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Extraction, physico-chemical, corrosion and exhaust analysis of transesterified neem *(Azadirachta indica)* oil blends in compression ignition (ci) engine.

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ABSTRACT: *In this work, Neem oil was extracted from its kernel, corrosive ability and physico-chemical properties of the oil were determined. The emission characteristics of a single cylinder, four stroke air-cooled diesel engine when fuelled with diesel and Neem-diesel blends at various loads were evaluated. The results showed that the fuel properties of Neem biodiesel were within the set standards for B100 and comparable with the conventional diesel. The corrosion rate of Neem biodiesel and diesel were both tested in copper and mild steel respectively. The test revealed that Neem biodiesel corrodes both test samples more than diesel. Air-fuel ratio values of Neem oil biodiesel blends are less than diesel, except for B25 and B30 NOME-Diesel fuel blends. Exhaust heat lost and exhaust temperature were lower in diesel than all blends. However, there is an appreciable decrease in HC and CO emissions while the decrease in CO is least for B20.*

Keywords: *Azadirachta indica, renewable fuel, extraction, physico-chemical analysis, exhaust emission.*

I. INTRODUCTION

Conventional energy sources, such as coal, oil and natural gas, have limited reserves that are expected not to last for an extended period. As world reserves of fossil fuels and raw materials are limited, it has stimulated active research interest in nonpetroleum, renewable, and non-polluting fuels. Biodiesel have received significant attention both as a possible renewable alternative fuel and as an additive to the existing petroleum-based fuels. Edible oils are much more valuable as a cooking fuel and as such, our concentration is going to be on development of biodiesel from non-edible oils only. Biodiesel has been defined as the monoalkyl esters of long-chain fatty acids derived from renewable feedstocks, such as vegetable oils or animal fats, for use in compression ignition (diesel) engines (Krawczyk., 1996). Transesterfication also called alcoholysis, is the displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis, except that alcohol is used instead of water.

Straight vegetable oils (SVOs) have their fair share of problems in unmodified CI engines. These problems include: cold-weather starting; plugging and gumming of filters lines; excessive engine wear; and deterioration of engine lubricating oil. Vegetable oils decrease power output and thermal efficiency while leaving carbon deposits inside the cylinder. Most of these problems with vegetable oil are due to high viscosity, low cetane number, low flash point, and resulting incomplete combustion. To avoid some of these problems, vegetable oils have been converted via a chemical process (transesterification) to result in a fuel more like fossil diesel. The resulting fuel is biodiesel, a biodegradable and nontoxic renewable fuel. Furthermore, biodiesels have reduced molecular weights (in relation to triglycerides), reduced viscosity, and improved volatility when compared to ordinary vegetable oils.

As far as environmental considerations are concerned, unlike hydrocarbon-based fuels, the sulphur content of vegetable oils is close to zero and hence, the environmental damage caused by sulphuric acid is reduced. Moreover, vegetable oils take away more carbon dioxide from the atmosphere during their production than is added to it by their later combustion. Therefore, it alleviates the increasing carbon dioxide content of the atmosphere.

Neem (Azadirachta indica) which belongs to the family Meliaceae, originated from South Asia, but grows widely in India, Pakistan and other tropical and sub-tropical parts of the world (Bokhari and Aslam, 1985;Von Maydell, 1986). The tree was introduced in Nigeria from Ghana, and it was first grown from the seeds in Maiduguri, in the then Bornu Province (now Borno State), Nigeria, in 1928 (National Research Council, 1992; Nwoeabia, 1994).

The Neem tree is significant in Nigerian forestry because it constitutes the largest population of trees, especially in the Northern States. It was nicknamed 'Dogon Yaro' after the first caretaker of the Neem tree Nursery in Maiduguri. It is a large evergreen tree usually 12–18m high, and grows on almost all kinds of soil. It thrives well in arid and semi-arid climate with maximum shade temperature as high as 49^oC and the rainfall is as low as 250 mm (Bringi 1987, Narain and Satyavir 2002). A Neem tree can produce many thousands of flowers, and in one flowering cycle, a mature tree may produce a large number of seeds. The fruits are yellowish green when ripe and have a sweetish pulp containing one seed. Neem trees start bearing harvestable seeds within 3–5 years, and full production may be reached in 10 years, and this will continue up to 150 – 200 years of age. A mature neem tree may produce 30–50 kg of fruit each year. Neem seeds yield 40–60% oil (Narwal et al., 1997).

II. MATERIALS AND METHODS

100g of pounded neem seed was wrapped up in a filter paper which was placed in an extraction thimble of a soxhlet apparatus. The extraction flask was then fitted with 250ml of petroleum ether using an electric heating mantle, the petroleum ether was gently heated. The filter paper was removed, dried and its content weighed to determine the change in mass. The Percentage yield of the oil was calculated thus:

$$\%\ Oil\ yield = \frac{Wieght\ of\ oil}{Wieght\ of\ sample} \times 100$$

Physico-chemical properties such as; saponification value (S.V), iodine value (I.V), free fatty acid (FFA), acid value (A.V) where determined. Fatty Acid Methyl Ester (FAMES) of Neem was prepared by preheating 280g of oil to 45- 50^oC and adding 192g of methanol in a plastic container to maintain 6:1 alcohol to oil molar ratio. 2.8 g of KOH was also weighed on a digital beam balance and dissolved in a beaker containing 100ml of distilled water (H_2O) to give 1% of KOH solution based on weight of crude Neem oil. 1% of KOH solution was then introduced into the methanol in the plastic container and gently swirled. The preheated Neem oil was then added and stirred for 5 -10 minutes, and then heated to a temperature of 60^oC - 80^oC in a water bath to initiate the transesterification reaction (Lele, 2009; Belsor and Hedlund, 2007). The mixture was then poured into a separating funnel and allowed to settle for about 7-8 hours for the complete separation of glycerol from Neem oil methyl ester (NOME). About 10ml of 50^oC distilled water was added to the crude product and swirled slowly and left for some time to stand, the lower layer was then run off. This washing process was repeated until the product was clear. A small quantity of anhydrous magnesium sulphate was added and stirred for 5 minutes and the magnesium sulphate was allowed to settle. The biodiesel was later filtered using a filter paper to separate the biodiesel from the hydrated magnesium sulphate. The yield of the biodiesel was calculated thus:

$$\%\ yield\ of\ biodiesel = \frac{Volume\ of\ biodiesel}{Volume\ of\ oil}$$

Biodiesel blends of B10, B15, B20, B25 and B30 were respectively prepared and fuel qualities such as flash point, pour point, cloud point, kinematic viscosity, calorific value and corrosion tests were all tested.

Armfield corrosion test kit was used to test for the corrosive tendency of the produced Neem oil biodiesel using milled steel and copper strips as coupons. The test kit was set at normal environmental conditions away from direct sun light and test duration of four (4) weeks. The initial mass of the samples at the beginning of the test and that at the end of the test were taken to determine the change in mass of the samples. Average weight loss, Corrosion Penetration Rate (CPR) and Corrosion Ratio (CR) of biodiesel and diesel samples were calculated.

Surface area of coupons $= 2\{T \times L + L \times W + W \times T\} - \frac{2\pi}{4}D^2$

Where T = Thickness

L = length

W = Width

D = Diameter of hole drilled on coupons = 0.5cm

Thickness of copper coupons = $T_c = 0.3$ cm

Length of copper coupons = $L_c = 5.9$ cm

Weight of copper coupons = $W_c = 2.2$ cm

$\therefore$ *Area copper coupons* $= 2\,(0.3\times 5.9 + 0.3 \times 2.2 + 5.9 \times 2.2) - \frac{2 \times \pi \times 0.5^2}{4}$

$$A_{CC} = 30.43cm^2$$

Thickness of milled steel coupons = $T_m = 0.3$cm

Length of milled steel coupons = $L_m = 4$cm

Width of milled steel coupons = $W = 2.0$cm

$\therefore$ *Area milled steel coupons* $= 2\,(0.3 \times 4 + 2 \times 4 + 0.3 \times 2) - \frac{2 \times \pi \times 0.5^2}{4}$

$$A_{MSC} = 19.21cm^2$$

Average weight loss $= \frac{\Sigma \text{ of change in mass for the weeks}}{\text{number of weeks}}$

Corrosion penetration rate (CPR) $= \frac{W}{\rho A T}$

Where W = weight loss

T = Time in days

$A_{CC} = 30.43cm^2$

$A_{MSC} = 19.21cm^2$

$\rho_{CC} = 8.92g/cm^3$

$\rho_{MSC} = 7.88g/cm^3$

Corrosion Ratio (CR) $= \frac{\text{CPR for a coupon type in biodiesel}}{\text{CPR for the same coupon type in diesel}}$

AVL DiGas 4000 gas analyser was used to measure concentration of gaseous emissions [Unburned hydrocarbon (UHC), carbon monoxide (CO)] to evaluate and compute the behavior of the diesel engine at constant speed of 1500 rev/min and varying load of between 500g – 3000g. The results obtained from performance analyses was tabulated and necessary graphs plotted. Technical detail of the engine is given in table 1. A hydraulic dynamometer was coupled to the engine for torque measurement.

Table 1: Technical specifications of engine test rig

Type	Single cylinder, four stroke, air-cooled
Bore * Stroke	65 mm x 70 mm
Brake power	2.43kW
Rated speed	1500rpm
Starting method	Manual cranking
Compression ratio	20.5:1
Net weight	45Kg
Manufacturer	TQ Educational Training Ltd

III. RESULTS AND DISCUSSION

Percentage oil yield: The oil yield of Neem was found to be 58.3% this compares favourably with 40–60% oil yield by Narwal et al (1997). The high oil yield of Neem seed is an indication that Neem is a good source of oil. Yield of biodiesel from different non-edible oils (Jatropha curcas, Pongamia pinnata, Madhuca Indica and Azadirachta Indica) which are commonly available in India were examined and the results recommended the biodiesel production from Azadirachta Indica oil on the basis of high yield and quality of biodiesel (Mathiyazhagan et al., 2011). The economic evaluation has also shown that biodiesel production from Neem is very profitable (Mathiyazhagan et al., 2011).

Physico-chemical Properties: The physico-chemical properties of Neem oil, Neem methyl ester, Neem blends and conventional diesel (D2 or No2) are presented in Table 2 The physico-chemical properties of Neem biodiesel were within the limits and comparable with the conventional diesel. Except calorific value, all other properties of Neem biodiesel and its blends were found to be higher as compared to diesel.

Table 2: Summary of the properties of tested samples

Fuel Property	**Diesel**	**Neem Oil**	**B100 (NOME)**	**B10**	**B15**	**B20**	**B25**	**B30**
Density at 15°C (kg/m^3)	830	920	872	845	849	854	860	865
Kinematic Viscosity at 40°C (mm^2/s)	4.0	35.52	5.5	4.4	4.45	4.5	4.55	4.6
Flash Point (°C)	65	212	136	72	79	87	94	101
Pour Point (°C)	-16	10	2	-11	-10	-8	-7	-5
Coud Point (°C)	-12	19	9	-7	-6	-5	-4	-3
Saponification Value (mgKOH/g)	-	188.1	-	-	-	-	-	-
Acid Value (mgKOH/g)	-	6.77	-	-	-	-	-	-
Iodine Value (mgI/g)	-	64	-	-	-	-	-	-
Specific gravity	0.830	0.920	0.872	0.845	0.849	0.854	0.860	0.865
Cetane Number	45-55	31-51	48-53	-	-	-	-	-
Calorific Value (MJ/kg)	44.76	34.10	36.70	43.80	43.30	42.80	42.40	41.90
FFA Value wt %	-	3.40	-	-	-	-	-	-

Corrosion value: The corrosion rate of Neem biodiesel and diesel were both tested in copper and mild steel respectively. The test revealed that Neem biodiesel corrodes both test samples more than diesel (Table 3). Biodiesel is believed to contain residual water because of transesterification; this could be responsible for higher rate of corrosion. Many of the parts in the diesel fuel injection system are made of high carbon steels; thus, they are prone to corrosion when in contact with water. Water damage is a leading cause of premature failure of fuel injection systems. The water content of petroleum diesel is usually quite low since hydrocarbons are generally hydrophobic. Fatty acid esters, on the other hand, are hygroscopic and will contain high water content by merely being exposed to moist air during storage. Water will cause corrosion of vital fuel system components fuel pumps, injector pumps, fuel tubes, etc (Ayhan, 2009). Also the higher the acid value of biodiesel the more it corrodes. Neem biodiesel has an appreciably high acid value above the benchmark standards given by both ASTM and DIN V 51606.

Table 3: Corrosion test of biodiesel using armfield corrosion test kit

Sample immersion	**Metal coupons**	***W***	**CPR**
Samples immersed in biodiesel	Milled steel	0.015	1.416×10^{-5} cm / day
	Copper	0.0525	2.763×10^{-5} cm / day
Samples immersed in petroleum diesel	Milled steel	0.0125	1.180×10^{-5} cm / day
	Copper	0.0225	1.184×10^{-5} cm / day

Corrosion Ratio (CR)

CR for Copper coupons = $\frac{2.763 \times 10^{-5}}{1.184 \times 10^{-5}} = 2.334$

CR for Milled steel coupons = $\frac{1.416 \times 10^{-5}}{1.180 \times 10^{-5}} = 1.2$

Exhaust Temperature: Figure 1 shows the variation of exhaust gas temperature with load for various blends and diesel. The results showed that exhaust gas temperature increases with increase in load for all blends. At all loads, diesel was found to have the lowest temperature and the temperatures for various blends showed an upward trend with increasing concentration of NOME in the blends . This increase in exhaust gas temperature with load is obvious from the simple fact that more amount of fuel is required in the engine to generate that extra power needed to take up the additional loading. Biodiesel contains oxygen, which enables the combustion process; hence the exhaust gas temperatures are higher. Moreover, the engine being air-cooled runs hotter, this resulted in higher exhaust gas temperatures. The heat release may occur in the later part of the power stroke this may result in lower time for heat dissipation and higher exhaust gas temperatures. The increased exhaust gas temperature of the engine with the use of biodiesel blends may be caused by the rise in peak cylinder pressure resulting in higher peak combustion temperature as reported by Ecklud, (1984).

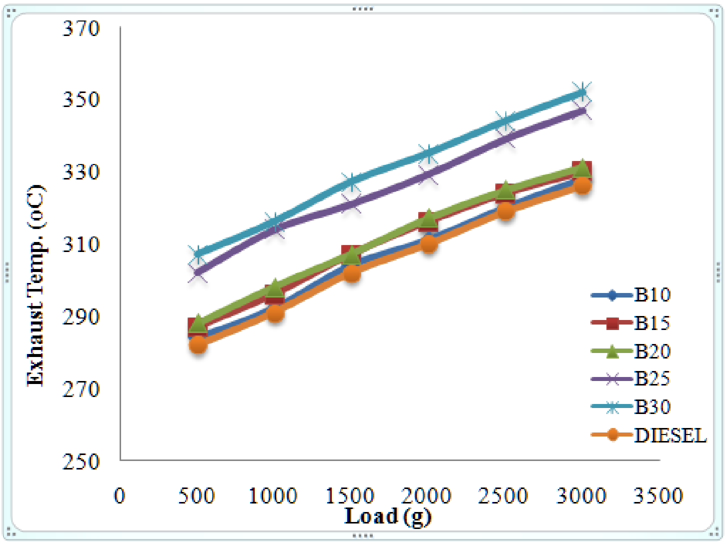

Figure 1: *Variation of Exhaust Temp. For all samples with increase in load*

Air/Fuel ratio(AFR): Figure 2 shows the variation of air-fuel ratio with load for all tested samples. In C.I engines at a given speed the air flow do not vary with load, it is the fuel flow that varies directly with load. As seen from the figure below, as percentage blend increases the air fuel ratio decreases but increases with increase in load. Air-fuel ratio values of neem oil biodiesel blends are less than diesel. Except for B25 and B30 NOME-Diesel fuel blends, which demonstrated average difference of 6.18% and 9.4% from the AFR of diesel fuel, B10, B15 and B20 NOME-Diesel fuel are 1.78%, 2.24% and 2.81% lower than diesel fuel benchmark respectively. The observation made from this finding is that all tested fuel samples reached maximum power output and torque at higher than the stoichiometric AFR values (i.e. 18-25) for compression ignition engines. In this case, maximum power output is achieved at lean mixture, therefore causing the engine to run at higher engine temperature. The bound oxygen on the biodiesel molecule may also play a role in creating a leaner air-fuel ratio, which in turn leads to high air-fuel ratio higher than the stoichiometric AFR values for all the samples tested.

According to Goering (1992), the stoichiometric AFR values of engines running on biodiesel are usually lower than diesel fuel because more oxygen presence is evident in biodiesel due to the methanolysis of sheanut oil, and it enabled SHOME blended fuel samples to burn much richer than diesel fuel.

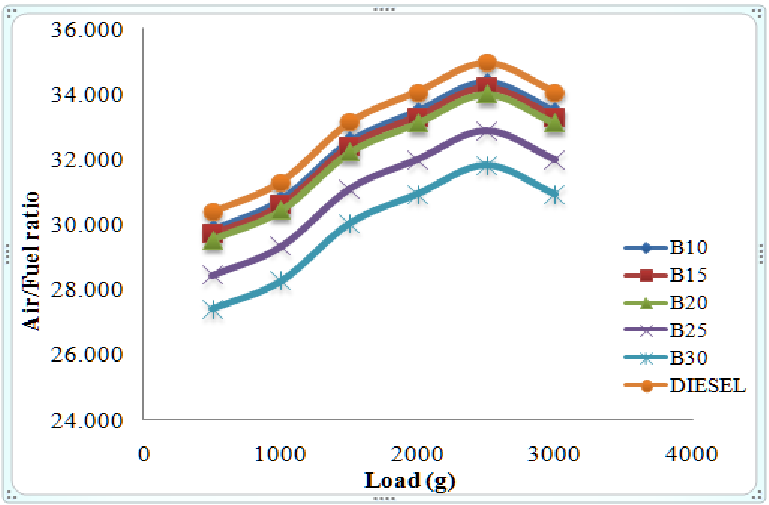

Figure 2: *Variation of Air/Fuel ratio for all samples with increase in load*

Percentage heat loss

From figure 3, all NOME-Diesel blends show evidence of higher heat losses in engines than diesel fuel by average values of 0.65% for B10, 2.90% for B15, 3.86% for B20, 6.29% for B25 and 5.65% for B30 respectively. The higher heat loss recorded could be explained in terms of lower calorific (heating) valve, increase in fuel density, the difference between the exhaust and ambient temperatures and the size of the engine. The temperature difference existing between the fuel blends and diesel fuel benchmark in figure 3 also presented a proportional increase in the heat carried away by the exhaust. However, for heat unaccounted for by losses is partly a function of the engine size, because for smaller engines, considerable conductive and radiative heat losses are usually caused by inefficient combustion (Plint and Partners, 1984). High exhaust gas temperature and reduction in thermal efficiency with increase in blend is an evidence of increase in heat loss. The mean exhaust gas temperature of B20, B40, B60 and B100 were 7%, 9% 10% and 12%, respectively, higher than the mean exhaust gas temperature of diesel. This could be due to the increased heat loss of the higher blends, which are also evident from, their lower brake thermal efficiencies as compared to diesel (Sharanappa et al., 2009).

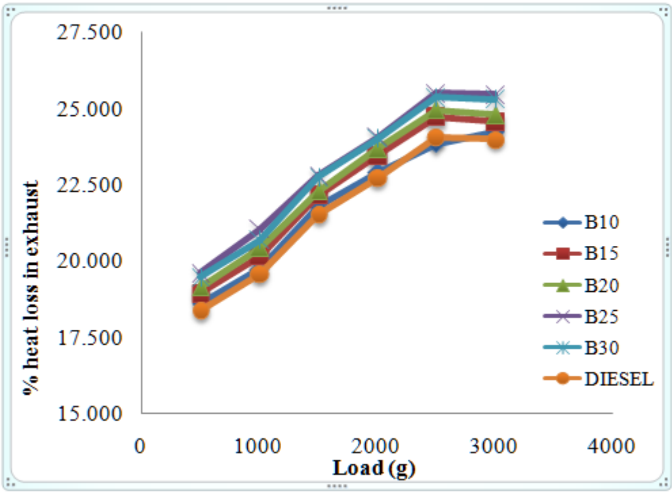

Figure 3: *Variation of % heat loss for all samples with increase in load*

CO Emission of diesel and Neem biodiesel blends: The variation of CO emission with load is shown in figure 4. It was observed that the engine emits more CO for diesel at all load conditions when compared to the blends. However, as the proportion of NOME in the blend increases the percentage emission decreases due to higher oxygen content and lower carbon to hydrogen ratio in biodiesel compared to diesel. The percentage variation of carbon monoxide for all the blends when compared with base line diesel is very much less. These lower CO emissions of biodiesel blends may be due to their more complete oxidation as compared to diesel. Some of the CO produced during combustion of biodiesel might have converted to CO_2 by taking up the extra oxygen molecule present in the biodiesel chain (Biodiesel has up to 11% oxygen content :Ragit et al, 2010) and thus reduced CO formation. It can be observed from figure 4 that the CO initially decreased with load and latter increased at higher load. This trend was observed for all the fuel blends tested. At lower biodiesel concentration, the oxygen present in the biodiesel aids for complete combustion. However as the biodiesel concentration increases, the negative effect due to high viscosity and small increase in specific gravity suppresses the complete combustion process, which produces small amount of CO (Sureshkumar and Veltra., 2007). Last et al. (1995) fuelled a heavy-duty engine with 10%, 20%, 30%, 50% and 100% soybean-oil biodiesel. All the blends reduced CO emissions with respect to the diesel fuel, but such decreases did not depend on the biodiesel percentage (10%, 8%, 18%, 6% and 14% reductions, respectively). Also, Rakopoulos et al. (2004) reported lower CO concentration in the exhaust line when oxygen in the combustion chamber was increased either with oxygenated fuels or oxygen-enriched air.

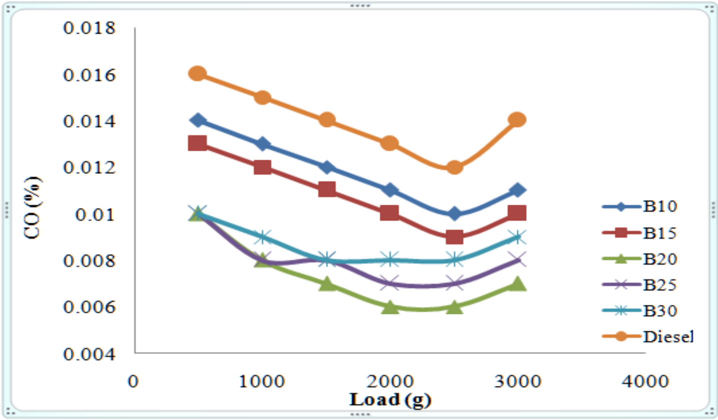

Figure 4: *Emission of CO for all samples with increase in load*

HC Emission of diesel and neem biodiesel blends: The HC emission variation for different blends is indicated in figure 5. All blends have lower values than diesel owing to higher combustion chamber temperature, which helps in cracking and faster burning. It was observed that the HC emission decreased up to a load of 2500g and then increased slightly with further increase in load for all the samples. The HC emission for the blends also followed a similar trend but comparatively the values were lower. The presence of oxygen in the Neem blends aids combustion and hence the hydrocarbon emission reduced. It was suggested that the short ignition delay when using biodiesel could also attribute to lower HC emissions (Lapueta et al., 2008). Rakopoulos et al. (2004) concluded in to their review that HC emissions decreased as the oxygen in the combustion chamber increased, either with oxygenated fuels or oxygen-enriched air. Sahoo et al. (2009) reported that the reduction of HC was of the order of 32.28%, 18.19% and 20.73% for JB20, JB50, JB100, respectively.

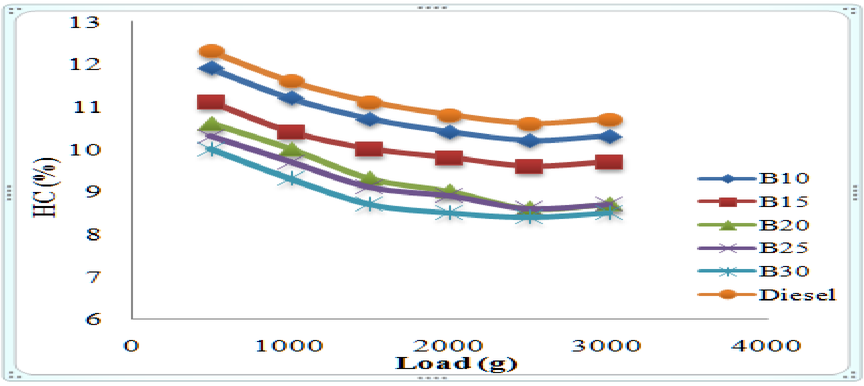

Figure 28: *Emission of HC for all tested samples with increase in load*

IV. CONCLUSION

The physico-chemical properties of Neem biodiesel were within the set standards for B100 and comparable with the conventional diesel. Except calorific value, all other physico-chemical properties of Neem biodiesel were found to be higher as compared to diesel. Exhaust gas temperature emission increases with increase in percentage blend and load for all the cases. The engine being air-cooled the exhaust gas temperatures are higher. As percentage blend increases the Air-fuel ratio decreases but increases with increase in load. Air-fuel ratio values of Neem oil biodiesel blends are less than diesel. B30 gave the best and least ratio. The lean mixture of the engine was observed to be responsible for the high temperature operation which is also linked to the increased heat loss. Percentage heat loss in exhaust increases with increase in blend and load. Diesel showed the best performance as regards heat loss while B10 showed the closest value to it amongst all the blends. It was observed that all samples showed a close relation in heat loss. The engine emits more CO for diesel at all load conditions when compared to the blends, however, as the proportion of NOME in the blend increases the percentage emission decreases. HC emission was lower in blends than diesel and decreases with increase in load. The lowest value obtained for CO emission was with B20 blend. Hydrocarbon emission decreased with increase in percentage NOME in the blend and decreased with increase load until 2500g load where it showed an increase value. HC emission in all blends are lower than that of diesel and the least value was obtained with B30 blend.

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