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Gas-Liquid Process, Thermodynamic Characteristics (19 Blends), Efficiency & Environmental Impacts, SEM Particulate Matter Analysis and On-Road Bus Trial of a Proven NOx Less Biodiesel

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1. Introduction

Biodiesel has gained worldwide attention as renewable and blending agent with some lower gas emissions, besides a slight increase of NO_x emission (Michael & Robert ,1998)in the exhaust gas compared to the petroleum diesel. Vegetable oils (Srivastava & Prasad,2000; Prasad & Mohan,2003) namley soybean, sunflower, cottonseed and rapeseed have been examined for fuel without/ with a small modification in the engine. A number of problems, mainly high viscosity, are associated with vegetable oils when directly used as fuel in the CI engines (Agarwal , 1998; Sinha & Misra , 1997; Roger & Jaiduk 1985). It is difficult to reduce particulate matter (PM) and oxides of nitrogen (NO_x) (Mohamad and et al., 2002) simultaneously owing the trade-off between NO_x and PM. Moreover, methyl esters of vegetable oils are sulphur free and possess good lubricating properties (De-Gang et al., 2005). Depending upon the climate and the soil conditions, different countries looking for different type of vegetable oil(Goering et al., 1982; Fernando et al., 2003; Antolin et al., 2002; Freedam et al., 1986; Noureddini & Zhu, 1997; Mohamad et al., 2002; Yi-Hsu & Shaik, 2005; Sukumar et al., 2005) used for the biodiesel production; soybean oil in US, rapeseed oil in Europe, palm oil in Malaysia and Indonesia, and coconut oil in the Philippine are being considered (Barnwal & Sharma , 2005).

In India, out of more than 125 million tons (Arumugam et al., 2003) of rice production, about 6 million tons of rice bran and 1 million ton of RBOBD is produced annually (Table 1). General characteristics of refined rice bran oil are as follows: sp gr, 0.916 kg/m^3 ; ref index 1.47; Cloud index, 17; iodine value, 99-108; saponification value, 180-190; unsopanifiable matter, 3.5(%); smoke point, 213 °C; and fire point, 352 °C. General properties of vegetable oil based biodiesel (Table 2) show many variations that might be due to the conversion to biodiesel through different raw materials and different processes.

	Country	Rice	Rice bran	Oil	
	China	181	14.5	2.47	
	India	137	6.8	1.02	
	Indonesia	50	4.0	0.68	
	Bangladesh	38	3.0	0.51	
	Vietnam	32	2.6	0.44	
	Thailand	24	1.9	0.32	
	Myanmar	20	1.6	0.27	
	Philippines	13	1.0	0.17	
	Japan	11	0.9	0.15	
	Brazil	10	0.8	0.14	

Table 1. Annual production (metric million tons) of rice, rice bran and oil in the world.

This chapter presents process for rice bran oil biodiesel (RBOBD) production, composition and physico-chemical properties of RBOBD, engine test results, scanning electron microscope (SEM) image, particulate matter in exhaust gas and emission reductions.

Table 2. Properties of Biodiesel from vegetable oils.

2. Process description

Oil having specific gravity in the range of 0.85 – 0.96 and iodine value not exceeding 208 is heated to a temperature not exceeding 120°C for not less than 2hrs to adjust the moiture content at a level not exceeding 0.5% and is transesterified using 8 to 42% w/w, of alcohol of general formula R-OH, where R represents (C_nH_{2n+1}) , n being any integer between 1 and 5, by known method in presence of not more than 0.5% w/w, of a known catalyst, at a temperature higher than the boiling point of the alcohol but not exceeding 215°C for not less than 30 minutes under continuous turbulent condition at rpm in the range of 100-150 to get a mixture of ester and glycerol. The Reynolds number (N_{Re}) is maintained at not less than 4000 irrespective of the type of the reactor. The mixture of ester and glycerol is subjected to separation by known method for a period of not less than 4 hrs and the top layer ester is purified by conventional method for a period of not less than 8hrs. The process of separation as well as purification is repeated for not less than three times in succession to get biodiesel.

Fig. 1. Lab scale experimental setup

In lab scale experimental setup Fig.1, RBO was taken in the continuous stirred tank glass reactor (1 l) with reflex condenser, temperature control and agitation control setup. In another reactor, NaOH (50 g) was dissolved in methanol (300 ml). This solution was added slowly at the reactor maintained at 65-70 \degree C for 150 min. Then the entire mixture kept in the separating funnel. The top layer, biodiesel, is taken for the removal of methanol in the ROTO vacuum distiller. Then the methyl ester washed of distilled water (1 l) in the same reactor for 30 min. After washing, top layer in the separating funnel has to be washed with saline water for two times. Finally, clear biodiesel was kept in the oven for 4 h at 100° C. The ready to use biodiesel few samples shown in Fig.2.

Fig. 2. Ready to use biodiesel samples

In the bench scale level, Rice Bran Oil (RBO) experiments were carried out with standardized process conditions in high-pressure Parr Reactor (Fig.3.) inbuilt sophisticated controlling systems of reactor (20 l). Rice Bran Oil Biodiesel RBOBD (>150 l) was produced.

Fig. 3. Bench Scale lab Parr Reactor

In each lot, biodiesel sample has been analyzed for the conversion, fuel properties and composition. Quality consistency conformed by C_{13} and Proton of JEOL ECA 500 MHz NMR analysis and the composition by GCMS. All chemicals used were of LR/AR grade. A typical NMR spectrum show in Fig.4.

The brief process description has been followed at the Pilot-scale preparation of biodiesel (Fig. 5. (a)), which was used for on-road trails from rice bran oil is following.

Rice bran oil is filtered to remove any impurities. 69 lit. of moisture free refined oil is taken in a Pilot Plant scale reactor (Fig. 5. (b)) of capacity 120 lit. Fitted with a reflux condenser and heated with agitation to 65ºC. Then 345 gms of sodium hydroxide, 20.7 lit. of methanol are mixed separately and the mixture is slowly added to oil at 65ºC.

The reaction mixture is mixed well, temperature is maintained at 65-70ºC throughout the reaction and the reaction time is 150 min. When the reaction is complete, the contents are allowed to cool and transferred to a separating tank. After overnight settling, the mixture gets separated into two layers due to density difference.

The bottom layer-Glycerol is separated. The top layer - biodiesel is distilled at 65ºC to recover unreacted alcohol. Then the methyl ester is washed for 30 minutes at 50ºC with equal volumes of 0.1% dil. acetic acid to remove any traces of un reacted alkali. In case of emulsion formation after washing, saline water is used for second washing. The pH of the ester layer is adjusted to neutral while washing. After washing, the layers are allowed to settle for 30 min. The top layer is separated and biodiesel is dried in a pan drier for 2 hrs at 110ºC. Then it is filtered to separate any traces of impurities. The final ready to use biodiesel product is found to be 60 lit.

Fig. 5. (a) Pilot-scale preparation of biodiesel (Fig. 5. (b))

Few thousand liters of Biodiesel produced in the pilot level which is used as feul in the on-Road bus trails. More than 26000 km exprimental trials were carried out in the Metropolitan Transport Corporation (MTC) buses in Chennai, Government of Tamil Nadu. Few clipings of MTC bus trails are shown in Fig.6. Initialy four buses have been taken for on-road trials in a single root but fuelled with different biodiesl percentage namely, B5, B10,B20 and B50. Then all the buses fuelled with 100% Biodiesel. The MTC, government of Tamil Nadu, has submitted the officeal report about the on –raod trials. The Fig. 7 showing the highligts signed by the MTC highre officails of the report in the reginal language namely TAMIL and Fig 8. Showing its translation in English.

3. Engine testing and exhaust gas analysis

RBOBD was tested in Kirloskar four stroke, single cylinder, water cooled, direct injection IC engine (Fig.9) with following parameters: bore, 80 mm; stroke, 110 mm; swept volume, 553 cm³; clearance volume, 36.87 cm³; compression ratio, 16.5:1; rated output, 3.7 kW at 1500 rpm; rated speed, 1500 rpm; injection pressure, 240 bar; fuel injection timing, 24 BTDC; type of combustion chamber, hemispherical open; lubricating oil, SAE 40; connecting rod length, 235 mm; valve diam, 33.7 mm; and maximum valve lift, 10.2 mm.

Fig. 5. (b) Pilot Plant scale reactor

Fig. 6. Few clipings of MTC bus trials

Fig. 7. Showing the highligts signed by the MTC highre officails

Fig. 8. Highlights Translation in English

Fig. 9. A Test engine

DYNALOG, PCI 1050 system has been used for digital data acquisition during the engine trial. Online engine calibration (Fig.10) with a special software namey "Engine-soft". The very brief specifications are Number of channels (16); Resolution (12- bit A/D); Input range $(\pm 10 \text{ V}, \pm 5 \text{ V}, 0 - 10 \text{ V})$; Accuracy (0.025%) and Conversion time (8 µs).

Engine was coupled to a swinging field separating exciting type DC generator and loaded by electrical resistance bank to apply various load. An iron-constantan thermocouple measured exhaust gas temperature and mercury thermometer measured cooling water temperature. Carbon monoxide (CO), nitrous oxide (NO*x*) and hydrocarbons (HC) were measured by DELTA 1600-L and MRU OPTRANS 1600, a fully microprocessor controlled system employing nondestructive IR technique. A U-tube manometer measured specific fuel consumption. TI diesel tune, 114-smoke density tester measured smoke particulate number.

Fig. 10. Engine Calibration with "Engine-Soft"

The engine was started on neat diesel fuel and warmed up till liquid cooling water temperature was stabilized. During the performance of each trail, data were collected on time taken for 10 ml of fuel, load, exhaust gas temperature, cooling water inlet and outlet temperature, CO, CO_2 , O_2 , HC, NOx, smoke and sound. Graphical comparisons are described in the results and discussion. Smoke samples were collected in a white filter paper; this was taken for Scanning Electron Microscope (SEM) analysis to find the size of the particulate matter and to visualize the quantity of agglomeration. The SEM image is shown

in Fig 10. Based on the data, specific fuel consumption, indicative thermal efficiency, brake thermal efficiency, mechanical efficiency and total fuel consumption were estimated. Similar procedures were repeated for RBOBD.

4. Results and discussion

4.1 Process conditions and compositions

RBOBD contains (GC-MS) esters of following acids: palmitic, 16; stearic, 2; oleic, 42; linoleic, 38; linolenic, 1.4; and arachidic, 0.6%. Quality consistency was conformed by C_{13} and Proton of JEOL ECA 500 MHz NMR. Physico-chemical characteristics of RBOBD and its 19 blends (Table 3) show that most of the parameters comply with international standards of biodiesel. An NMR spectrum is already shown in Fig 4.

4.2 Comparison of Brake Power and Specific Fuel Consumption (SFC)

SFC of diesel, RBOBD and its various blends at different load (0-3.78 kW) were estimated and graphical representaion is shown in Fig 11. In comparison to diesel, a slight increase (10-15%) of SFC was found for RBOBD, B40, B50, B60 and B80 throughout all loads . At the maximum load (3.78 kW), SFC of B60 was found higher in comparison to the other blends. In particular to B20, the result shows that SFC was lower than diesel and other RBOBD and its blends in all the loads. The maximum increase (11.6%) was found at load 1.89 kW.

Fig. 11. Comparison of brake power and specific fuel consumption

4.3 Comparison of Brake Power and Fuel Consumption Time (FCT)

FCT of RBOBD and its various blends have been found less than the FCT of diesel, graphical representaion is shown in Fig 12. Slight decrease (5-10 %) of FCT was found for all fuels. Maximum decrease of FCT (12.5 %) was found at the brake power of 3.78 kW for B50 and B60. But, in particular, for B20, there was slight increase of FCT for the entire range of brake power. Maximum increase of FCT (12 %) was at 1.89 kW and minimum (3 %) at 3.78 kW.

Table 3. Physico-chemical characteristics of RBOBD and its 19 blends

Fig. 12. Comparison of brake power and fuel consumption time

4.4 Comparison of Brake Power and Total Fuel Consumption (TFC)

TFC increased with increase of brake power, graphical representaion is shown in Fig 13. A maximum increase (13%) was at the load 1.89 kW. TFC's of RBOBD blends, B40, B50, B60 and B80, are higher (5-10%) than the TFC of diesel. But B20's TFC is slightly lesser than diesel and all the other RBOBD blends from the minimum load to the maximum load. Maximum TFC decrease (10%) was observed for B20 at 1.89 kW. Overall trend shows that the percentage decrease in TFC is inversely proportional to the brake power. At the maximum load, the increasing order of TFC is B20, Diesel, B40, B80, RBOBD and B60.

Fig. 13. Comparison of brake power and total fuel consumption

4.5 Comparison of Brake Power and Exhaust Gas Temperature (EGT)

EGT increases with increase of brake power, graphical representaion is shown in Fig 14.

Fig. 14. Comparison of brake power and exhaust gas Temperatur

In comparison with EGT of diesel in each load, EGT of RBOBD and all the blends were higher. The highest value of EGT (395°C) was found with B40 at the maximum load of 3.78 kW, whereas corresponding value of normal diesel was 294°C only. Percentage increase of EGT of RBOBD decreased with the increase of load. Maximum increase (40%) of EGT was found at lower load of zero brake power. EGT of B40, B50, B60 and B80 were 350-400°C at the maximum load. The percentage increase (20-40%) of EGT of B60 was higher than all the loads. EGT of B20 was found to be slightly lower than EGT of normal diesel in all loads (0- 3.78 kW). The minimum EGT decrease (9.5%) and maximum decrease (16.3%) of RBOBD and blends were found at 1.1 Kw and 2.98 kW respectively as compared to diesel. EGT of B20, B50 and B80 were found to be lower than EGT of diesel at 0-2.97 kW.

4.6 Comparison of Brake Power and Brake Thermal Efficiency (BTE)

BTE increases with increase of load, graphical representaion is shown in Fig 15.

Fig. 15. Comparison of brake power and brake thermal efficiency

BTE of RBOBD was less (5%) than diesel with respect to all loads. All other RBOBD blends (B40, B50, B60 and B80) were within 5 % only. Maximum reduction (20%) of BTE was found at 1.89 kW and minimum increase (7%) at 2.9765 kW. BTE of B20 was found higher than BTE of normal diesel in all loads. At maximum load (3.78 kW), BTE for B20 (29.7%), B40 (28.6%), B50 (25.6%) and B60 & B80 (25.5%) are higher than BTE of diesel.

4.7 Comparison of Brake Power and Indicative Thermal Efficiency (ITE)

ITE of RBOBD, B50, B60 and B80 were found lower than ITE of diesel, graphical representaion is shown in Fig 16 . ITE of B20 and B40 were slightly more than ITE of diesel. Maximum ITE (57.9%) was found for B20 at 1.89 kW. Reduction (5-15%) was observed in ITE of various blends. But the ITE of all fuels shows that values are almost steady throughout the entire brake power.

Fig. 16. Comparison of brake power and indicative thermal efficiency

Fig. 17. Comparison of brake power and mechanical efficiency

4.8 Comparison of Brake Power And Mechanical Efficiency (ME)

ME of the engine run with RBOBD and various blends increases with increase of brake power in comparison with normal diesel, graphical representaion is shown in Fig 17. ME of RBOBD was less (5%) than diesel with respect to all loads. There was a slight increase of ME for all RBOBD blends in the order of B40, B50, B20, B80 and B60. Highest ME (68%) observed for B60 was at the maximum load. Overall trend shows that percentage increase of ME was decreased with increase of load. The result of B60 shows that the minimum increase (12%) was found at maximum load and maximum increase (24%) at minimum load.

4.9 Comparison of Brake Power and Hydrocarbons (HC)

HC increased with increase of brake power, graphical representaion is shown in Fig 18. RBOBD and its five blends showed lower HC (50-100%) than diesel. Reduction of HC (60%) of RBOBD and its blends are at the maximum load; B50 shows higher HC reduction than other blends at all loads.

Fig. 18. Comparison of brake power and hydrocarbons

4.10 Comparison of Brake Power and CO emission

CO emission increased with increase of brake power, graphical representaion is shown in Fig 19. There was decrease of CO emission (50-80%) of RBOBD and its all five blends in comparison with CO emission of diesel. Reduction (>50%) of CO emission was found at 2.97 kW. Within blends, B20 shows lower CO emission (70-80%), which decreased with increase of load.

4.11 Comparison of Brake Power and CO2 emission

CO2 emission increased with increase of brake bower, graphical representaion is shown in Fig 20. There was slight increase in $CO₂$ emission of RBOBD and its blends as compared to diesel. More variation of percentage increase was found within all RBOBD blends at the load 1.89 kW. The overall trend shows that the $CO₂$ emissions are similar to diesel at each load.

Fig. 19. Comparison of brake power and carbon monoxide emission

Fig. 20. Comparison of brake power and carbon dioxide emission

4.12 Comparison of Brake Power and NOx emission

 NO_x increased with increase of brake power, graphical representaion is shown in Fig 21. There was a reduction (10-55%) of NO_x of RBOBD and its blends in comparison with NO_x values of diesel in each load. The trend shows that at minimum load, percentage reduction was maximum and at the maximum load, the percentage reduction of NO_x was minimum. The percentage reduction of NO_x decreased with increase of brake power. NO_x values at maximum load (3.78 kW) were found to be: diesel, 942; B80, 858; B50, 782; RBOBD, 753; B20, 677; and B60, 660 ppm. At 3.78 kW, maximum reduction (28%) was found for B20 and minimum (8.9%) for B80.

Fig. 21. Comparison of brake power and nitrogen oxides emission

4.13 Comparison of Brake Power and O²

O2 decreased with increase of brake bower, graphical representaion is shown in Fig 22. Deviations (5-10%) were found for RBOBD and its blends. At maximum load, O_2 (6.5) in B50 was less $(25%)$ than $O₂(9.1)$ of diesel.

Fig. 22. Comparison of brake power and Oxygen

4.14 Comparison of Brake Power and sound

Sound or noise increased with increase of load, graphical representaion is shown in Fig 23. Sound values of RBOBD and its blends are found lower (15-30%) than the sound values of diesel throughout the brake power. Within comparison of RBOBD and its blends, there was not much change in sound in all the loads. The minimum decrease (13.6%) was observed at the minimum load, and the maximum decrease (30%) at the maximum load (3.78 kW). At the higher load, sound reduction (21-30%) for RBOBD and all of its blends compared to diesel.

Fig. 23. Comparison of brake power and sound

Fig. 24. Comparison of Scanning Electron Microscope Image (SEM) of Diesel and RBOBD

4.15 Comparison of SEM image

During the engine trails, smoke was collected in the white filter paper. Few samples at the maximum load were taken for SEM analysis at different resolutions. SEM shows much reduction in the particulate matter in the biodiesel as compared to diesel (Fig. 24). The particle size in the smoke of RBOBD is less than $0.5 \mu m$.

5. Conclusions

This is most ambitious and successful technology development initiative for alternative energy options, which is the important global agenda, and will be good for the environment. These are the key component for energy security and have positive economic, social and environmental impacts. The conventional fossil fuel energy sources are the major cause of climatic changes, this biodiesel leads to minimize the emission to the environment and sustainable society. The biodiesel production with this technology may cut fossil fuel imports and dependency and thus, free up funds that can be invested in social and economic development. This is the process innovation for the less NO_x emitting Biodiesel. An international Patent has been filed through the Intellectual Property Management Division of Council of Scientific and Industrial Research (CSIR), New Delhi in the year 2003. *(Ref No. IMPD. 0290 NF 2003, PCT / IB 03/ 05349 (21.11.2003, US & JP 20050108927 and World Intellectual Property Organization-WO/2005/052103)., Patent granted Firest in Singapore, Patent Number 119411 on 31st May 2006 and then Australia , patent Number-2003282270 and Sri Lanka Patent Number - 13950*. In India, this is the first technological breakthrough in On-Road trials fuelled with biodiesel were successfully carried out in state government metropolitan Transport corporation (MTC) buses more than 26000 km without any engine modifications. There are 15% increases in the KMPL found after the trials. The MTC bus trials prove that this Biodiesel is substitute for diesel in the real conditions. On-Road trials fuelled with 100% Biodiesel (free biodiesel samples) by owner-driven in different vehicle models, namely, *Toyota Qualis, Mitsubishi Lancer, Bolero, Hyundai Acent etc.*

RBOBD was obtained by the optimized process conditions of transesterification and purification. RBOBD (> 150 l) is produced in Bench-Scale Parr reactor. RBOBD showed 15% increase in SFC, 25% increase in BTE, less exhaust gas temperature, 12% increase in FCT, more ITE and ME, 60% reduction in HC, more than 75% reduction in CO, minimum of 10% reduction in NO_x and 30 % sound reduction. The combustion of RBOBD and its blends are found to be lesser pollutants than compared to diesel. SEM shows reduction of particulate matter and the size of the solid particle is less than $0.5 \mu m$. This study proves that RBOBD is environment friendly alternate fuel for diesel without any engine modification.

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Biodiesel - Feedstocks and Processing Technologies Edited by Dr. Margarita Stoytcheva

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The book "Biodiesel: Feedstocks and Processing Technologies" is intended to provide a professional look on the recent achievements and emerging trends in biodiesel production. It includes 22 chapters, organized in two sections. The first book section: "Feedstocks for Biodiesel Production" covers issues associated with the utilization of cost effective non-edible raw materials and wastes, and the development of biomass feedstock with physical and chemical properties that facilitate it processing to biodiesel. These include Brassicaceae spp., cooking oils, animal fat wastes, oleaginous fungi, and algae. The second book section: "Biodiesel Production Methods" is devoted to the advanced techniques for biodiesel synthesis: supercritical transesterification, microwaves, radio frequency and ultrasound techniques, reactive distillation, and optimized transesterification processes making use of solid catalysts and immobilized enzymes. The adequate and upto-date information provided in this book should be of interest for research scientist, students, and technologists, involved in biodiesel production.

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