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***Argemone Mexicana*: A rarefied Splendour prickly foliage weed of sustainable bio additive for commercial diesel engines**

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Abstract

Using of crop-based foliage (edible oils) to produce sustainable energy increases the volume of low-carbon fuels, but detracts hunger in developing countries by pushing up global food prices and endangering local food security. In present work the authors investigated the possibility of an economic absolute and additive fuel package by pragmatic fuel synthesis from mixed feed stock of non-edible *Argemone mexicana* oil and recycled grease, with improved combustible properties by a strategic novel transesterification method, supplemented by fatty acid composition test, a susceptible engine performance, declining hazardous emissions to well below than that of petroleum diesel. The effects of mixed feedstock bio diesel in absolute and additive mode was ensured by rigorous engine test inferring 10 to 20% blend with petroleum diesel can be fuelled to commercial diesel engines without any adulteration or engine modification in engine hardware system.

Keywords : Foliage, CAO, FFA, FAME, PD, AME10, AME20, TEO, GC-MS

1. Introduction

Argemone mexicana is a rarefied splendour prickly foliage weed belongs to pappaveraceae family, grows widely in many parts of the globe. *Argemone mexicana*, commonly called prickly poppy, is an annual weed mainly known in India among herbalists for its several medicinal properties. *Argemone mexicana* oil has recently been reported as a unique source for biodiesel because of several favourable characteristics (Pramanik et al., 2012). *Argemone Mexicana* thrives on any type of soil, can endure long periods of draught and needs minimum nourishment. It has no serious production problems caused due to insects, pests or browsing by cattle and sheep. Propagation is by seed and the crop is harvested in the same season in just four and half months. The yields expected from *Argemone mexicana* would be close to 2.5 ton/ha, oil content varies from 35 to 40%. The present work investigates the possibility of an economic absolute and additive fuel package by a novel fuel synthesis from mixed feed stock of *Argemone mexicana* oil and recycled grease in laboratory scale, increasing volume of bio diesel production. Fig.1 shows *Argemone mexicana* foliage; at different stages of maturity.



Fig.1 *Argemone mexicana* foliage; at different stages of maturity.

2. Materials and methods

2.1. Novel economic fuel synthesis The present process of producing bio-diesel from mixed feed stock of *Argemone mexicana* oil and recycled grease involves extraction of oil from the seeds, removal of alkaloids and gums, a two step transesterification using dilute acid and crystalline potassium hydroxide as catalyst, characterization of the oil, fatty acid composition by gas chromatography, and a rigorous engine test to ensure the absolute and additive fuel package in laboratory scale. The recycled grease is esterified with methanol in presence of sulphur and dilute HCL (hydrochloric acid) as catalyst. The resultant of esterification is mixed with alkaloid and gum free crude *Argemone* oil (CAO) for transesterification using potassium hydroxide as catalyst. The resultant of transesterification is allowed to cool and the residues are separated by a separating funnel. The transesterified oil is refined by washing with hot water three times and excess methanol was recovered along with the catalyst. In this novel fuel synthesis method the recycled grease is converted to biodiesel, increasing volume of production of biodiesel as shown in flow diagram Fig.2.

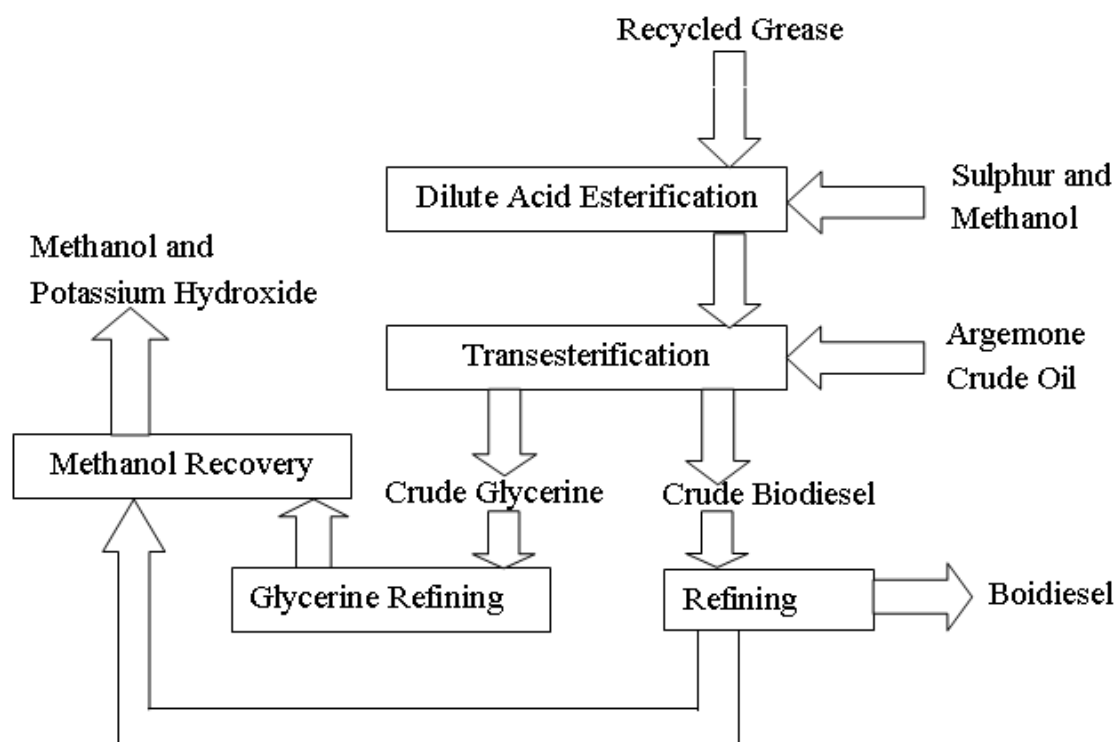


Fig.2 Flow diagram of bio diesel synthesis from mixed feed stock.

2.2 Extraction of Argemone oil

The different samples of Argemone mexicana seeds were sun dried for two weeks, cleaned and weighed. After cleaning the seed samples were cooked in an oven for 1.5 hour and then pressed in a screw expeller for five times at 130 rpm. The residual cake was collected and pressed in the expeller ones again. Now the cake and oil was collected and weighed and oil yield was calculated.

$$\text{Oil Yield} = \frac{AOW}{ASW} \times 100\%$$

Where AOW = Weight of Argemone oil extracted (g), ASW = Weight of Argemone seeds (g)

The oil content was found to be 35% to 40%.

2.3. Removal of alkaloids and gums

Argemone oil thus extracted contains gums and alkaloids such as phosphates, proteins carbohydrates, water residue and resins. The removal of alkaloids and gums meliorates the oxidation stability of biodiesel from Argemone mexicana. The extracted oil of 100ml was mixed with 25ml of phosphoric acid, stirred at 1000 rpm, 60⁰ C, for 30 minutes in a centrifuge machine. The alkaloids and gums resided at the bottom were separated by a separating funnel. The oil thus obtained is washed with distilled water at 100⁰C several times to remove suspended gums. After washing the water content in oil was evaporated by heating the oil at 100⁰C. The oil thus obtained is termed as alkaloid and gum free crude Argemone oil (CAO).

2.4 Two-step transesterification

A magnetic stirrer of 1000 ml capacity facilitating temperature and speed control was selected for esterification and subsequent transesterification reaction. For esterification recycled grease was allowed to react with methanol in presence of sulphur. Initially the stirrer speed was set at 600 rpm to eliminate possibility of mass transfer. The temperature of the stirrer was set at 65⁰ C and catalyst, diluted hydrochloric acid (HCL) was added to the reactor with pressure and stirrer speed set constant. After the reaction time of 90 min a high conversion to ester (biodiesel) was achieved quickly.

The same method was followed for transesterification by allowing the reactant to react with crude Argemone oil (CAO) by changing over to potassium hydroxide (KOH) as catalyst. The recycled grease in this novel process increased the volume of transesterified oil (TEO) or biodiesel. The transesterification requirement ascertained for producing the esters (biodiesel) was shown in Table-1.

Transesterification requirement

Table-1

Transesterification requirement	AOME(Argemone oil methyl ester)
Recycled grease	500gm
Crude Argemone oil	500gm
Methanol (6:1) with respect to oil	150 gm
Temperature	65 ⁰ C (±3 ⁰ C)
HCL (1%)	5 gm
KOH (1%)	5 gm
sulphur	5gm
Time period	90 min
RPM	600

2.5 Characterization of vegetable oils

Physical properties of crude Argemone oil (CAO), its different blends with petroleum diesel (PD), such as AME-10, AME-20(Argemone methyl ester 10% & 20% with PD), and transesterified oil of Argemone (TEO) was measured as per IS & ASTM specifications and compared with that of petroleum diesel as base line fuel, shown in Table-2.

Physical properties of oils

Table-2

Property	Unit	IS:15607 specificati on	CAO	AME10	AME 20	TEO	PD	Test methods
Specific gravity	-----	0.87-0.90	0.928	0.86	0.84	0.88	0.831	IS:1448
Cloud point	⁰ C	-----	8	-3.7	-2.6	-1	-4	ASTM D2500
Acid value	mgKOH/gm	≤ 0.5	19.517	0.45	0.47	1.251	0.367	ASTM D 974
Iodine value	gmI ₂ /100gm	-----	325.92	84.5	86.3	92.61	70.50	ASTM D 1510
Viscosity @ 40 ⁰ C	Cst	3.5-5.0	38.85	3.51	3.89	4.981	3.64	ASTM D445
Flash point	⁰ C	≥ 120	220	68	70	128	65	ASTM D 93
Cetane number	-----	≥ 51	40	55.6	56.4	42-48	45	ASTM D 613

2.6. Gas chromatography–mass spectroscopy (GC-MS)

Fatty acid composition of crude Argemone oil (CAO), and the corresponding ester composition after transesterification(TEO) was investigated employing gas chromatography–mass spectrometry (Varian, gas

chromatography 4000 Ion Trap mass spectroscopy) equipped with Column: VF 5-MS (30m x 0.25mm ID x 0.25um df), helium being the carrier gas at flow rate of 1 ml min⁻¹. The temperatures of both injector and detector were set to 350⁰C. The temperature programme used was: initial retained temperature was at 45⁰C for 1 min., raised from 45⁰C to 55⁰C at 1⁰C min⁻¹ increment then raised from 55⁰ C to 290⁰C at 15⁰C min⁻¹ increments and finally kept for 5 min. at 290⁰C. Total runtime for each sample was 35 min. Gas chromatograms of CAO and TEO were presented in Figs. 5 and 6 respectively.

2.7. Engine test

Engine testing was investigated by fuelling petroleum diesel (PD), crude Argemone oil (CAO), transesterified oil (TEO) and its blends with petroleum diesel at 10% and 20% proportions in a commercial diesel engine having specification given in table-3.

Table-3.

Engine specifications

Description	HA57L165
Type	Diesel four stroke,6cylinder water cooled, direct injection, inline overhead valve
Aspiration	Turbo charged with inter cooler
Maximum output	165KW@ 2500rpm
Maximum torque	800Nm @ 1400-1900rpm
Bore and stroke	104x113mm
Piston displacement	5.759 litres
Compression ratio	17.5 :1
Firing order	1-4-2-6-3-5
Fuel injection type	Common rail system CPN2.2(CRS)

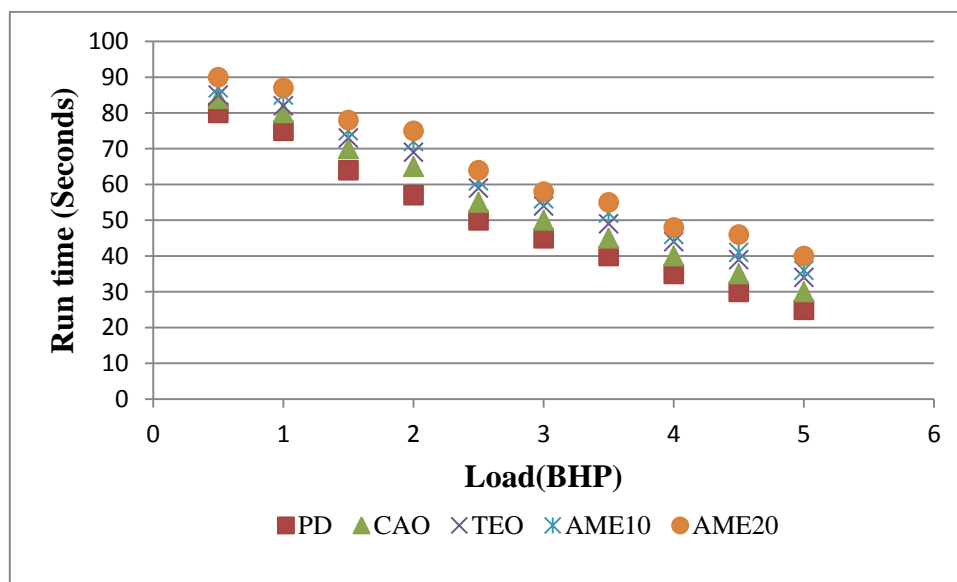


Fig. 3(a) Engine run-times for 20 ml oil at different load factor

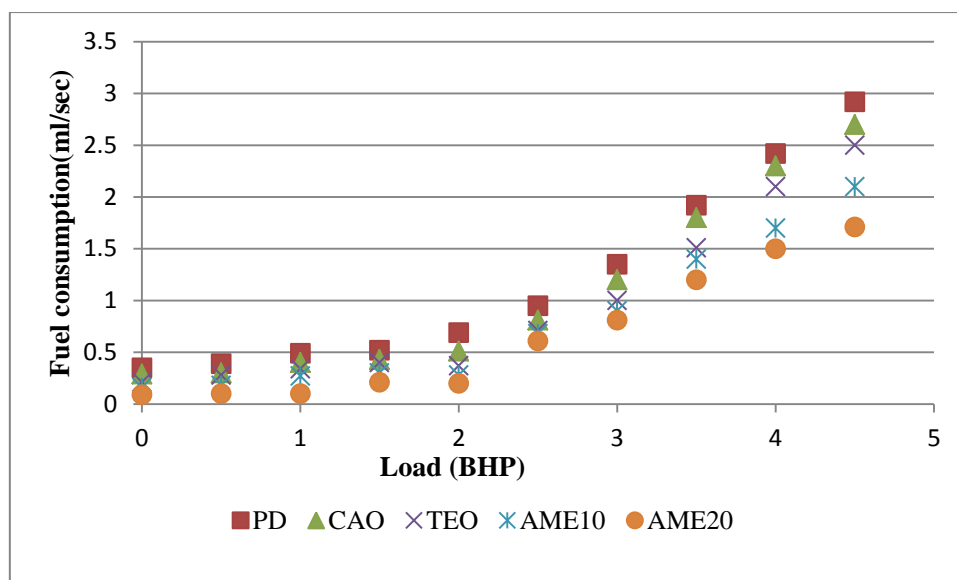


Fig. 3(b) Fuel consumption per second at different load factor

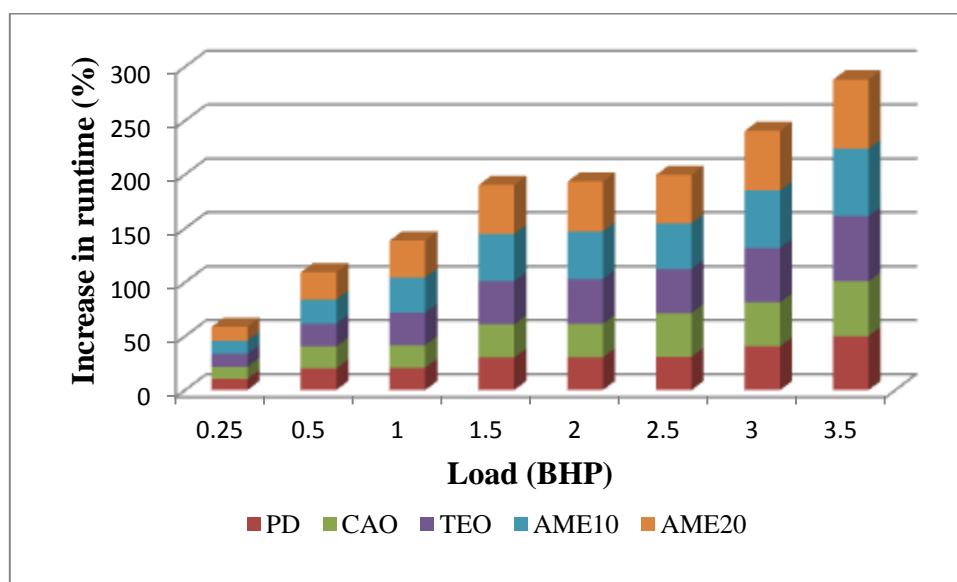


Fig. 4. Percent increase in run-time for different oils at variable load.

Engine runtime (seconds), fuel consumption (ml/sec), percentage increase in run time (%) with increase in load(BHP) were compared with reference to petroleum diesel being baseline fuel as shown in Fig.3(a),3(b) and Fig.4 respectively. The engine performance test was done according to BIS: 5994-11 (Test code 1979). Hazardous emissions of the engine like carbon monoxide (CO), carbon dioxide (CO₂), nitrogen oxides (NO_x) and hydrocarbons (HC) were measured using AVL make gas analyser as shown in Fig.7(Table-4). Fuel consumption was measured by U-tube manometer.

3. Results and discussion

3.1 Properties of oil

Successive esterification and transesterification employing mixed feedstock of recycled grease and crude Argemone oil (CAO) with methanol lowered specific gravity of CAO. Transesterification drops specific gravity of TEO (transesterified oil) to a comparable value with that of PD (petroleum diesel) and an increase in volume of production of TEO (Table 2).

The temperature at which a cloud of wax crystal first appears in a liquid when cooled under controlled conditions during a standard test is called cloud point. The novel synthesis lowered the cloud point from 8⁰c (CAO) to -1⁰c (TEO) as shown in table-2. Lower cloud point comparable to that of petroleum diesel improves the low temperature operability quotient of the TEO both in absolute and additive package when fuelled to commercial diesel engines.

The acid value, also called neutralization number or acid number is milligrams of potassium hydroxide (KOH) that is required to neutralize the acidic constituents in one gram of bio diesel sample. The present investigation by novel fuel synthesis method dropped the acid value of TEO (Table 2) which indicates low free fatty acid content, less corrosive fuel, less probability in fuel filter clogging and the presence of water in biodiesel is less. Too much amount of free fatty acid can cause functioning problems at low temperatures and fuel filter clogging. This parameter can also be used to measure the freshness of the biodiesel. Fuel that has oxidized after long-term storage will probably have a higher acid value.

The iodine value (IV) or iodine number is a quality standard for evaluating biodiesel stability to oxidation and a measurement of total instauration of fatty acids measured in grams of iodine/100 grams of biodiesel sample, when formally adding iodine to the double bonds. Biodiesel with high IV is easily oxidized in contact with air. Novel fuel synthesis dropped the IV to an acceptable range close to that of petroleum diesel (Table 2). However TEO with a slightly higher IV than that of petroleum diesel tends to polymerize and form deposits on injector nozzles, piston rings and piston ring grooves but AME10 and AME20 have IV proximal to PD and have no adulteration or engine hardware modification problem.

Viscosity of fuel is a specified standard for diesel engines within a fairly narrow range. In order to develop the high pressures needed in modern injection systems, the clearance between the plunger and barrel is approximately ten-thousandth of an inch. In spite of this small clearance, a substantial fraction of the fuel leaks past the plunger during compression. Low viscosity causes leakage and a significant power loss. High viscosity results insufficient supply of fuel to fill the pumping chamber and thereby loss in power. Acceptable higher limit of viscosity for a biodiesel is 6.0 centi-poise (cst). The novel fuel synthesis plummets the viscosity of TEO to 4.981cst. However AME10 and AME20 have viscosity which is very close to that of petroleum diesel (Table 2) tending to negligible power loss.

The flash point is the lowest temperature at which a combustible mixture can be formed above the liquid fuel. The flash point is determined by heating a sample of the fuel in a stirred container and passing a flame over the surface of the fuel. The novel fuel synthesis dropped the flash point of TEO to an acceptable range close to that of petroleum diesel (Table 2). However AME10 and AME 20 have flashpoint comparable to PD, thus no combustion problem.

The quality of the fuel to auto-ignite at the temperatures and pressures present in the cylinder when the fuel is injected is measured by a dimensionless number called cetane number. It implicates the ignition delay time a fuel experiences from injection to combustion. Generally, higher the cetane number, shorter the ignition delay time, and higher the tendency of the fuel to auto ignite. The novel fuel synthesis brought the cetane number close to that of petroleum diesel (Table 2). However AME10 and AME20 have cetane number slightly higher than that of PD tends to have no ignition delay problem.

3.2. GC-MS analysis of oil

Fatty acid(FFA) composition of crude Argemone oil (CAO), and the corresponding fatty acid methyl ester(FAME) composition of TEO after transesterification was analysed by GC-MS. Analysis inferred that both free fatty acid(FFA) and fatty acid methyl ester(FAME) were present in both the oil samples, but in different proportions. After the retention time, the concentration of FAME (Fatty acid methyl ester) is augmented and the concentration of corresponding FFA in oil is lowered. Substantially higher concentrations of FAMEs are responsible for lowering viscosity of transesterified oil as compared to CAO.

Several additional FAMES, named 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 Argemone oil. CAO 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.

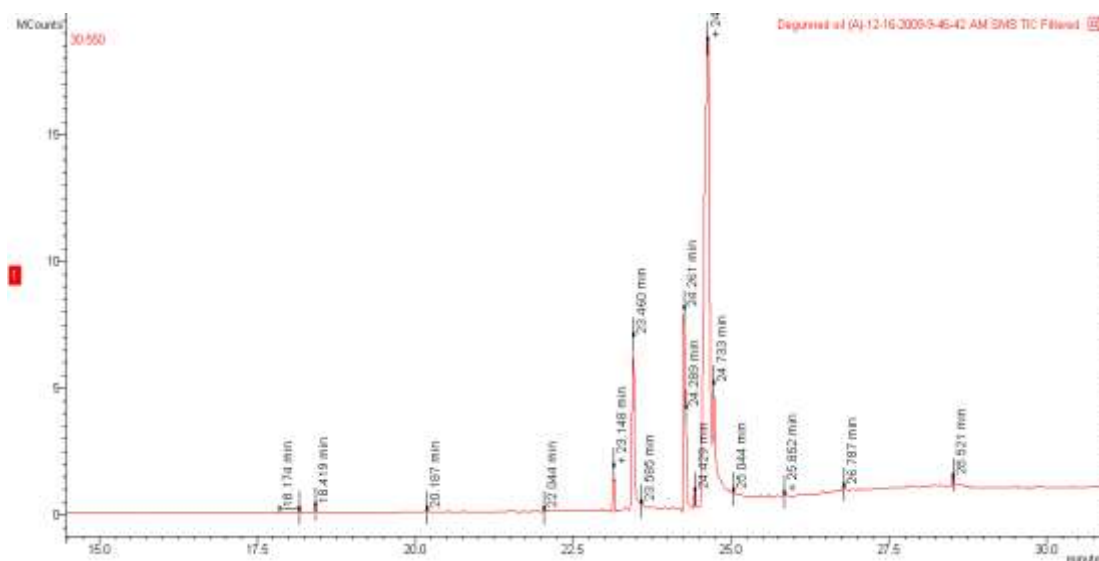


Fig. 5. Gas chromatography diagram of CAO

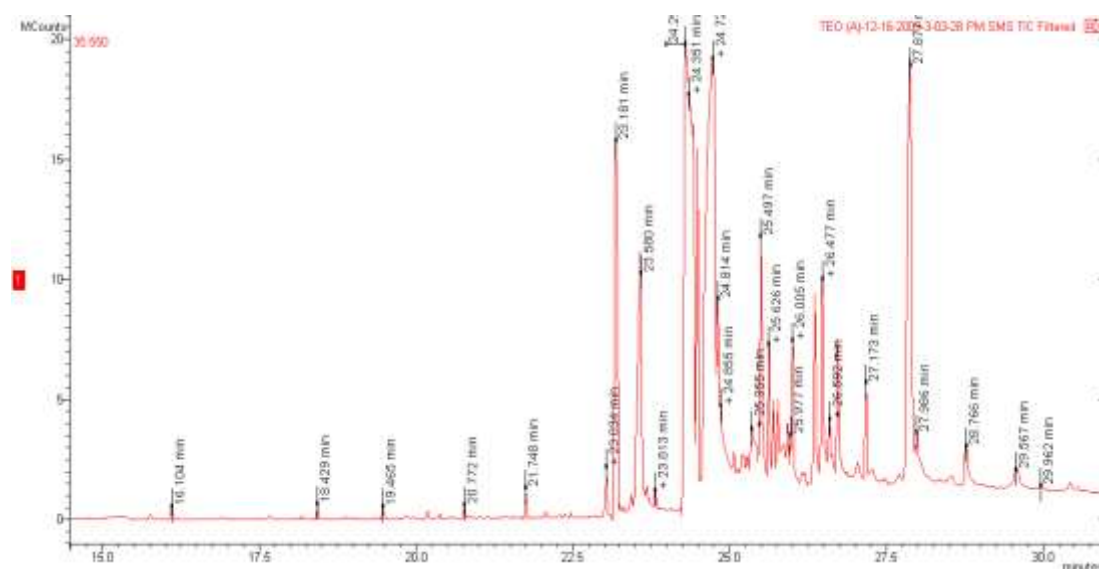


Fig. 6. Gas chromatography diagram of TEO

3.3. Engine test analysis

At different loads (BHP) the engine runtime (seconds), fuel consumption (ml/sec), and increase in run time (%) for 20 ml oil were noted and plotted as shown in Fig. 3(a), Fig. 3(b) and Fig. 4 respectively. Outcome revealed that engine runtime per unit amount of oil was lowered with increasing load on engine, which in turn increased fuel consumption of the engine. Reportedly parallel observations were also found (Meng et al., 2008). Initially at low load, runtime difference between PD and methyl ester blended PD was comparatively less, but with increasing load that disparity becomes more. It infers that both CAO and TEO were effective to increase diesel engine fuel efficiency, especially in presence of high load. Percentage increase in runtime for PD, CAO, TEO, AME10 and AME20 oils (Fig. 4) also indicated that fuel efficiency of TEO added PD (AME10 & AME20) over neat PD increased with increasing load on the engine. Data revealed that maximum 50% increment in runtime was recorded when 20% TEO was mixed with 80% PD.

3.4. Exhaust emission analysis

Hazardous emissions of carbon monoxide, carbon dioxide, unburned hydrocarbons, nitrogen oxide and free oxygen in the exhausted smoke were investigated by AVL gas analyser and the results for all the tested fuels were presented in a bar chart as shown in Fig 7(Table-4). These emissions were reduced due to TEO blending with PD. Additive package of 10% and 20% TEO lowered the smoke percent and concentration of unburned hydrocarbon (HC), CO, CO₂ and NO_x in emitted smoke as compared to neat PD. Gas chromatography infers the presence of comparatively higher concentration of several unsaturated FAMES in TEO than CAO and possibly responsible for more energy generation. Researchers (Schumacher et al., 1995; Sheetan et al 1998) investigated that additive package of biodiesel has propensity to lower the formation of combustible mixture and possibly be addressed to comparatively higher carbon monoxide and unburned hydrocarbon concentrations in emissions.

Table-4

Hazardous emissions

Emissions	PD	CAO	TEO	AME-10	AME-20
CO in%	0.30	0.31	0.06	0.49	0.65
HC in ppm	40	19	17	18	19
CO ₂ in %	3.10	2.81	3	3.20	3.30
O ₂ in %	15.20	15.41	16.52	15.90	16.18
NO _x in ppm	198	146	142	108	105

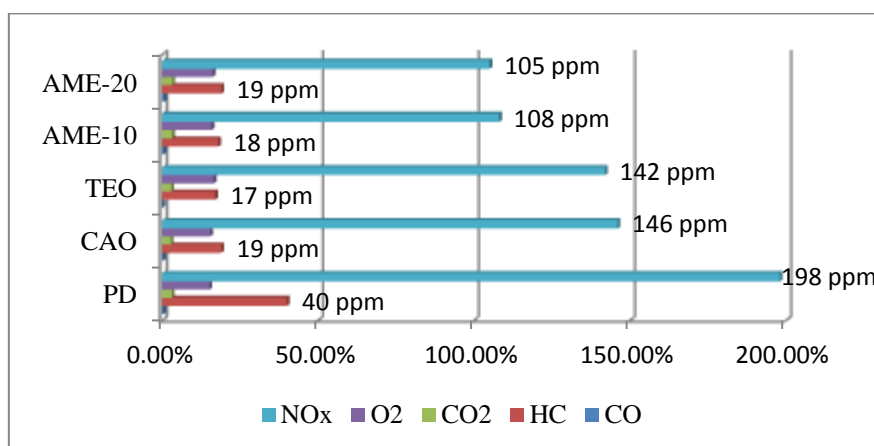


Fig 7 Emissions of hazardous gas in % and ppm.

4. Conclusion

The present investigation unveiled that mixed stock of Argemone oil with recycled grease, increased the volume of production of transesterified oil (TEO). Crude Argemone oil (CAO) was alkaloid and gum free Argemone oil. Mixed feed stock was treated with two-steps successive esterification and transesterification processes in presence of acid and alkali catalyst respectively to obtain transesterified oil (TEO). Investigation of several physical, chemical properties and engine fuel efficiency testing inferred that 10% to 20% blending of TEO with PD led to comparable results as that of neat PD, with higher fuel economy and negligible power loss. Except for very high load, the engine fuel efficiency after 20% TEO adulteration was also similar to that of 10% blended PD. The synthesis of biodiesel is expensive. The present novel synthesis of biodiesel in laboratory scale using recycled grease would promote innovative thinking to produce it in industrial scale. To summarise, the additive package of 10% to 20% TEO with PD was economically more viable than neat PD and fuelling TEO or CAO in absolute package would lead to low temperature operability problem.

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