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Olax scandens Roxb : A Rarefied Non Edible Potential Leafage of Bio Diesel for Diesel Engines

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Abstract

In India, edible oils are in short supply and country has to import up to 40% of its requirements and use of edible oils for biodiesel production is ruled out. Availability of raw materials, collection of seeds and processing mechanism for the seeds are not well standardized. Olax scandens Roxb is one such plant species identified by authors as a promising source of oil for biodiesel production. The present work focuses on (a) Standardization of extraction procedures of olax oil from seeds.(b). Standardization of esterification of crude olax oil (c).Standardization of trans-esterified olax oil.(d). Study of different physical and chemical parameters of the processed oils for their biodiesel properties including elaborate analysis by gas chromatography (GC) (e). Engine testing using Olax biodiesel. Result revealed petroleum diesel blended with 10% to 20% olax biodiesel can be fuelled to diesel engines without any modification in engine hardware irrespective of a negligible power loss.

1. Introduction

The depleting reserves of fossil fuels and the growing environmental concerns have made renewable energy an exceptionally attractive alternative energy source for the future [1, 2]. Biodiesel is one of these promising alternative resources for diesel engines. It is defined as the mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats and alcohol with or without a catalyst. It is renewable, biodegradable, environmentally friendly, non-toxic, portable, readily available and eco-friendly fuel [3–6]. *Olax scandens* Roxb of the family Olacaceae is a scandent shrub found often in ravines, stream banks in the sub-Himalayan tract in Kumaun, upper Gangetic plain, Bihar Orissa, Madhya Pradesh, Deccan and Western Ghats. It has regular bearing habit and high productive potential even in marginal lands that can provide huge quantities of oil rich seeds. Though it is a climber it can be trained as a standard and about 4,000 plants can be put in to cultivation in one acre of land.

Fig.1 shows leafage with fruiting habit in *Olax scandens;* at different stages of maturity.

Fig. 1 Leafage with fruiting habit in *Olax scandens*: at different stages of maturity

The fruits of *Olax scandens* were collected from three different places of Odisha (India) namely Charichhak in Boudh, Banigochha in Nayagarh and Munduli in Cuttack districts. Seeds were extracted from the drupes,

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cleaned thoroughly and dried in shade. Weight of the seeds extracted from 1kg of fruits was recorded in replications to assume the production of the pulp and seed. The fresh weight and dry weight of the seeds were compared to study the water/oil content in the seeds (table-1).

Table-1: Seed and oil from 1kg of fruits (Drupe)

Sl no	Drupes(Kg)	Fresh weight of seeds(g)	Dry weight of seeds(g)	Moisture content (%)	Oil extracted In %
1	1.0	189	143	24.33	59.44
2	1.0	229	166	27.51	59.03
3	1.0	229	162	29.26	59.25
4	1.0	221	159	28.05	59.11
5	1.0	232	167	28.01	59.28

Vegetable oils having high free fatty acids content (more than 2%) are very difficult to convert into biodiesel through direct alkali catalyzed transesterification process. For biodiesel preparation, such oils are treated through two consecutive acid–alkaline transesterification processes [7]. In the first step, crude vegetable oil is treated with methanol and sulphuric acid mixture for 4 h to convert free fatty acids to their methyl esters and then the resultant oil was transesterified by treating oil with methanolic costic soda solution for 3 h. Therefore, this is a fairly expensive and time consuming process. In present study methyl esters of *olax scandens Roxb* oil (biodiesel) was prepared by a cost-effective simple method and blended to petroleum diesel (PD) in different proportions to assess its possibility as an additive to petroleum diesel and its use as absolute fuel in diesel engines with pros and cons. The engine performance and emissions were also evaluated by fuelling methyl esters of *olax scandens Roxb* in absolute and additive mode to a naturally aspirated single cylinder diesel engine using petroleum diesel as base line fuel.

2. Materials and methods

2.1 Extraction of oil

The Figure.2 shows oil extraction from seed kernels. Extracted oil was compared using three different organic solvents ie hexane, petroleum ether, and chloroform separately in soxhlet apparatus. Hexane extraction yielded higher percentage of oil, hence subsequent extraction were done with hexane. The yield of oil varied from 59.11 to 59.44.The experiments were repeated three times and data were recorded (table-2).

Fig.2. Extraction of oil from the seeds of *Olax scandens* using Soxhlet apparatus

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Table-2: Oil extraction from the kernels of Olax using three different solvents.

Sl no	Quantity of seeds(g)	Solvent used	Oil extracted(ml)	% of oil extracted
1	100	Hexane	59.5	59.5
2	100	Ether	58.8	58.8
3	100	Chloroform	55.6	55.6

2.2 Removal of alkaloids and gums

A reaction glass apparatus was designed for separation of gum/resin from the crude olax oil. Isopropanol was added to oil in the ratio Oil: Isopropanol: 2:1 stirred mechanically and allowed to stand overnight for settling down of the gums and resins in the reaction apparatus. The supernatant was taken out, centrifuged at 10,000rpm for 15 minutes in Remi Research centrifuge R-23/24 to remove traces of gum in the degummed oil. The degummed oil was collected; the tubes were cleaned for further use. The left out oil in the gum in reaction apparatus was filtered and degummed oil was collected for esterification. The degummed oil (DO) thus obtained is called refined olax oil. After the whole process, up to 94% of the crude olax oil (CO) was converted to (DO).

2.3 Two-step transesterification

Since the reactivity of methoxide radicals is higher than that of ethoxide radicals[8], methanol was used for transesterification of olax oil. For esterification, degummed and alkaloid-free oil (DO) was mixed with sulphuric acid and methanol in the proportion of 50:10:1 (oil:MeOH:H2SO4, v/v/v) and stirred mechanically at 200 rpm at 60^0C for 3 h for esterification. After completion of esterification process, two layers were separated within 30 min. The lower layer was discarded and followed by neutralization with methanolic costic soda solution and methanol was recovered from oil. The neutral oil was then mixed with sodium hydroxide and methanol in a ratio of oil:alkali:methanol (25:0.2:5) and stirred well mechanically at 200–250 rpm for 4 h at 50^0C. After transesterification, oil was separated from lower layer by separating funnel or by centrifugation. Figure 3 shows stages from crude olax oil to transesterified oil.

Fig. 3. 2/1- Crude oil, 2/2- Degummed oil., 2/3- Esterified oil, 2/4-Transesterified oil

2.4 Purification

The transesterified oil was taken in a separating funnel; warm distilled water was added for removal of the Sodium hydroxide; to prevent foam formation, a little amount of Ortho-phosphoric acid (10-15 drops in 500 ml of warm water) was added. The transesterified oil in water was shaken and allowed to stand for some time. The oil being lighter than water occupied the upper layer and water at the bottom layer was drained from the separating funnel. This process was repeated until clear water was obtained. Then the resultant transesterified oil (TEO) was collected from the separating funnel and centrifuged for removal of moisture from oil and was stored for further analysis. After two-step transesterification, 91% of the degummed oil (DO) was converted to

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transesterified oil (TEO) and therefore, overall about 86% of crude oil (CO) was converted to TEO. The pH of the transesterified oil was noted and other physical and chemical parameters studied.

3. Standardization of physical and chemical properties.

The different physical and chemical properties of crude oil(CO),degummed oil(DO), and transesterified oil(TEO) were tested at the bio fuel laboratory of the North-Eastern Institute of science and Technology (CSIR), Jorhat, Assam (Table-3)

Table-3 physical and chemical parameters analysed in the laboratory of (NEIST), jorhat.

Sl. No.	Testing Parameters	Specifications	Olax seed Oil			
			CO	DO	TEO	PD
1	Kinematic Viscosity at 40^{O}C (cSt)	IS:1448[P:25]:1976 (Reaffirmed 2006)	30.5	29.9	5.981	3.64
2	pH	-	9.6	9.6	8	
3	Refractive index at 40^{O}C	IS:548[P:1]:1964 (Reaffirmed 2006)	1.4772	1.4771	1.4833	
4	Specific gravity at 15^{O}C	IS:548[P:1]:1964 (Reaffirmed 2006)	0.911	0.908	0.888	0.90
5	Acid value (mg KOH/gm)	IS:548[P:1]:1964 (Reaffirmed 2006)	2.4	2.1	1.8	0.8
6	Iodine Value		214.21	192.89	103.16	70.50
7	Ash content (%)	IS:1448[P:4]:2008	0.42	0.45	5	
8	Saponification value (mg KOH/g)	IS:548[P:1]:1964 (Reaffirmed 2006)	212	211	340	
9	Water content (%)	IS:326[P:21]:2001 (Reaffirmed 2006)	traces	traces	0.13	
10	Pour point	IS:1448[P:10]:1964 (Reaffirmed 2006)	-15	-14	-15	
11	Distillation Characteristics	IS:1448[P:18]:1964				
	IBP (^{o}C)		72	71	72	
	50% recovery (^{o}C)		320	321	318	
	95% recovery (^{o}C)		-	-	-	

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3.1 Standardization of test

The specific gravity of seed oil was determined by specific gravity bottles method using IS:548[P:1]:1964 at 15°C. The refractive index of seed oils was determined by employing the principle of the critical angle using diffused daylight as suggested in IS:548[P:1]:1964. The experiments were conducted at 40°C. The acid number of oil was determined by the procedures of AOAC. (1975). The iodine number was determined by Hanus iodine method (AOAC, 1975). The saponification number was estimated as per the method of AOAC. (1975).

3.2 Results and discussion

Two-step transesterification lowered specific gravity of *Olax scandens* seed oil (Table 3). Specific gravity of petroleum diesel (PD) was comparable with that of TEO, DO (0.908) prepared in this experiment also had lower specific gravity than that of crude oil. The acid number and free fatty acid of oil was determined by the procedures of AOAC. (1975). Acid value is defined as milligrams of potassium hydroxide necessary to neutralize total acids in 1 g of sample.

$$\text{Acid value} = (V \times N \times 56.1) / W$$

Where, V = volume of potassium hydroxide used, N = normality of Potassium hydroxide, W = weight in g of the sample

$$\text{Free fatty acid as oleic acid, per cent by weight} = (28.2 \times V \times N)/W$$

Two-step transesterification drastically lowered acid values of Olaxoil. Data suggested that overnight incubation of oil with dilute NaOH solution also efficiently reduced the free fatty acid (FFA) content of oil. However, acid value of PD was much lower than both the biofuels and this might be due to high acid value of crude oil.The iodine value is a measure of unsaturation of oils and is expressed in terms of the number of grams of iodine absorbed per 100 g of sample (% iodine absorbed), determined by Hanus iodine method (AOAC, 1975). Though iodine value (IV) was used as a measure of the oxidative stability of oil, it does not include allylic and bis-allylic unsaturated fatty acids. For precise measurement of oxidative stability of oil, parameters like oil stability index (OSI), allylic position equivalent (APE) and bis-allylic position equivalent (BPE) ought to be assayed. But, values of IV give an overall idea about the oxidative stability of oil. Results of this experiment indicated that nature of oil is the major factor affecting the iodine value of the esters. Two-step transesterification decreased the iodine value of Olax oil. Results revealed that iodine value of TEO, prepared from Olax oil, was 50% higher than that of petroleum diesel. This comparatively higher iodine value of transesterified oil may be due to the very high iodine value of crude Olaxoil (Table 3). Iodine value of DO was also significantly lower than that of crude oil, though it was slightly higher than TEO. Acceptable higher limit of viscosity for a biodiesel is 29.5 centipoise. Higher viscosity of Olax oil was due to the presence of higher molecular weight molecules such as triglycerides, polymerized triglycerides. Though, DO had marginally higher viscosity than highest limit, its 10% and 20% mixture with PD had viscosity well under this limit.

3.3 Gas chromatography–mass spectroscopy analysis of DO and ltransesterified Olax oil.

Fatty acid components of methyl esters, present in oils, were identified using gas chromatography–mass spectrometry (Varian, gas chromatography 4000 Ion Trap mass spectroscopy) equipped with VF 5-MS (30 m _ 0.25 mm ID _ 0.25 lm df) column. Helium was used as carrier gas at flow rate of 1 ml/min. The temperatures of both injector and detector were set to 350^0C. The following oven temperature programme was used: initially the temperature was retained at 45^0 C for 1 min., raised from 45^0C to 55^0 C at 1^0 C/ min increment then raised from 55^0 C to 290^0 C at 15^0 C/ min increments and finally kept for 5 min. at 290^0C. Total run-time for each sample was 31 min. Gas chromatograms of DO and TEO were presented in Figs. 4-a and 4-b respectively.

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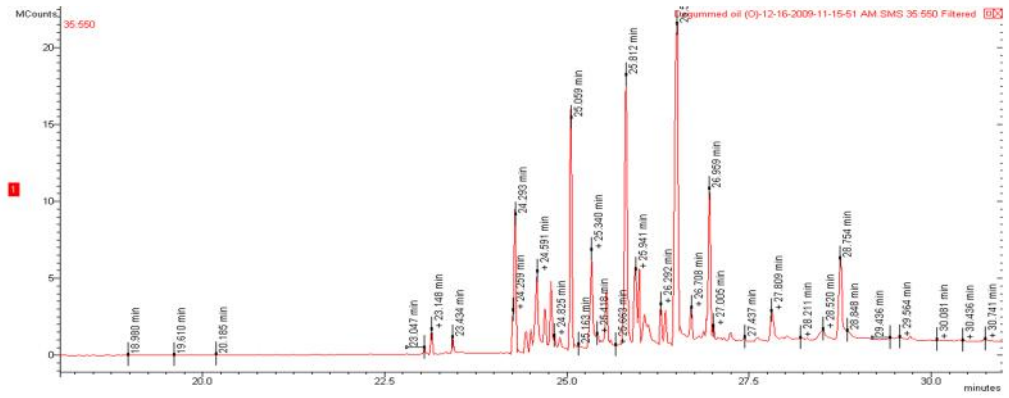

Fig. 4 a. GC-MS Chromatogram of Degummed *Olax* oil

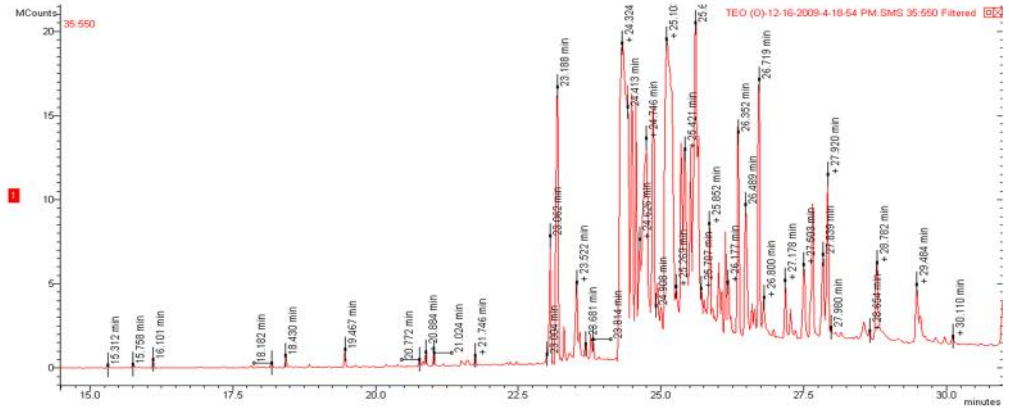

Fig. 4 b. GC-MS Chromatogram of Transesterified *Olax* oil

Analysis of fatty acids and their methyl esters of DO and TEO of Olax oils by GC–MS revealed that both FFA and FAME were present in all the oil samples, but their proportions varied in different oils. Comparison of peak areas suggested that the concentrations of unsaturated fatty acids (appeared after retention time (RT) 23 min during GC–MS analysis) in the modified Olax oils were much higher than that of saturated fatty acids. Methyl esters of eicosanoic acid (RT: 24.72), linolaic acid (RT: 24.264), oleic acid (RT: 24.290) and palmitolic acid (RT: 24.733) were detected in these oils. However, concentration of methyl eicosanoate (RT: 24.72 min) was the highest in these oil samples. Transesterification increased the concentration of these FAME and lowered the concentration of corresponding FFA in oil. Comparatively higher concentrations of FAMEs might be responsible for lower viscosity of transesterified oil as compared to DO and crude oil. Several additional FAMEs names methyl esters of palmitic acid (RT: 23.181), linoleic acid (RT: 24.25) and arachidonic acid (RT: 27.877) etc. were detected in TEO of Olaxoil. CO possibly contains palmitic acid, linoleic acid and arachidonic acid as free fatty acids and two-step transesterification converted those fatty acids to their methyl esters.

4. Engine setup

A four stroke, water cooled and single cylinder engine coupled with edicurrent dynamometer was used for present study Fig. 2. The engine was computerised with engine soft (software) to measure the engine performance parameters. AVL gas analyser was employed to note the exhaust emissions such as carbon

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dioxide, hydrocarbon, carbon monoxide, oxygen, and nitrous oxides. Performance and emission parameters were noted for TOOD10, TOOD20, DO, using petroleum diesel as base line fuel. The test was conducted at 1500 rpm with varing loads. Table 4 shows the engine specifications.

Table 4 Engine specification

Engine	Kirloskar TV1
General details	4 stroke CI water cooled single cylinder computerised
Bore x Stroke	87.5 mm x 110 mm
Compression ratio	17.5 : 1 (varing from 16:1 to 18:1)
Displacement	661 cc
Power	3.5 kW
RPM	1500

5. Engine performance analysis

5.1 Brake power

The variation of brake power with load is shown in Fig. 5. The brake power of TOOD10 stands high subsequent to petroleum diesel with increasing load on the engine. TOOD20 shows slight inferior values subsequent to TOO. All these facts is addressed to higher viscosity, flash or fire point and low calorific value of blended TOO than that of petroleum diesel. However the power loss is very negligible close to 2% to 5%. The low temperature operability persists in using TOO in absolute mode. Hence TOOD10 and TOOD20 can be recommended for fuelling diesel engines without any modification or adulteration nevertheless negligible power loss. Absolute TOO can be used in diesel engines with little modification in engine hardware or preheating up to 60-100^{0}C.

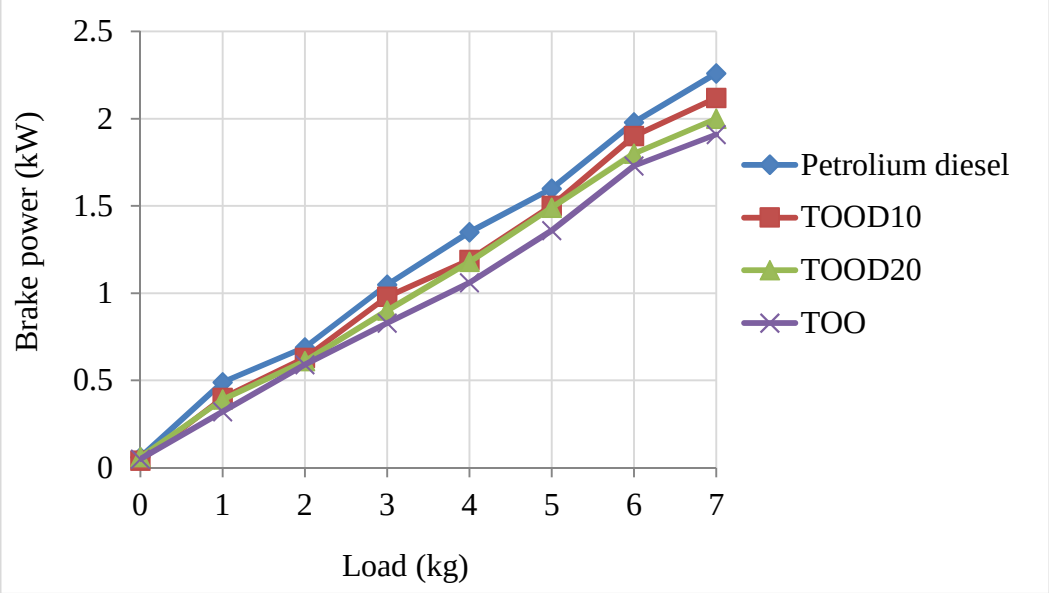

Fig. 5 Variation of brake power with load

5.2 Brake mean effective pressure

The variation of bmep with load is shown in Fig. 6. The bmep of absolute TOO lies below subsequent to petroleum diesel, TOOD10 and TOOD20 with increasing load on the engine. This is attributed to higher viscosity, flash point and low calorific value than that of petroleum diesel. A pressure loss nearly equal to 4% is obtained at the highest load of 7 kg performed by the engine. Furthermore the low temperature operability of absolute TOO is still imperative. However TOOD10 and TOOD20 show a pressure loss of 1% to 2% at the

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same load with better cold flow properties close to petroleum diesel. Hence TOOD10 and TOOD20 can be recommended as an absolute fuel for diesel engines without any modification or adulteration, nevertheless negligible pressure loss. Absolute TOO can be used in diesel engines with little modification in engine hardware or preheating up to 60-100^{0}C.

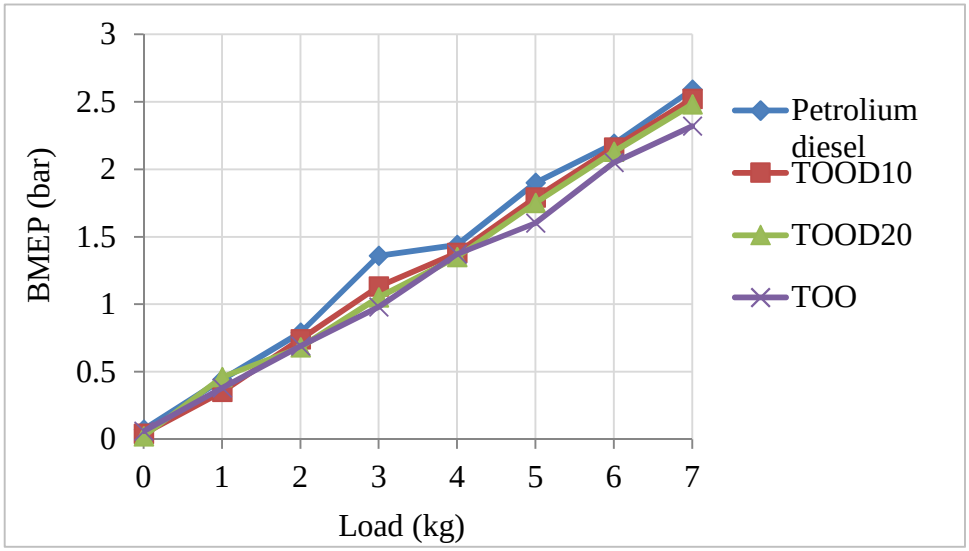

Fig. 6 Variation of BMEP with load

5.3 Brake thermal efficiency

The variation of brake thermal efficiency with load is given in Fig. 7. Brake thermal efficiencies of TOOD10 lies over that of TOOD20, and absolute TOO subsequent to petroleum diesel with increase in load on the engine. TOOD10 shows a thermal efficiency loss near to 2% and TOOD20 shows an efficiency loss of 3%. Absolute TOO shows an efficiency loss about 5%. This may be attributed to higher viscosity, flash point and low calorific value than that of petroleum diesel. However the low temperature operability of absolute TOO is imperative. Hence TOOD10 and TOOD20 can be recommended as an absolute fuel for diesel engines without any modification or adulteration, nevertheless negligible loss. Absolute TOO can be used in diesel engines with little modification in engine hardware or preheating up to 60-100^{0}C.

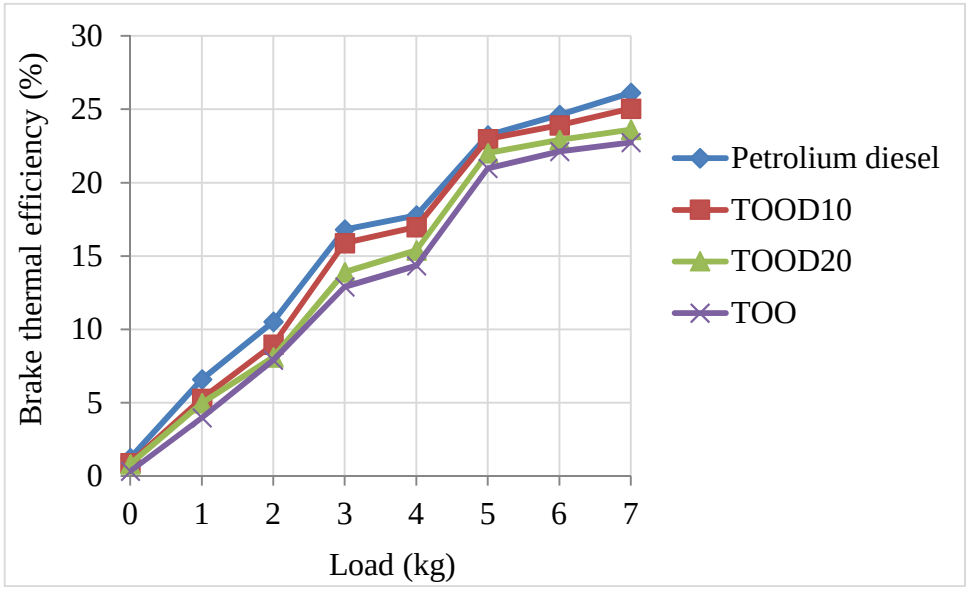

Fig. 7 Variation of brake thermal efficiency with load

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5.4 Specific fuel consumption

The variation of specific fuel consumption with load is presented in Fig. 8. With increase in load, the fuel consumption per unit power generation decreases which is a desired engine performance. TOOD10 has a slightly more fuel consumption than that of TOOD20.Absolute, TOO has slightly more fuel consumption than that of petroleum diesel which may be attributed to a low calorific value. Hence TOOD10 and TOOD20 can be recommended as an absolute fuel for diesel engines without any modification or adulteration, nevertheless slightly higher fuel consumption per unit power generation. Absolute TOO can be used in diesel engines with little modification in engine hardware or preheating up to 60-100^{0}C.

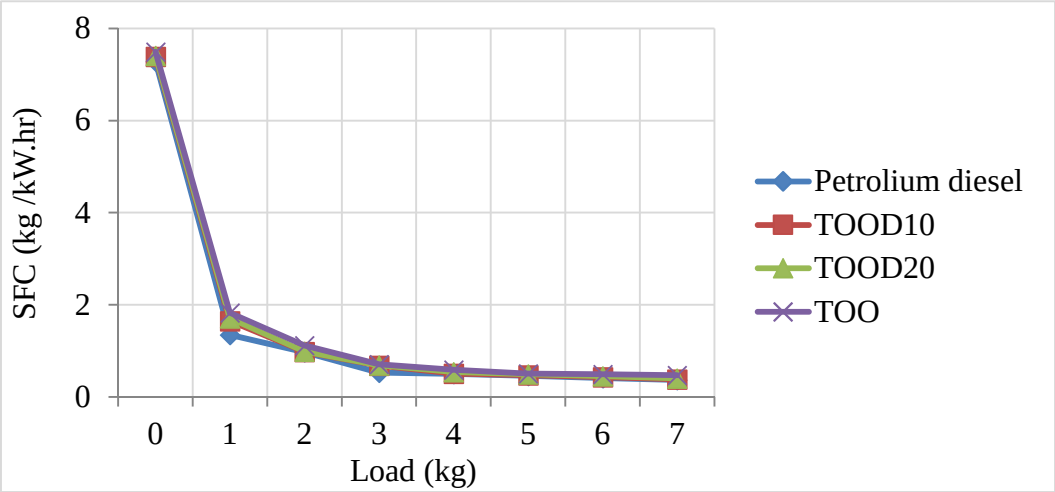

Fig. 8 Variation of specific fuel consumption with load

6. Engine emission analysis

6.1 Emission analysis of TOOD10

The emission comparison of TOOD10 with petroleum diesel is shown in Fig. 9. The combustion of TOOD10 reveals emissions of CO and CO_2, less in comparison to petroleum diesel. Hazardous unburnt hydrocarbon and nitrous oxide is also less than that of petroleum diesel. Free oxygen release is 2% more than that of petroleum diesel which indicates a proximal combustion to petroleum diesel with fewer emissions. Hence TOOD10 can be recommended as an environmental friendly absolute fuel in diesel engines without any modification or adulteration.

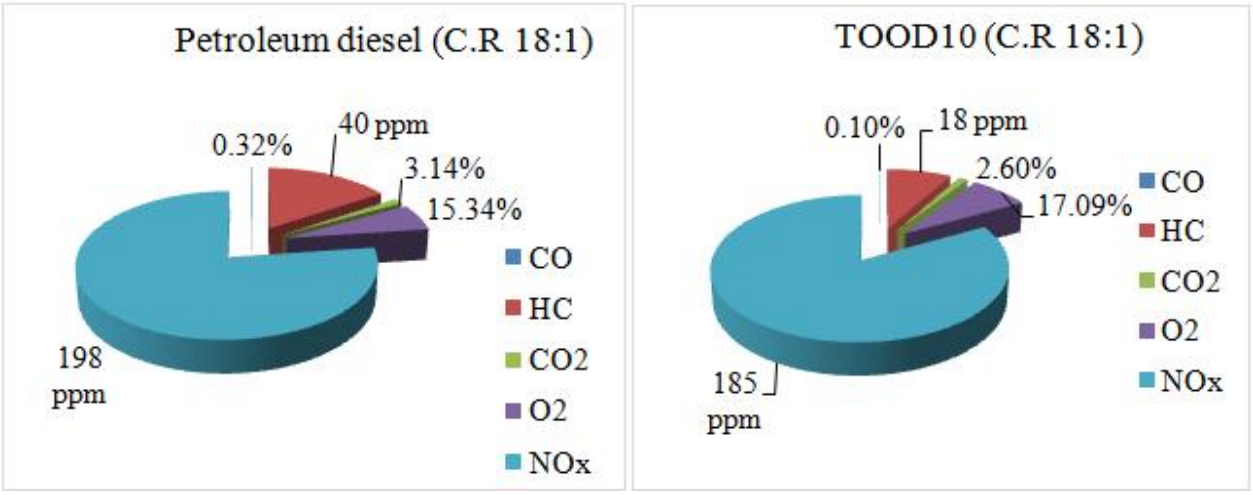

Fig. 9 Emission comparison of TOOD10 with petroleum diesel

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6.2 Emission analysis of TOOD20

The emissions comparison of TOOD20 with petroleum diesel is shown in Fig. 10. Combustion of TOOD20 depicts emissions of CO and CO_2, less in comparison to petroleum diesel. Hazardous unburnt hydrocarbon and nitrous oxide is also less than that of petroleum diesel. Free oxygen release is 2% more than that of petroleum diesel indicates a proximal combustion to petroleum diesel with fewer emissions. Hence TOOD20 is the best absolute fuel next to TOOD10 and petroleum diesel in diesel engines without any modification or adulteration.

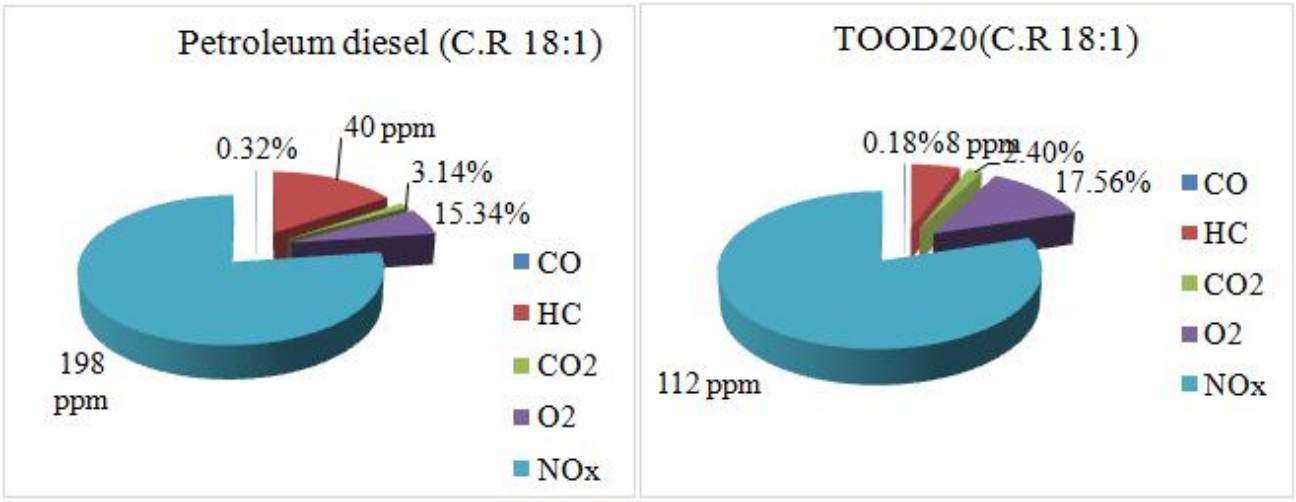

Fig. 10 Emission comparison of TOOD20 with petroleum diesel

6.3 Emission analysis of absolute TOO

The emission comparison of TOO with petroleum diesel is shown in Fig. 11. Combustion of absolute TOO depicts emissions of CO and CO_2, less than that of petroleum diesel. Hazardous unburnt hydrocarbon and nitrous oxide is less than that of petroleum diesel. Free oxygen release is nearly 2% more than that of petroleum diesel indicates a proximal combustion to petroleum diesel with fewer emissions. However poor cold weather operability is still imperative. Preheating up to 60-100^{0}C may mitigate the problem. Hence absolute TOO can be fuelled to diesel diesel engines with little modification in hardware design. Exhaust gas may be utilised for preheating which needs an aesthetic change in muffler design.

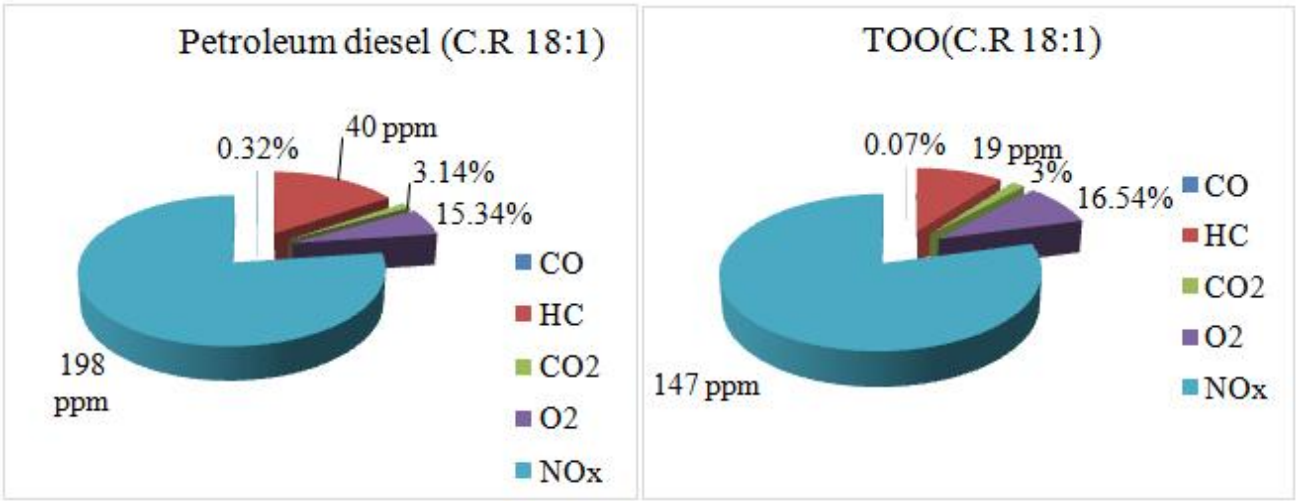

Fig. 11 Emission comparison of TOO with petroleum diesel

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7. Conclusion

An appropriate fuel development, the production of the corresponding biodiesel (methyl esters) of *Olax scandens* Roxb oil with evaluation of physical and chemical properties, fatty acid composition with GC-MS analysis and standardisation of properties was investigated in this work. The properties of the methyl esters (Olax oil) and its blends with petroleum diesel were experimentally observed to be close to those of petroleum diesel fuel. The effects of biodiesel in absolute mode and as an additive to petroleum diesel fuel on performance and emission characteristics of a single cylinder constant speed compression ignition engine have been investigated, and compared with the baseline diesel fuel. The main conclusions of this study are:

1 The existing diesel engine performs satisfactorily on TOOD10 and TOOD20 without any significant engine hardware modification.

2 The engine performance with biodiesel does not differ greatly from that of diesel fuel. A little power loss, combined with an increase in fuel consumption, is often encountered due to the lower calorific value of the biodiesel.

3 In view of the petroleum fuel shortage, TOOD10 and TOOD20 can certainly be considered as an esoteric potential saving up to 20% of global petroleum consumption.

8. Future work

Seedlings and vegetative propagated plants of *Olax scandens* are maintained in the nursery for experimental plantations. Seeds collected from different places of Odisha (India) namely Charichhak in Boudh, Banigochha in Nayagarh and Munduli in Cuttack districts are sprouted and nurtured in science foundation for rural and tribal resource development nursery (Fig.12) to verify and confirm the results for recommending transesterified Olax oil as an additive to petroleum diesel.

Fig. 12 Seedlings and vegetative propagated plants of *Olax scandens*

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