

## Bio-ketones from lignocellulosic biomass: experimental investigation on fuel properties, combustion and emissions characteristics of cyclopentanone blend with diesel in compression ignition engine

*Use of alternative fuels in compression ignition engines is the topic for many studies. This paper presents the results of lubricity, calorific value, viscosity, surface tension and density of a ketone blend with diesel to use as a fuel in compression ignition engine. Analyses of fuel properties are vital due to their effect on fuel system. In addition, this study is related to the development of future biofuels and it indicates the effect of oxygen double bond in molecular structure of ketones on important fuel properties. Cyclopentanone which has cyclic molecular structure was used; it can be produced from lignocellulosic biomass through various processing ways. This ketone was blended with diesel fuel at 10% vol. Results from fuel properties tests were compared to the conventional diesel fuel. In the next step this blend was tested in a research diesel engine to analyse its combustion behaviour and emission characteristics of exhaust gases; these results were compared with ultra-low sulphur diesel fuel. Results showed that cyclopentanone, as an additive to diesel, improved surface tension and density of the fuel but in contrast had negative effect on viscosity, lubricity and calorific value of the fuel, but still in the standard range. Combustion behaviour of this fuel in the diesel engine also showed longer ignition delay of ketone blend and also that gaseous emission such as CO and THC are higher than from diesel fuel and NO<sub>x</sub> emission is less than from conventional diesel fuel combustion.*

Key words: lubricity, viscosity, calorific value, diesel engine, cyclopentanone, emission

### 1. Introduction

The increasing demand for oil in transportation and production of energy has envisaged a possible oil crunch and hence lent some urgency to the discovery of alternative fuels with biofuels being a promising substitute. Biofuels are hence required to overcome uncertainties with relation to energy insecurities, increase economic development in both developed and developing nations and to mitigate climate change and achieve lower greenhouse gas emissions and a lower carbon footprint. According to the World Energy Council's 2015 resources survey, 86% of all power was generated using fossil fuels (oil, coal and gas). This detrimental reliance on fossil fuels pushes the need for a diversity of energy production [1]. Bioenergy is the largest renewable energy source with 14% out of 18% renewables in the energy mix and supplying 10% of global energy supply. The World Energy Resources report in 2015 further determined that biofuels are the most viable and sustainable option in replacing oil dependency. Future demand will come from the need for renewables in transport, followed by heating and electricity sectors. Biodiesel is defined as a fuel comprised of mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats. Vegetable and animal derived feedstock used to produce biodiesel is known as triglycerides. The suitability of a vegetable oil for biodiesel ultimately depends on its fatty acid composition and many of the fuel properties. These fatty acid ester-influenced biodiesel fuel properties include cetane number (CN), calorific value, kinematic viscosity (KV) and lubricity [2].

However, it is vital that the advanced biofuels are produced from cellulosic or other non-food feedstocks [3]. Further, the fuels used in road transportation are subjected to increasingly stringent regulations [4, 5]. Biofuels can

effectively reduce emissions in modern four-stroke compression ignition engines in comparison to petroleum diesel. Biofuels have been observed to reduce carbon monoxide (CO), unburnt hydrocarbons and particulate matter (soot) however, nitrogen oxide emissions (NO<sub>x</sub>) has been observed to increase [6, 7]. Although biofuels have considerable advantages, numerous properties such as power output, calorific (heating) value and NO<sub>x</sub> emissions are questionable.

In the quest for renewable energy sources, researchers have used various microbial organisms to transform lignocellulosic biomass into biofuels. Methyl Ketones are originally found in the fragrant evergreen plant, rue (*ruta graveolens*). Methyl Ketones are also found in tomatoes and other plants and insects and are usually used to provide the scent in essential oils. However, researchers are working on methods to produce them in large quantities using engineered *E. coli* bacteria to break down glucose [8].

Researchers found various methods to produce biofuels by endophytic fungal conversion of cellulose [9]. The present study investigates the physico-chemical properties, combustion and emission characteristics of one of the cyclic ketones i.e. Cyclopentanone compared with standard engine fuel characteristics. These bio ketone fuels are prototypes for biofuels which were produced by endophytic fungi. There are only a few results from researchers relevant to this work. Ketones behaviour in engine is not well understood and needs more investigation.

### 2. Experimental methods

#### 2.1. Fuel molecules investigated

One cyclic ketone was investigated i.e. cyclopentanone (Fisher Scientific, Acros Organics, 99+%, pure, molecular

weight 84.118 g/mole) with molecular formula  $C_5H_8O$ . Additionally diesel fuel was tested to provide reference data for comparison with these oxygenated ketone fuels. The Ultra-Low Sulphur Diesel (ULSD) fuel was supplied from Shell Global Solutions UK. The Molecular structure of this ketone is shown in Figure 1. The  $C=O$  double bond represents a carbonyl functional group. The oxygen atom in ketone makes it more combustible due to its electronegative properties.

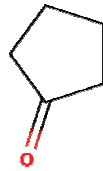


Fig. 1. Molecular Structure Cyclopentanone

The aim of the experiments was to investigate the effects on fuel properties, combustion and exhaust emissions of the following molecular features: blend of cyclic carbonyl group such as cyclopentanone with standard Ultra Low Sulphur diesel, which is a mixture of alkanes with approx. 20–25% aromatics.

## 2.2. Fuels characterization

For fuels characterization the proportion of ketone in blend with diesel blends was 10% vol. Lubricity, calorific value, viscosity, density and surface tension were measured by dedicated instruments and the effect is discussed. Other important properties were gathered from references and experimental studies summarized in Table 2.

### 2.2.1. Lubricity

Lubrication properties of Cyclopentanone were measured on a High Frequency Reciprocating Rig. This is shown schematically in Figure 2. The test specimens are 6mm diameter steel ball and steel disc. All of the lubricity tests were conducted according to the EN ISO 12156-1:2006 (max 460  $\mu m$ ) [10–13].

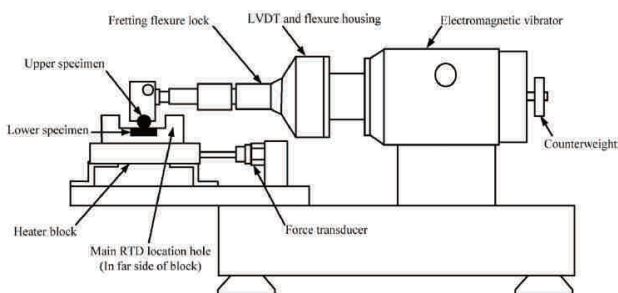


Fig. 2. Schematic diagram of the HFRR [10, 11]

The test conditions are: fuel temperature maintained at 60°C, humidity and temperature of test environment controlled in cabinet which is employed to provide the laboratory air conditions as shown in Figure 3. During the test the lower specimen (disc) is fully submerged in the fuel and the ball is pressed from above and subjected to reciprocating motion with frequency of 50 Hz lasting for 75 minutes. At the end of each test all the contacted components were cleaned by ethanol and acetone consecutively. All fuel

samples were tested two times and repeatability was demonstrated to be less than 20 $\mu m$  in the scar diameter. In addition, friction coefficient  $\mu$  and film concentration reported by the HFRR instrument computer. 100 x optical magnification microscope camera was used to measure the size of the wear scar on the ball in micrometres.

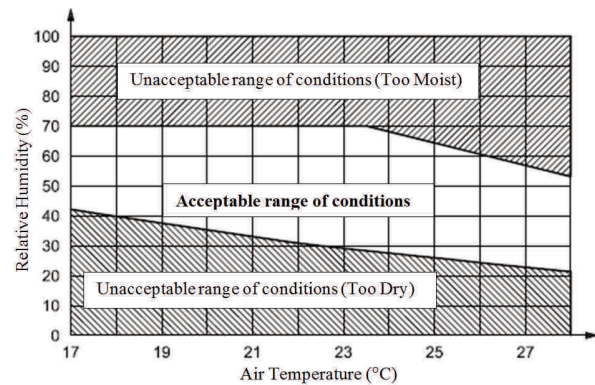


Fig. 3. Laboratory Air Conditions [10, 11]

### 2.2.2. Calorific value

Heat of combustion is determined in this test method by burning a fuel in an oxygen bomb calorimeter under controlled conditions [14]. The tests were carried out in IKA C200 calorimeter under the specific conditions and assumptions: temperature of 25°C for the fuel and its combustion products, decomposition vessel to be filled with 99.5% pure oxygen at a pressure of 30 bars to optimize the combustion process. The tests were done to adhere to the EN 14213 standard with minimum value of 35 MJ/kg. Gross (higher) calorific value (HCV) was measured and the Net (lower) calorific value (LCV) was then calculated based on the formula taking account of the hydrogen presence in fuel.

### 2.2.3. Surface tension

Surface tension values are essential in understanding fuel spray behavior and are an important fuel property which affects the stability of surface waves during the drop breakup process. A Sita Proline T15 tension meter system was used to obtain the surface tension measurements as it complies with the ASTM D971 standards. Maximum bubble pressure method was used to measure the surface tension of the sample fuels. Figure 4 shows the schematic

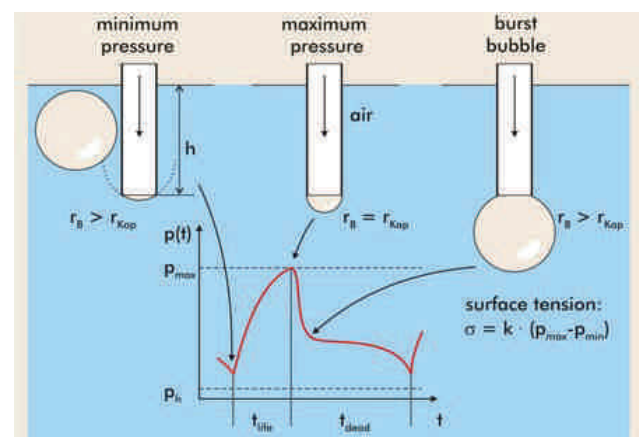


Fig. 4. Schematic diagram of tension meter structure [15]

diagram of the bubble pressure method. This method makes use of this bubble pressure which is higher than surrounding water environment, the gas which is pumped in to fluid creates some bubbles as shown in Figure 4 [15].

### 2.2.4. Viscosity

The automotive industry places a great deal of significance to the accurate measurement of dynamic viscosity at very high shear rates. The High Temperature High Shear (HTHS) viscosity at temperatures of 40°C, 60°C and shear rate  $10^7 \text{ s}^{-1}$  was measured using an Ultra Shear Viscometer. The USV can measure at high shear rates, it is connected to a DC servo motor capable of speeds of over 20000 rpm and an electromagnetic clutch which engages the rotor for only a very short period of time (100 ms) [16]. This brief shearing interval minimizes the shear heating in the lubricant. Schematic diagram of USV is shown in Figure 5. The process was fully automated with a simple to use software and a rapid turnaround. Results were analysed to adhere to the ASTM D6751-15a standard specifications where they are required it to be between 1.90 and 6.00 ( $\text{mm}^2/\text{s}$ ) [17].

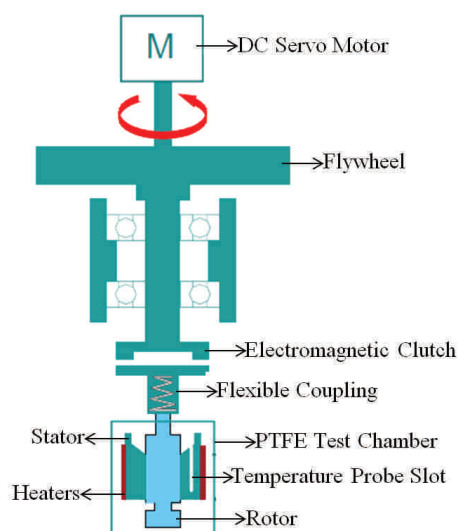


Fig. 5. Schematic diagram of the USV [16]

### 2.3. Engine and emissions

A modern single cylinder, water cooled, four stroke experimental diesel engine equipped with common rail fuel injection system was used in this paper. This engine is a single cylinder engine which was designed as one of the cylinder heads of V6 Jaguar Land Rover production engine. Full main specification of the engine is given in Table 1.

Table 1. Research engine specifications

Engine parameters	Specifications
Engine type	Diesel 1-cylinder, four stroke
Stroke type	four stroke
Bore	84 mm
Stroke	90 mm
Connecting rod length	160 mm
Compression ratio	16.1
Displacement	499 cc
Engine speed range	900–2000 rpm
IMEP range	< 7 bar
Fuel pressure range	500–1500 bar

Figure 6 indicates the schematic diagram of the experimental setup. This study in the engine was performed under a constant engine speed of 1500 rpm with an engine load of 2 bar IMEP (Indicated Mean Effective Pressure). An AVL GH13P pressure transducer mounted in the cylinder head and an AVL FlexiFEM 2P2 Charge Amplifier were used to record the in-cylinder pressure [18]. To measure the crank shaft position, a digital shaft encoder was used which produces 360 pulses per revolution. The data from the crank shaft position and pressure was combined to create an in-cylinder pressure trace. The fuel injection was split into pre- and main fuel injections with injection timing of 15 and 5 deg bTDC correspondingly, injection pressure of 500 bar and injection duration of 0.15 ms pre-injection and 0.250 ms to 0.500 ms for the main injection. A MKS MultiGas 2030 FTIR spectrometry based analyzer was employed for exhaust gaseous emissions measurement such as: Total Hydrocarbons (THC), carbon monoxide (CO), carbon dioxide ( $\text{CO}_2$ ), nitrogen oxides ( $\text{NO}_x$ ), nitrous oxide ( $\text{N}_2\text{O}$ ), and individual hydrocarbons species such as methane ( $\text{CH}_4$ ), ethane ( $\text{C}_2\text{H}_6$ ), ethylene ( $\text{C}_2\text{H}_4$ ).

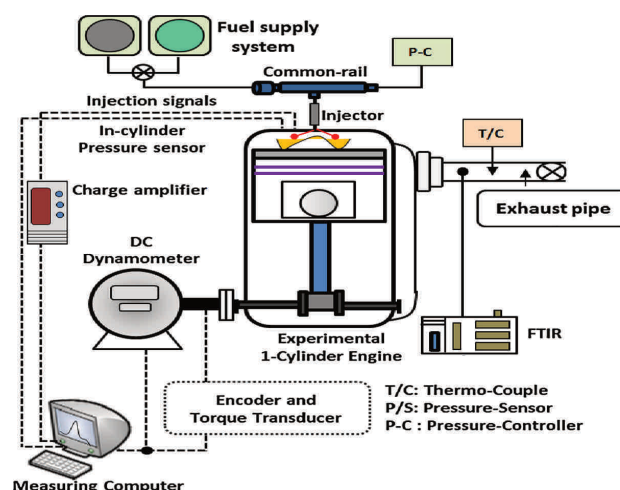


Fig. 6. Schematic diagram of test platform and sampling system

## 3. Result and discussion

### 3.1. Fuel characterization

The most critical properties of tested fuel are density, viscosity, lubricity, surface tension and calorific value. The results in Table 2 show that for a blend with approximately 10% by volume of cyclopentanone in ULSD. The density of cyclopentanone is higher than the maximum limit which is given in EN 590 ( $820\text{--}845 \text{ kg/m}^3$ ), blending of it with diesel make it more suitable for combustion.

As a result of lubricity tests, the average lubrication film concentration for ULSD was 49% and average friction coefficient is 0.203. However, the presence of oxygen in for pure cyclopentanone the average film concentration decreases to 28% with an increased average friction coefficient of 0.248. A positive effect is observed for the D90C10 blend where film concentration increases to 43% with a lower average friction of 0.197. EN590:2009 industry standards limit the wear scar for diesel fuels not to exceed  $460 \mu\text{m}$ . ULSD which is provided by Shell company shows the lubricity result of  $409 \mu\text{m}$ , pure Cyclopentanone fuel is

recorded to produce a wear scar diameter of 585 μm making it an unfavourable alternative fuel to be used in CI engines. However, the D90C10 blend produces a wear scar diameter of 419 μm which is less than Cyclopentanone. This proves that the D90C10 is a favourable replacement to petroleum diesel in CI engines which would reduce wear due to sliding and scuffing resulting in reduced failures.

EN 14213 specifications require a minimum calorific value of 35.00 MJ/kg (LCV). ULSD is recorded to have the highest calorific value amongst the fuels tested with a value of 44.90 MJ/kg (LCV). This research also observes that the presence of a carbonyl functional group results in a lower calorific value as displayed by that of Cyclopentanone with a value of 35.67 MJ/kg (LCV). This results in lower thermal energy and less deliverable power by the fuel. The D90C10 fuel blend, however, displays a higher calorific value in comparison to Cyclopentanone with a value of 39.96 MJ/kg (LCV) presenting characteristics of higher power availability thereby higher performance.

Surface tension determines the quality and effective combustion of the fuel. Thereby it requires increased atomisation for complete combustion. ULSD, pure Cyclopentanone and D90C10 fuels display surface tensions of 25.5, 29.5 and 25.7 mN/m respectively. The D90C10 blend displays an almost same surface tension as Diesel.

Kinematic viscosity measures the resistance to motion and a higher viscosity suggests delays in combustion and poor fuel spray. ASTM D6751-15a specifies a standard between 1.90 and 6.00 (mm<sup>2</sup>/s or cSt). The results recorded for the D90C10 blend denotes an average value of 2.1077 mm<sup>2</sup>/s. This suggests that the blend fits well within the specification providing sufficient lubrication without inducing deposits or limiting fuel flow. Measurement of viscosity of pure cyclopentanone was not possible because its high volatility and danger of fire in the high-shear instrument. The effect of adding 10% volume of cyclopentanone in diesel on main fuel properties are summarized in Table 2.

Table 2. Physical and chemical properties of tested fuels

Abbreviation	% volumetric makeup		
ULSD	100 Ultra Low Sulfur Diesel		
CycN	100 Cyclopentanone		
D90C10	90 ULSD + 100 CycNone		
Properties	ULSD	CycN	D90C10
Chemical formula	C <sub>14</sub> H <sub>26</sub>	C <sub>5</sub> H <sub>8</sub> O	–
Cetane number	53.9 [19]	–	–
Density at 15°C (kg/m <sup>3</sup> )	813.8	950	837.8
Kinematic viscosity at 40°C (cSt)	2.52	–	2.05
Lower calorific value (MJ/kg)	44.9	35.67	39.96
Lubricity at 60°C (μm)	409	585	419
Surface tension at 25°C (mN/m)	25.5	29.5	25.7
Carbon (wt%)	86.44 [19]	71.33	–
Hydrogen (wt%)	13.56 [19]	9.51	–
Oxygen (wt%)	0 [19]	19.16	–

### 3.2. Engine and emissions

Figure 7 shows the effect of the bio ketone blend with diesel on the in-cylinder pressure and the rate of heat release (ROHR) versus crank angle degree (CAD). The values of the peak pressure developed in the engine and the crank angle location for both ULSD and the D90C10 blend are displayed in Figure 7. It has to be noted that use of cyclopentanone as a blend affects the combustion behav-

our; this is attributed to the presence of double bond oxygen in the ketone hydrocarbons. It was observed that peak in-cylinder pressure for both fuels occurred at 368° with maximum pressures of 51.23 bar and 51.25 bar for ULSD and D90C10 respectively. Smooth readings were obtained for both ULSD and D90C10 with very little variation between the two lines. This further contributes to previous studies suggesting that there is no difference in in-cylinder pressure for diesel and moderate biodiesel percentage blends. It was observed that peak in-cylinder pressure for both fuels occurred at 368° with maximum pressures of 51.23 bar and 51.25 bar for ULSD and D90C10 respectively. Smooth readings were obtained for both ULSD and D90C10 with very little variation between the two lines. This further contributes to previous studies suggesting that there is no difference in in-cylinder pressure for diesel and moderate biodiesel percentage blends. This further determines that the Cyclopentanone-Diesel blend is a worthy substitute for petroleum diesel in CI engines as it provides sufficient pressure to aid in a similar performance to ULSD. However, it should be noted that research should be carried out for additional blends at varying loads to identify the full capabilities of Cyclopentanone-Diesel blends.

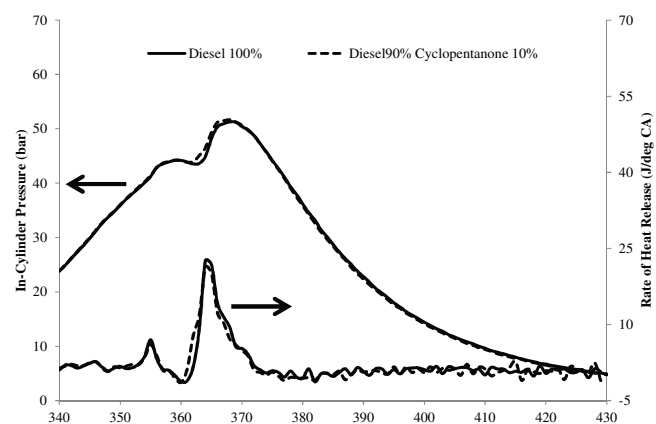


Fig. 7. Effect of tested fuels on combustion characteristics

Use of Cyclopentanone-Diesel blends as a fuel has effect on the gaseous emissions coming from the exhaust. Figure 8 show the CO and THC engine-out emissions for the investigated fuels. It can be concluded that both CO and THC for the tested cyclopentanone blend were higher compared with diesel combustion in engine. Also Figure 9 indicates that ethylene, ethane and methane emissions are higher than those from diesel fuel.

In contrast to all of these results, Figure 10 shows that NO<sub>x</sub> concentrations from the tested fuel were lower than from diesel fuel. The carbonyl function group in cyclopentanone is thought to result in lower NO<sub>x</sub> emissions which were tested at low loads. Slight increase of NO<sub>2</sub> can be due to the presence of oxygen content in cyclopentanone, so oxygen content and lower cetane number of the cyclopentanone affect the decrease of total NO<sub>x</sub> species in the exhaust emissions [18].

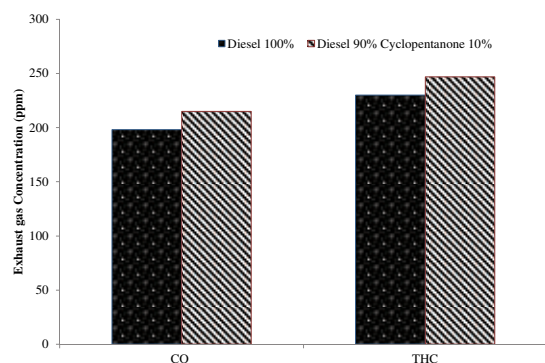


Fig. 8. Exhaust CO and THC species measured for each tested fuels

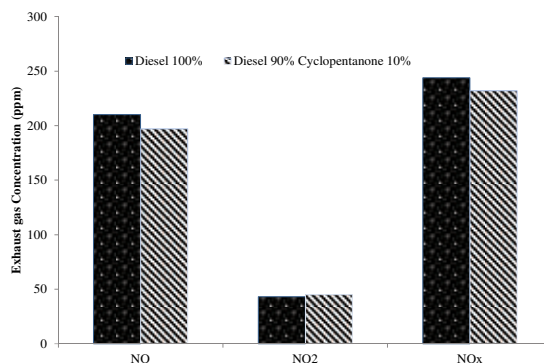


Fig. 10. Engine exhaust NO<sub>x</sub> species concentration for each tested fuels

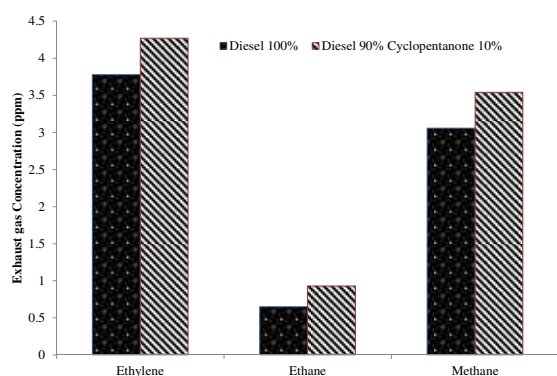


Fig. 9. Engine exhaust hydrocarbon species measured for each tested fuels

Uses of appropriate Diesel Oxidation Catalysts, Selective Catalytic Reduction and NO<sub>x</sub> absorber catalyst were suggested for further investigation in different engine experiment conditions.

#### 4. Conclusion

Use of alternative fuels such as biofuels in the last years becomes more interesting due to the scarcity of fossil fuels and their environmentally negative impact on human

health. Bio-ketones which are produced from lignocellulose biomass are one of the new alternative fuels to use in the current engine without any modifications. Results show that ketone blends have proper fuel properties to use in the diesel engine without use of any additives or lubricant. This blend had slightly longer ignition delay than diesel because of carbonyl groups which make H-abstraction more difficult from ketone compared to diesel (Alkanes) [20]. NO<sub>x</sub> emissions from combustion of this fuel is slightly lower than diesel fuel which make this fuel more interesting for further investigation. Increase of other gaseous emissions requires some more studies based on use of after treatment strategies in various engine conditions. From the literature, due to the presence of oxygen content in carbonyl groups a reduction in particulate mass can be expected; this will be studied in the future by using Scanning Mobility Particle Sizer Spectrometer.

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#### Nomenclature

CI	compression ignition	IMEP	indicated mean effective pressure
ULSD	ultra low sulphur diesel	CAD	crank angle degree
CycN	cyclopentanone	bTDC	before top dead centre
CN	cetane number	ROHR	rate of heat release
KV	kinematic viscosity	CO	carbon monoxide
LCV	lower calorific value	CO <sub>2</sub>	dioxide
HCV	higher calorific value	NO <sub>x</sub>	nitrogen oxides
HFRR	high frequency reciprocating rig	N <sub>2</sub> O	nitrous oxide
HTHS	high temperature high shear	THC	total hydrocarbon
USV	shear viscometer	CH <sub>4</sub>	methane
WSD	wear scar diameter	C <sub>2</sub> H <sub>6</sub>	ethane
FTIR	Fourier Transform Infrared Spectroscopy	C <sub>2</sub> H <sub>4</sub>	ethylene

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