME5, NME10, NME15 and NME20 were found to be 0.47, 0.49, 0.50, 0.52, 0.54 KW-hr respectively at brake power 0.79 KW. Among the NME blends, NME5 shows the lowest BSFC.

Conclusions

Biodiesel can be extracted from neem seed oil. The optimum condition has been 20 % methanol and 1.0% NaOH catalyst with reaction temperature and time is 60 ° C and 90 min respectively. The physicochemical properties of the neem biodiesel are similar to standard biodiesel. Neem biodiesel blends have been tested in a single cylinder, 4-stroke diesel engine. Brake thermal efficiency and BSFC of NME are comparable to DF. The higher brake thermal efficiency is 20.38% for 5 % blend of neem methyl ester. The results reveal the possibility of neem seed oil as a potential source of biodiesel. As Bangladesh does not have petroleum resources, renewable fuel of this kind may be very helpful to solve our present fuel oil crisis.

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