Measurement of the ignition delay period of cotton methyl ester and cotton ethyl ester biodiesel fuels

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Abstract

This paper describes an experimental effort for the production of cotton methyl ester (CME) and cotton ethyl ester (CEE) from neat cottonseed oil (CSO) for use as a biodiesel fuel and investigation the ignition delay times of these fuels using the shock tube. The transesterification of the neat CSO with methanol or ethanol have been performed using different variables to determine the optimum conditions for the preparation. The experiments were simulated at reaction temperature, 60 ^{0}C. The optimum parameters were cottonseed oil / alcohol molar ratio, 1:6; NaOH amount, 1% by the weight of the oil and reaction time, 75 min. The 89% of the neat CSO was converted to CME or CEE and the use of different alcohols (methanol or ethanol) present few differences with regards to the kinetics of reaction but the final yield of esters remains almost unchanged. The physical properties of all the tested fuels are measured. The ignition delay times were measured using a piezo-electric pressure transducer; charge amplifier, data acquisition card, IBM computer and LabVIEW program. For the all fuels, the minimum ignition delay times occur near the stoichiometric condition and the CME and CEE are suitable for the diesel engine without any modifications.

Keywords: Biodiesel**,** Transesterfication**,** Ignition delay times**,** Shock tube

1. Introduction

The idea of using vegetable oil as a diesel engine fuel has been around for a long time and dates back to the beginning of last century when the diesel engine was invented by Dr Rudolf Diesel. The current effort is directed at improving air quality as well as increasing domestic energy security.

Alternative fuels and the vehicles powered by them are viewed as ways to reduce harmful air pollutants and greenhouse gases. In this context, vegetable oil is an alternative fuel for diesel engines that is receiving great attention world wide. Although it attracts the most attention because it is renewable, it can be used either pure or in blends with diesel fuel in unmodified diesel engines, and it reduces some exhaust pollutants. It is also attractive because it can be produced easily from common feedstocks. Nevertheless, various technical and economic aspects require further improvement of these fuels. Numerous different vegetable oils have been tested as biodiesel. Biodiesel can be derived from food grade vegetable oils, nonfood grade vegetable oils, animal fats, and waste restaurant greases. Often the vegetable oils investigated for their suitability as biodiesel are those which occur abundantly in the country of testing. Therefore, soybean oil is of primary interest as biodiesel source in the United States while many European countries are concerned with rapeseed oil, and countries with tropical climate prefer to utilize coconut oil, hazelnut or palm oil [1-3]. Other vegetable oils, including sunflower, rubber, etc., have also been investigated. Furthermore, other sources of biodiesel studied include animal fats, salmon oil and or waste cooking oils [4-7].

Raw or refined vegetable oil, or recycled greases have significantly different and widely varying properties that are not acceptable for use in modern diesel engines. The higher viscosity and chemical composition of unprocessed oils and fats have been shown to cause problems in a number of areas [8-10]: (i) piston ring sticking; (ii) injector and combustion chamber deposits; (iii) fuel system deposits; (iv) reduced power; (v) reduced fuel economy and (vi) increased exhaust emissions. Use of unprocessed oils or fats as neat fuels or blending stock will lead to excessive fuel condensation and corresponding dilution of the engine’s lubricating oil that may result in sludge formation. Any or all of these conditions may result in reduced engine life, increased maintenance costs, or catastrophic engine failure. More over, the problems associated with the use of raw vegetable oil or animal fat may not become evident until a significant amount of damage has occurred over an extended period. The significantly higher viscosity of raw vegetable oils (27-54 mm2/s) compared to petroleum diesel fuel (2.6 mm2/s) [11] alters fuel injector spray patterns and spray duration, adds stress on fuel injection systems, and results in incomplete combustion and high dilution of the engine lubricating oil. Transesterfication is a way to lower the viscosity of the vegetable oil by breaking up the triglyceride molecule and separating the fatty acid molecules from the glycerin molecule. This makes the properties of the vegetable oils and animal fats closer to diesel fuel, solving the problems due to the high viscosity of vegetable oils. The methyl esters of vegetable oils (biodiesel), which are produced by combining methanol with the vegetable oil, are of particular interest; these fuels tend to bum cleaner, perform comparably to conventional diesel fuel.

Many studies have been reported by several researchers at the Helwan University involving different types of fuel as a primary source of energy for diesel engines [12-17]. Numerous experimental methods have been developed in order to measure the combustion characteristics fundamental as the laminar burning velocity and ignition delay of a combustible mixture for coal derived fuels or jojoba methyl ester by using constant volume bomb or shock tube method. Also, many studies have been done for these fuels on diesel engine for determining the performance and exhaust emissions. In this work we are interested in determining the ignition delay of cotton seed oil as a vegetable oil fuels for diesel engines in the shock tube. Although numerous studies have been conducted on cotton oil production as biodiesel and exhaust emission testing for many years [18-23], the ignition delay of neat CSO, CME or CEE have not been searched in any diesel engine or another test rig yet. However, ignition delay is an important variable that influences the combustion chamber design, rate of pressure rise, peak cylinder pressure, exhaust gas temperature, and exhaust emissions. This was the basic motivation behind the research in this paper.

The main objective of the present work was the preparation and shock tube investigation of comparative ignition delay times of neat CSO, CME and CEE so an extensive test program is set up in the following steps:

1. Optimum conditions for the transesterification of neat CSO with methanol or ethanol have been determined for production CME or CEE.
2. Analysis of the physical properties for neat CSO, CME, CEE and diesel fuel were carried out.
3. The ignition delay times have been measured for neat CSO, CME and CEE over a wide range of equivalence ratios at initial charge temperature and pressure.

2. Materials and methods

2.1 Cotton plant

The plant is cultivated mainly for fibers. Cotton is probably known since 3000 B.C., cotton came from India to Egypt and was brought in Europe by Alexander the Great (330 B.C.) but its worldwide use was born only in the 19th century. The kernel comprises about 50% of the seed and contains about 30% of oil. From a technological point of view, the fuel property of cottonseed oil seems to meet the fundamental requirements of diesel engine [18,19]. Therefore, use of cottonseed oil blended with diesel oil as a substitute for conventional diesel oil in diesel engine is reasonable and prospective. Much of the published research carried out cotton seed oil up to the present time have been showed that it can be used pure (B100), methyl esters or blends with diesel fuel, B30 (30% biodiesel and 70% conventional diesel) in unmodified diesel engines [22]. And also can be used as a fuel source in a multi-fueled burner. Holt et al [21] showed that cottonseed oils performed well in the multi-fueled burner and displayed a promising potential as an alternative fuel source for cottonseed oil mills and cotton gins in their drying operations.

Engler et al. [24] found that engine performance tests using raw sunflower and cottonseed vegetable oils as alternative fuels gave poor results. Engine performance tests for processed vegetable ils produced results slightly better than similar tests for diesel fuel. However, carbon deposits and lubricating oil contamination problems were noted, indicating that these oils are acceptable only for short-term use as a fuel source. International Harvester Company [25] reported that cottonseed oil, diesel fuel blends behaved like petroleum-based fuels in short-term performance and emissions tests.

The experimental fuels performed reasonably well when standards of judgment were power, fuel consumption, emissions, etc. However engine durability was an issue during extended use of these fuel blends because of carbon deposits and fueling system problems. The effect of supercharging is studied on the performance of a direct injection Diesel engine with the use of untreated cotton seed oil under varying injection pressures [26]. The investigation revealed that cotton seed oil, in general vegetable oils, can best be utilized if supercharging is employed at the recommended IP of the engine. The performance of the engine is evaluated in terms of brake specific fuel consumption, exhaust gas temperature and smoke density. It is observed that when cotton seed oil is used as a fuel, there is a reduction in BSFC of about 15% when the engine is run at the recommended IP and supercharging pressure of 0.4 bar.

He et al [22] have been studied the cottonseed oil blend with diesel fuel in single-cylinder diesel engine. The experimental results obtained showed that a mixing ratio of 30% cottonseed oil and 70% diesel oil was practically optimal in ensuring relatively high thermal efficiency of engine, as well as homogeneity and stability of the oil mixture. Also, Computer-aided optimization of the working parameters for 30:70 cottonseed oil/diesel oil mixed fuel was achieved. It was concluded that the predominant factor affecting the specific fuel consumption was fuel-delivery angle θ, the approximate optimal value of which, in this specific case, was 3–58^0 in advance of that for engine fuelled by pure diesel oil. A predictive analytical study of the effect of viscosity, density and surface tension at 80 ^{0}C on atomization characteristics for biodiesels, and biodiesel blends in a diesel engine was performed by [10]. The atomization analysis showed that B100 coconut biodiesel had similar atomization characteristics to D2, because of its similar properties, i.e. density, surface tension and viscosity. No significant difference in drop size was observed for all B5 blends, and B20 blends and B100 biodiesels of palm, soybean, cottonseed, peanut and canola. It implies these stocks of biodiesels and their blends can be used in a DI engine with similar atomization characteristics.

An experimental investigation is conducted to evaluate and compare the performance and exhaust emission levels of sunflower and cottonseed oil methyl esters of Greek origin as supplements in the diesel fuel at blend ratios of 10/90 and 20/80, in six-cylinder, turbocharged and direct injection diesel engine [9]. It is found that all the tested bio-diesel blends can be used safely and advantageously in the present bus diesel engine, at least in these small blending ratios. The cottonseed bio-diesel blends tended to produce lower emitted soot and CO values than the corresponding ones for the sunflower case. The engine performance with the bio-diesel blends of sunflower or cottonseed oil bio-diesels was similar to that of the neat diesel fuel. Recep et al [20] have been studied the effects of vegetable oil fuels and their methyl esters such as raw sunflower oil, raw cottonseed oil, raw soybean oil and their methyl esters, refined corn oil, distilled opium poppy oil and refined rapeseed oil on a direct injected, four stroke, single cylinder diesel engine performance and exhaust emissions The results showed that both vegetable oils and their esters are promising alternatives as fuel for diesel engines. Because of their high viscosity, drying with time and thickening in cold conditions, vegetable oil fuels still have problems, such as flow, atomization and heavy particulate emissions.

2.2 Biodiesel production

Transesterification is the process of using an alcohol (e.g., methanol or ethanol) in the presence of a catalyst, such as sodium hydroxide or potassium hydroxide, to chemically break the molecule of the raw renewable oil into methyl or ethyl esters of the renewable oil with glycerol as a by-product. A simplified form of the transesterification reaction is shown below.

Oil (Triglycerides) + Alcohol + Catalyst → Alkyl esters + Glycerine

There are several factors that affect the quality of conversion by the transesterification process. The first factors to be concerned with are the free fatty acid and moisture content. Since the presence of free fatty acids and water causes soap formation, consumes catalyst and reduces the conversion of oil to ester [11]. Important reaction parameters such as molar ratio of alcohol to oil, type of alcohol, amount of catalyst and reaction time were investigated to determine the best strategy for converting the free fatty acids to usable esters [11].

The molar ratio of alcohol to triglyceride is one of the most important variables affecting the yield of ester. The stoichiometric reaction requires one mol of a triglyceride and three mol of the alcohol. However, the transesterification reaction is an equilibrium reaction so the ratio will be higher than the stoichiometric ratio to drive the reaction towards the desired products. It was reported that the largest ester yield is obtained for 6/1 methanol/triglyceride molar ratio which is the twice of stoichiometric ratio [11,27,28]. Ethanol rather than methanol can be use to produce ethyl ester of vegetable oils. Production of ethanol from biomass is one way to reduce both the consumption of crude oil and environmental pollution [11,29]. Therefore In this study, the molar ratio of methanol to glycerides was used as 6/1 and then the molar ratio of ethanol to glycerides was used as 6/1 to obtain the best fuel properties of biodiesel.

The transesterifications were enhanced by the use of potassium hydroxide, sodium hydroxide, sodium methoxide, or sodium ethoxide as catalysts. NaOH was preferred as a catalyst due to its high activity [11]. Care should be exercised when adding sodium hydroxide to compensate for a high acid value. The addition of excess sodium hydroxide will cause soap to form which will increase the viscosity of the formation of gels and interfere with the reaction and the separation of glycerol [11]. It has been observed that high catalyst concentrations greater than 1.5% lead to the production of large amounts of soaps [11,28]. In this work, three sets of the reaction mixtures were prepared with 0.5%, 1.0% and 1.5% by the weight of the oil of NaOH catalyst and allowed react.

Other variables that affect the process yield are reaction time, temperature, and mixing intensity. It has been observed by [30] that the longer the reaction time the better the conversion percentage and the rate of transesterification rises with the increase of reaction temperature. However, the maximum operating temperature cannot exceed the boiling point of the alcohol used in the process. So, in this work the oil and alcohol were heated at 60 ^{0}C. Mixing is very important to get the reaction started, but once the two phases are mixed the reaction will continue on its own.

In this study, transesterification process was carried out using different variables are summarized below:

Cottonseed oil / alcohol molar ratio: 1:6.

Type of alcohol: methanol or ethanol.

The amount of catalyst: 0.5%-1.5% by the weight of the oil of sodium hydroxide.

Temperature of reaction was constant at 60 ^{0}C.

Time of reaction mixture: 30-120 min.

The transesterification experiments were performed in a beaker of 2000 ml volume. The beaker is located in a thermostatic bath to keep the temperature constant throughout the reaction and the beaker was stirred by eclectic motor. The process can be summarized as follows:

1. The cottonseed oil was filtered to remove solid precipitate in the oil and then was subjected to drying by heating at 100 ^{0}C for 15 min to obtain very minimal amounts of water in the oil. As any water in the system will consume some of the catalyst and slow the transesterification reaction [11]. Then wait until the oil returns to room temperature.
2. The solid catalyst (NaOH) was added to 200 milliliters of alcohol (methanol or ethanol) and was dissolved by vigorous stirring. Then the prepared mixture was poured into 800 milliliters of the cottonseed oil.
3. The beaker was stirred at various times holding the temperature at 60 ^{0}C by a thermostatic bath.
4. The reaction mixtures allowed react for 30, 45, 60, 75, 90 and 120 min respectively. Then, the heater was turned off.
5. The reaction was arrested by adding ice crystal of water [13]. Products was allowed to settle and separate into phases as the bottom layer was water then the middle layer of glycerol while the upper layer was the ester, as they have different densities.

6. The top ester layer was poured into another beaker and was washed from the remaining ester and residual catalyst with 200 ml of water three times.

7. After 10 min, the water phase containing the remaining alcohol and catalyst would settle, leaving a clear ester phase on top and then the ester was dried with silica jell [13].

8. The final product was run through a media to filter from silica jell particles. Once this stage was complete, the biodiesel production was ready for quality testing.

3. Results and discussion

After establishing the experiments were simulated at reaction temperature, 60 ^{0}C. The optimum parameters of base-catalyzed transesterification reaction of CME or CEE were cottonseed oil / alcohol molar ratio, 1:6; NaOH amount, 1% by the weight of the oil and reaction time, 75 min. It is found that 89% of the cottonseed oil was converted to biodiesel using the transesterification process described above. The effect of reaction time on final yield of CME and CEE is illustrated in Fig. 1. It should be noted that both of the CME and CEE yields increase with the reaction time up to 75 min. Also, it is noticed that the use of different alcohols (methanol or ethanol) present few differences with regards to the kinetics of reaction but the final yield of esters remains almost unchanged.

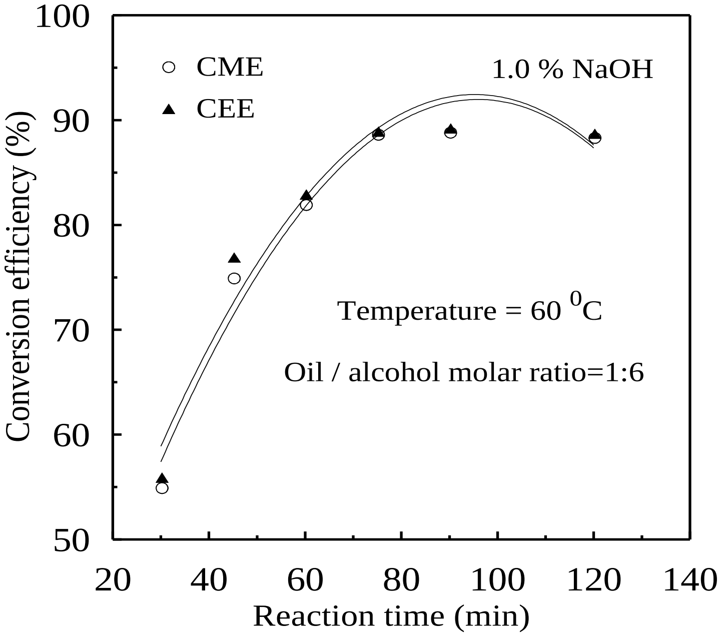

Fig. 1 Effect of reaction time on conversion efficiency of CME and CEE

3.1 Characteristics of CME, CEE and diesel fuel

The neat CSO, CME and CEE examined in this study with the properties of diesel fuel used as a reference are listed in Table 1. It should be noted that the major difference of physical properties between neat CSO and diesel fuel are density, viscosity, and cetane number as shown in Fig. 2. The density of neat CSO and diesel fuel was found to be 919 kg/m^3 and 832 kg/m^3 respectively and the kinematic viscosity of the neat CSO was 35.2 cst and for diesel fuel was 3.25 cst. It is know that a high viscosity fuel will produce a larger drop of fuel in an engine combustion chamber which may not burn as clean as a fuel that produces a smaller drop. Unburned oxidized fuel will build up in the engine around valves, injector tips and on piston sidewalls and rings [8-10]. Also, cetane number varies considerably among the neat CSO was 38 and diesel fuel was 52. So it was expected that, the ignition quality, engine performance, engine combustion roughness and exhaust emissions are different between neat CSO and diesel fuel.

Table 1 shows the properties of the CME fuel versus diesel fuel

Properties	Cottonseed oil	CME	CEE	Diesel fuel
Density @ 15 ^{0}C (kg/ m^3)	919	881	877	832
Kinematic viscosity @ 40 ^{0}C (cst)	35.2	4.63	5.2	3.25
Flash point, ^{0}C	254	170	175	80
Calorific value (kj/kg)	38443	40630	40610	44162
Cetane number	38	49	46	52
Molecular weight	331	227	224	181
Carbon residue, %	1.09	0.01	0.01	0.13
Sulfur, %	Nil	nil	nil	0.03
Ash content, %	0.1	0.01	0.01	0.57

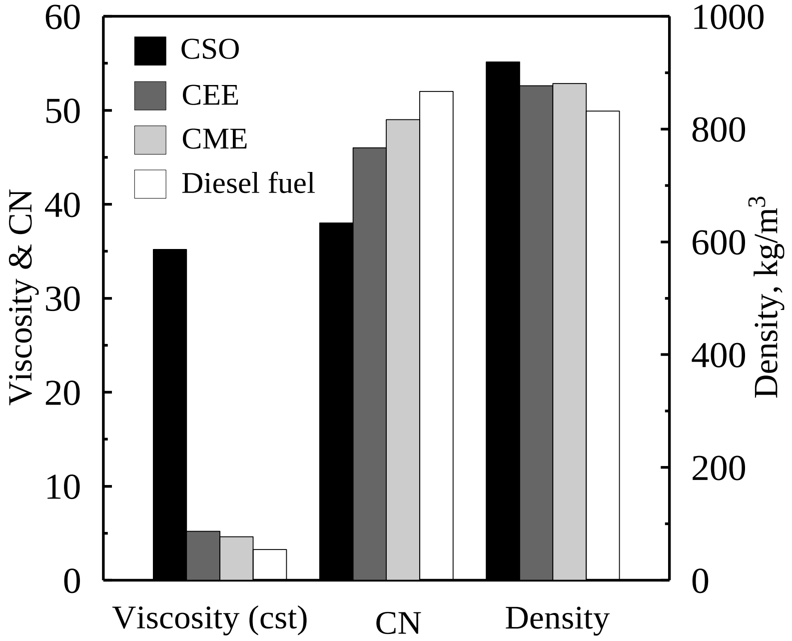

Fig. 2 The change in physical properties of CSO, CEE, CME with respect to diesel fuel

The properties of biodiesel (CME, CEE) produced are very important and should be taken into consideration before testing it in the shock tube. The properties of CME, CEE and Diesel fuel are given in Table 1. It can be seen that the properties of CME, CEE are in the acceptable ranges of the ASTM standards [31] and have a viscosity much closer to diesel fuel than neat CSO as seen from Fig. 2. Also, use of the different alcohols (methanol or ethanol) presented few differences in physical properties of CME and CEE with regards to the kinetics of reaction but the final yield of esters remains unchanged as seen in Fig. 1. The viscosities of the ethyl esters are slightly higher but the cetane number is slightly lower than those of the methyl esters.

3.2 Measuring of the ignition delay times for the biodiesel tested fuels

After completing the physical property measurements for biodiesel and its neat, an experimental study in the shock tube was developed to investigate the ignition delay times. Shock tube consists of two sections; one called the driver section and the other called the driven or test section as shown in Fig. 3. The driver section was equipped at the right hand side with a sparking plug fed by the electrical energy from battery and coil ignition system. That section can also be connected to the vacuum pump through a special valve in the gas supply and regulating device. At the left hand side of the driver section, 0.3 mm thick plastic diaphragm was fitted. The driven section was furnished by a vacuum gauge for vacuum measurement. The test section is equipped with piezo electric pressure transducer to measure the pressure-time trace and quartz window to observe the combustion process. A brass venturi was installed at the end of the driven section in the passage of the charging air. At the throat of such venturi where the pressure is a minimum, a hypodermic needle was fitted and a syringe was used to inject the tested liquid fuel in the passage of the incoming air. The temperature inside the driven section was measured using type K thermocouple. More detailed about the shock tube used and experimental procedures are given elsewhere [32,33]. The ignition delay time was recorded by measuring pressure trace with a piezo-electric pressure transducer as shown in the Fig. 4. This signal was fed to Dual Mode Charge Amplifier, the output voltage signal was sent to the National Instruments NI-DAQmx data acquisition card. Data acquisition was managed on a computer using a LabVIEW program.

In order to ensure the consistency of the experimental observations and to establish a basis for comparison of the results, preliminary tests were carried out with the present the diesel fuel. Five runs of tests were performed under identical conditions to check for the repeatability of all results. The measure delay period for all experiments agreed to within the experimental error [34].

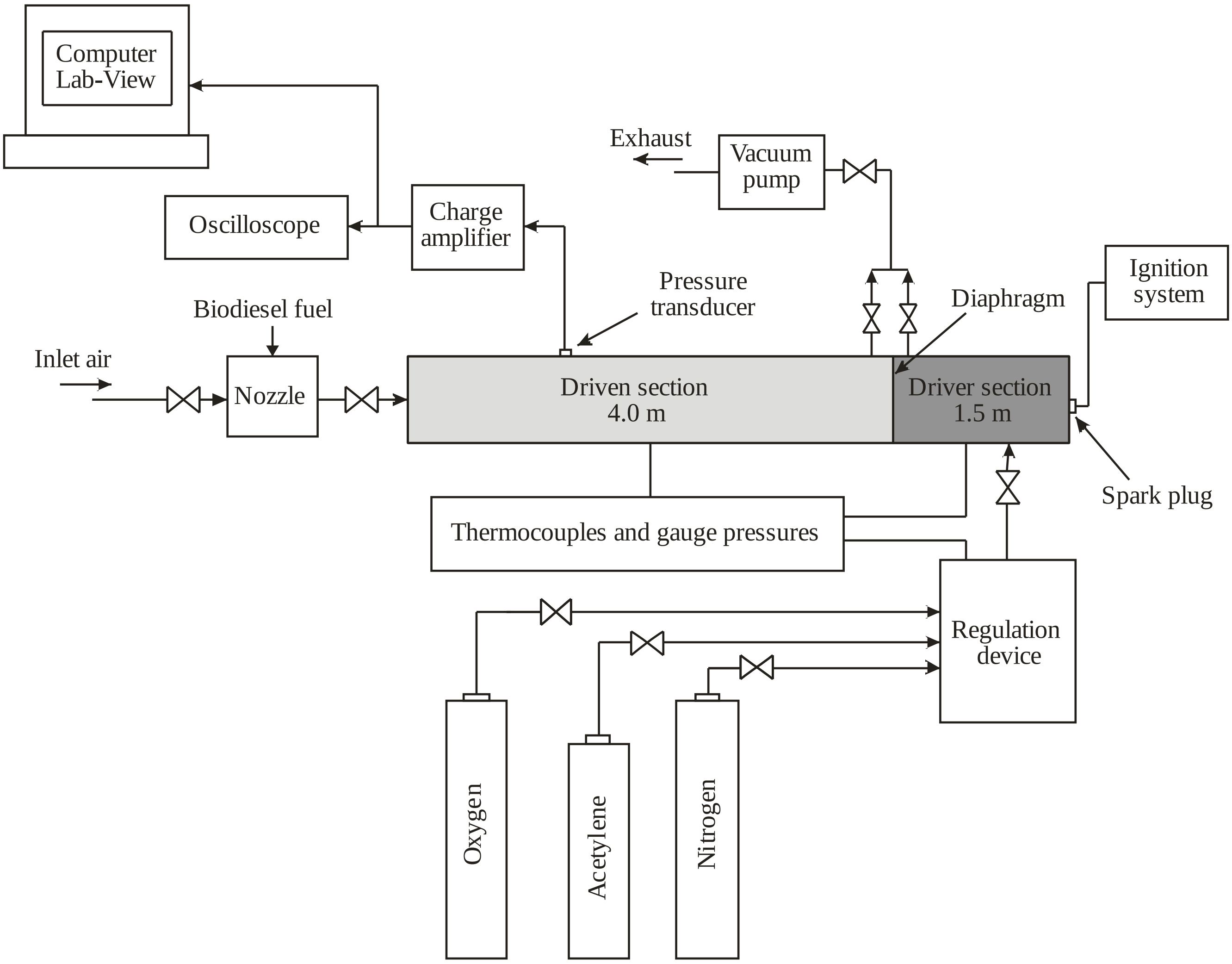

Fig. 3 Schematic of the shock tube testing

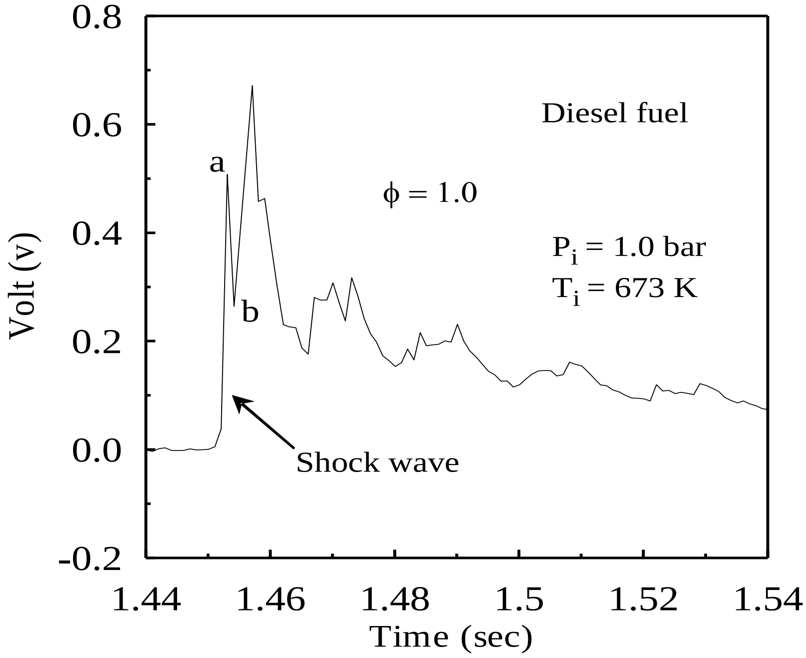

Fig. 4 Pressure history recorded with indication of ignition delay
(time interval between a-b)

Also, in order to validate the present measurement system, the measured ignition delays of diesel fuel-air mixture for different equivalence ratio were measured and the results were compared with previous data in the literature [32,33,35,36] as shown in the Fig. 5. The present data with cetane number of 52 agree within 14% with the previous data with cetane number of 49 on the same test rig [32].

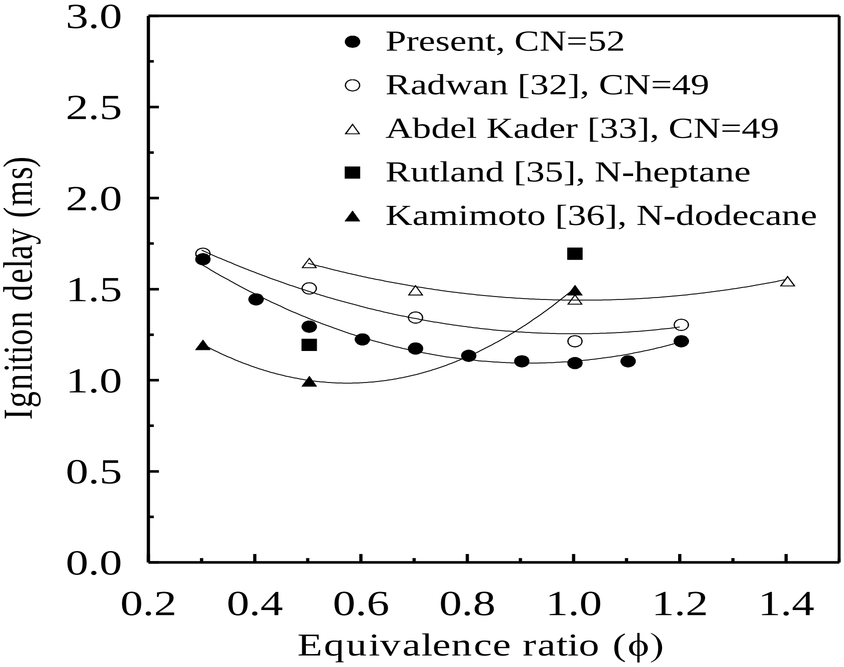

Fig. 5 Comparison of ignition delays for diesel fuel at various equivalence ratios

The fuels tested in the experiments were 100% neat CSO, 100% CME and 100% CEE. Experimentally, the ignition delay time trends observed between the different fuel types and diesel fuel are compared at different conditions of equivalence ratio (0.3 - 1.4), initial pressure of 1.0 bar and the initial temperature of 723 K. It will be seen as shown in the Fig. 6 that the equivalence ratio variation with the all fuels exhibits the familiar U-shaped behavior and this agrees with measurements by [37]. Minimum ignition delay times occur near the stoichiometric condition, while the ignition delay times rapidly increases to the fuel lean side and modestly increases on the rich side. The energy release per unit mass is greatest for all fuel-air mixtures at the stoichiometric condition at ϕ =1.05.

This has the primary effect of causing the greatest temperatures and therefore greatest overall kinetic rates at the stoichiometric condition. Therefore, it is expected that the ignition delay times would have a minimum at an equivalence ratio of 1.05 and increasing sharply in the lean side ($0.3 < \phi < 1.0$), and monotonously in the rich side ($1.0 < \phi < 1.4$).

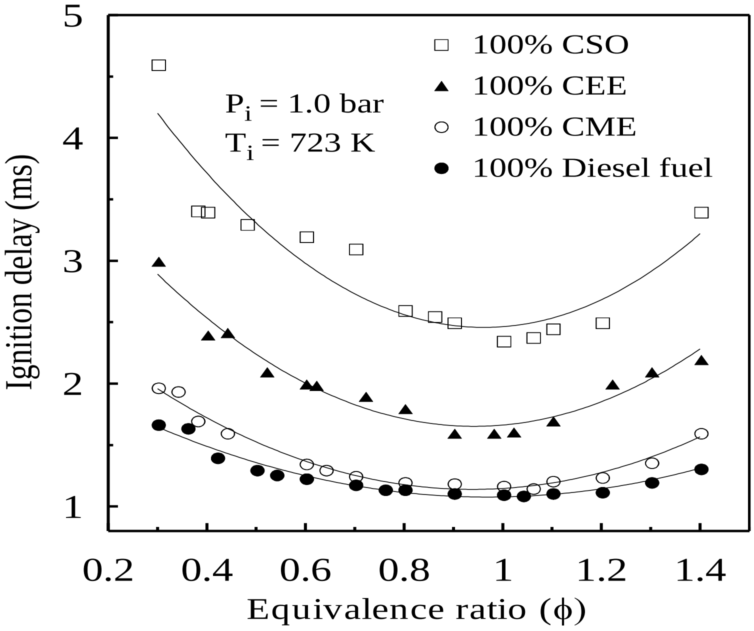

Fig. 6 Effect of equivalence ratio on ignition delay times of CSO, CME, CEE and diesel fuel

For neat CSO expected to show fairly long ignition delays since the ignition delay increased from 1.1 msec for the diesel fuel at $\phi = 1.0$ until 2.38 msec for the neat CSO and this is equivalent to a reduction of the cetane number from CN= 52 to CN = 38 as shown in table 1.However, there is very little variation in the ignition delay times of CME with CN=49 and diesel fuel with CN=52. Improvements (reduced ignition delay) are observed and that may be due to the physical properties of CME obtained after transesterification much closer to diesel fuel than neat CSO. Since the test results indicate that CSO exhibit longer delay periods than those observed with diesel fuel. The CME and CEE as alternative diesel engine fuels can be used successfully without modifications to the engine or the injector system

4. Conclusions

This paper describes an experimental effort for the production of CME, CEE and CEE-diesel blends from neat CSO for use as a biodiesel fuel and investigation the ignition delay times of these fuels using the shock tube. Effects of equivalence ratios on the ignition delay times were investigated. The results obtained from this work are summarized as follows.

1. The transesterification of the neat CSO with methanol or ethanol have been performed and the optimum parameters were cottonseed oil / alcohol molar ratio, 1:6; NaOH amount, 1% by the weight of the oil and reaction time, 75 min.
2. The 89% of the neat CSO was converted to CME or CEE and the use of different alcohols (methanol or ethanol) present few differences with regards to the kinetics of reaction but the final yield of esters remains almost unchanged.
3. The physical properties of all the tested fuels are measured as the viscosities of the ethyl esters are slightly higher but the cetane number is slightly lower than those of the methyl esters.
4. The CME and CEE are suitable for the diesel engine without any modifications.
5. The equivalence ratio variation with the ignition delays for the all fuels exhibits the familiar U-shaped behavior and the minimum ignition delay times occur near the stoichiometric condition.

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