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# Greener diesel fuels developed from water macro emulsions: stability and spray property

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## ABSTRACT

The spray and combustion characteristics of water emulsified diesel fuel with different blending ratio were experimentally investigated in a constant volume combustion chamber with different injection pressures and under various ambient temperatures. The bubbles' size of the water phase has been measured using microscope and stability tests have been conducted for all the prepared emulsions. All emulsified fuels tested were stable within a range of two weeks. The fuels were later injected and combusted in a constant volume chamber with optical access. Compared with some of the previous studies, both the ambient temperature and injection pressure has been widened to a larger range to see if the trends are still hold for the emulsified fuels in light of the spray penetration and cone angle. It is shown that both W10 (10% water by volume) and W20 were featured with longer liquid penetration, especially under low ambient temperatures, which can be attributed by the low volatility of the water. Notable increased cone angles were observed for emulsified fuel at the beginning stage of injection indicating the occurrence of micro-explosion.
**Key words:** water emulsified fuel, micro-explosion, liquid penetration, spray cone angle

## 1. Introduction

A number of emission reduction strategies have been emerged and developed for the diesel engines as the emission regulations are getting stringent. One of the promising technologies is to add water component into the fuel. The fuel is so called either emulsified fuel or micro emulsified fuel, provides the opportunity to solve the classic diesel engine dilemma known as the "Particulate Matter (PM) -NOx trade-off" since a reduction of both exhaust emissions has been found by using water-in-diesel fuels in direct injected compression ignition engines [1-4].

The difference between emulsified fuel and micro emulsified fuel is mainly on the drop size as the drops diameter in the emulsion are in the range of 1-10 μm, while in a microemulsion are much smaller, e.g. 5-20 nm [5]. The two fuels are also different in the thermodynamic stability.[5,6] Microemulsions, usually transparent, are thermodynamically stable, while emulsions, usually present an opaque and milky appearance, are thermodynamically unstable and will eventually separate into two phases, although the separation can be delayed by carefully choosing the surfactants and polymers. The emulsified fuel's capability of reducing the NOx can be attributed by the vaporization of water, which lowers the local burning temperature and thus notably reduce the NOx emission. As for the soot reduction, it can be explained by the better air fuel mixing process featured by the enhanced atomization since micro-explosion may occur due to the drastic volatility difference between the different phase of the fuel, moreover, the water dissociation can form hydroxyl radicals during the combustion which help to oxidize the soot thus reduce the soot emission [7].

Although emulsified fuel is considered an environmentally preferable alternative with tremendous emission reduction potential to be incorporated into the current fleet of the diesel engines without major engine modification, there are several issues remained to be solved before the further commercialization of the fuel, among which the stability should probably be most concerned. [8-11]. The ignition delay of the emulsified fuel combustion is another issue as the ignition delay time will increase greatly with the presence of water such that diesel engines cannot start [12]. Ghojel et al. [13] has found that the ignition delay is always longer than that of the diesel fuel. Whereas the injection pressure has little impact on the ignition delay, the ambient temperature could significantly influence the ignition delay especially at higher water content, thus injection modification might be required to maintain the engine performance. In another study [14], it is reported that the ignition process of multi-components is mainly controlled by the more volatile component, and the ignition delay time reduces significantly with the amount of the more volatile component.

The reduction of soot by using the emulsified fuel has been partially explained by the enhanced atomization which is due to the micro-explosion phenomena. Although numerous researches has shown the existence of the micro-explosion for a single fuel droplet [15-22], the presence of such phenomena and the conditions that favors such phenomena in a spray flame is still open to question. The discovery of micro-explosions in droplet combustion arouses interest of researchers in finding similar evidence in the real engine combustion. The velocities of the spray droplets as well as the conditions surrounding the droplets, however, are substantially different from those conducted in single droplet experiments. Therefore, the assumption that micro-explosions can occur in spray combustion needed to be supported by experimental evidence derived from spray studies. The injection and ambient conditions that favor the occurrence of micro explosion in spray combustion is also need to be verified if the assumption holds. The presence of micro-explosion in atomized emulsion sprays were demonstrated in separate experiments by a number of investigators [23-29]. The direct flame photographs, temperature profiles and micro-explosion frequencies have been shown by Fuchihata et al [23]. They reported observation of small droplets whose diameter were less than 50 μm exploding in the spray flame. Wu et al [28] used the laser holography shadowgraph to visualize the spray in a diesel/water/ethanol emulsion in which an apparent raised part can be seen in the main jet body and claimed as the evidence of the micro-explosion. In a recent study of [29], the "glowing spots" has been reported and might have been resulted from micro-explosion.

To the author's knowledge, there is no well-established criterion for the verification of micro-explosion phenomena in the spray flame so far. Observations such as increased cone angle, unusual jet body detachment on the tip/periphery of the spray, glowing spots are generally used as the evidence to support the existence of micro-explosion, yet most of the results are not so convincing as the image were subjected to either poor resolution or background noise, both of which could pose a huge barrier on the identification of the micro-explosion. Moreover, the largest challenge should still remain in the interior property of the phenomena itself as the micro-explosion is a highly transient process and is very sensitive to the fuel properties as well as ambient conditions. In the single droplet tests [16], it has been illustrated that the micro-explosion can occur over a broad range of temperature and waiting times and probability density functions have usually been used to depict the its occurrences under specific conditions, it is reasonable to assume similar scenario for micro-explosions in spray flame that its occurrence is on a statistic base. All these factors make the visual proof of micro-explosion in a spray flame very challenging. On the other hand, with increasingly aroused interest in the studies in multi-component fuel, such as the ternary blends of ethanol-biodiesel-diesel and butanol-biodiesel-diesel, people have speculated the possible existence of micro-explosion by burning these kinds of alcohol oxygenated fuel and have associated the phenomena with the emission reduction potential benefited by adding water, since the volatility of the respective components are sufficiently large to reach micro-explosions. Such speculations have been confirmed by a number of numerical studies [30], yet experimental evidence is still not well understood and need to be explored.

The aim of this study, on the first place, is to explore the evidence of micro-explosion in spray, thus the emulsified diesel fuel of which the droplet micro-explosion has been confirmed in previous studies was applied in an optical constant volume chamber. Diagnostics has been used to investigate the spray characteristic of the fuel with different water content at different ambient temperature and injection conditions. The bubbles' size of the water phase has been measured using microscope and stability tests have been conducted for all the prepared emulsions. The fuel was later injected and combusted in the chamber to investigate the impact of the ambient temperature and injection pressure on the spray penetration and cone angle. Compared with some of the previous studies, the ambient conditions have been widened to a larger range to see if the trends are still hold. Moreover, natural flame images obtained from a slight modification on the experimental setup, together with the spray images, have indicated a more convincing visual proof of the micro-explosion in the spray flame which can be used as a verification criterion in the future studies.

## 2. Experimental Method

### 2.1 Preparation of emulsified heavy fuel oil

An ultra low sulfur diesel (ULSD) obtained by Illini FS was used as a base fuel and the oil phase in emulsified diesel in current study. The cetane index, 90% distillation point, total sulfur, flashpoint, and viscosity of the base fuel regulated by American Society for Testing and Materials (ASTM) were tabulated in Table 1.

Table 1 Base line fuel properties

| cetane index | 40 (min.) |
|---|---|
| 90% distillation point | 293.3-332.2$^{o}$C |
| Total sulfur | 7-15 ppm |
| Flashpoint | 54.4$^{o}$C |
| Viscosity | 1.5-4.5 cSt |

In previous study, the three phase oil-in-water-in-oil (O/W/O) emulsions were reported more stable than two phase water-in-oil (W/O) emulsions [8]. Thus a two-step procedure was utilized to prepare the O/W/O emulsions in this research. A hydrophilic surfactant polyoxyethylene sorbitan monooleate (TWEEN 80) with hydrophilic-lipophilic-balance value (HLB) 15 was added into water for reducing the interfacial tension and retarding the flocculation, coalescence, and creaming between oil and water phases. [9] On the other hand, the lipophilic surfactant Sorbitan oleate (Span 80) with HLB equal to 4.3 was added into ULSD to stabilize the oil phase. A magnetic stirrer (Temper, Fisher Scientific Inc.) was employed to mix and heat the water and ULSD while the TWEEN 80 and Span 80 were added in, respectively.

An O/W emulsion was first prepared by adding 1/9 in volume USLD into water-TWEEN 80 mixture and blended at 10000 rpm for 5 minutes. The above emulsion was then gently poured into the specific amount of ULSD-Span 80 mixture and emulsified for a period of time at 50$^{o}$C and 10000 rpm to form O/W/O emulsion. The blending time period, including 5, 10, 20, 30 minutes, were optimized by the later stability tests. In addition, the HLB value is the most referable parameter of surfactant selection and addition in emulsification process while the higher HLB stands for more hydrophilic tendency of a surfactant. A different HLB values were prepared by Span 80 and TWEEN 80 while the combined HLBs were calculated by the following equation:

$HLB_{pool} = HLB_S \times W_S + HLB_T \times W_T$

Where S and T stands for Span 80 and TWEEN 80; W represents the mass ratio of each surfactant ($W_S + W_T = 1$). In the current study, the tested HLB were used from 5.0~8.0 to prepared 700 mL O/W/O emulsion while the optimization of HLB value took place by stabilizing 20 vol.% water in a O/W/O emulsion and were tested in term of their stability. The water contents varied from 5 to 20 vol.% in this study with the fixed 2 vol.% total surfactant ratio.

**2.2 Fuel stability tests**

There were three parameters have to be optimized by emulsion stability tests, including blending duration, HLB values, and water contents. The following two methods were employed to characterize the stability of the emulsion: (1) a two-week (14-day) continuous record of fuel daily changes; and (2) observation and analysis of W/O droplet sizes using an optical microscope (OLYMPUS BX51TF, TOKYO, JAPAN) with 400x amplification and a charge-coupled device (CCD) video camera (OLYMPUS DP20). The first method observed the destabilization of emulsion after the short term storage. The 15 mL of each tested fuel would be stored in a centrifugal tube at 25$^{o}$C right after their production. The higher separate volume at the bottom of tube after 14-day standing means the less stability of emulsion. In order to estimate the fuel condition after a long-term storage, the second method took place to catch the image and calculate the sauter mean diameter (SMD) of O/W/O bubbles by using the Image-Pro Plus software version 5.0.2.9. By the bubble size distribution and SMD measurement, the tendency of phase separation could be described while the 14-day test showed no separate layer.

**2.3 Experimental Setup and Procedure**

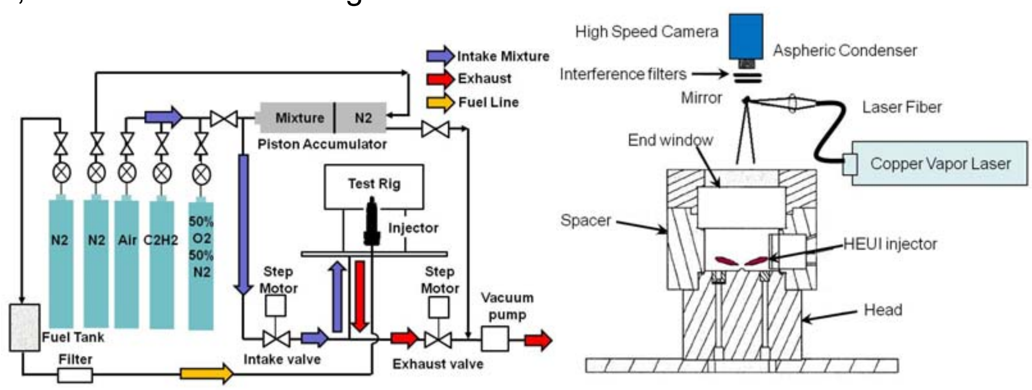
A constant volume chamber with a bore of 110 mm and a height of 65 mm is used in this study. The chamber can imitate the spray and combustion process of a diesel engine, allowing a maximum operating pressure of 18 MPa. The chamber has an open end on the top with a Dynasil 1100 fused silica end window installed opposite to the injector, allowing optical access. The fused silica end window, sealed by a Tamshell energized spring seal, is 130 mm in diameter and 60 mm in thickness, with a high UV transmittance down to 190 nm. A Caterpillar hydraulic-actuated electronic-controlled unit injector (HEUI) is mounted at the center of the chamber head, the configuration of which is tabulated in Table 2.

Table 2 Injector Parameters

| | |
|---|---|
| Nozzle type | VCO |
| No. of nozzle holes | 6 |
| Orifice diameter | 0.145mm |
| Injection duration | 3.5 ms |
| Fuel Temperature | 350 K |

Four injection pressures ranging from 67 MPa to 134 MPa were used in this study. The cylinder wall is heated to 380 K by eight Watlow Firerod heaters, to mimic the wall temperature of a diesel engine as well as to prevent water condensation on the optical windows. Nevertheless, the oil line and fuel line inside the chamber head are kept at 350 K to simulate the situation in an actual engine and stop the fuel evaporation before injection. A Kistler 6121 quartz pressure transducer is embedded in the chamber wall in conjunction with a 5026 dual mode differential charge amplifier.

Studies with similar setups and working principles can be found in Ref [31-36]. To summarize, the procedure is started by filling the chamber to a specified density with a premixed, combustible-gas mixture, including acetylene ($C_2H_2$), 50/50 oxygen and nitrogen, and air as shown in Fig.1.




a. b.

Figure 1. (a) Schematic of the experimental setup; (b) Schematic of test rig

The mixture is pushed into the chamber by a piston accumulator and then ignited with a spark plug. By burning the mixture, a high-temperature, high-pressure environment in the chamber is created. Acetylene, with unity C/H ratio, is used as the combustible gas for its flammability and low window contamination. Equation (1) shows the chemical reaction of the mixture,

$$4\ C_2H_2 + (11 + \zeta)\ O_2 + 65\ N_2 \rightarrow 8\ CO_2 + 4\ H_2O + \zeta\ O_2 + 65\ N_2 \qquad (1)$$

where $\zeta$ denotes the amount of excess oxygen. The chamber ambient contains 21% oxygen, 66.7% nitrogen, 8.2% carbon dioxide and 4.1% water vapor by volume after burning the mixture. The molecular weight for the post-combustion gas mixture is 29.738 kg/kmole, and the density is 14.8 kg/m$^3$.As the products of combustion cool over a relatively long time (~2 s) due to heat transfer to the vessel walls, the vessel pressure slowly decreases. When the desired experimental conditions are reached, the HEUI injector is triggered and the fuel injection, auto-ignition and combustion processes ensue. The ambient gas temperature, density, and composition at injection are determined by the pressure at the time of fuel injection and the initial mass and composition of gas within the chamber. For the experiments presented in this paper, three different ambient temperatures were considered: 800, 1000 and 1200 K, covering both low-temperature combustion and conventional combustion in diesel engine.

**2.4 Spray Studies.**

High speed images for both spray and combustion studies are obtained with a Phantom V7.1 non-intensified high speed digital camera, located above the optical chamber. For the spray studies, the light source is supplied by a copper vapor laser (Oxford Lasers LS20-50) which can be externally controlled to run up to a maximum frequency of 50 KHz with pulse duration of 25 ns. The high-speed camera and the copper-vapor laser were synchronized up to 15,037 frames per second to produce time resolved measurement at a spatial resolution of 512×256 pixels. A Nikkor 105 mm focal length lens was used for the high-speed imaging and an exposure time of 3  s was used. The copper-vapor laser had two-color output, at 511 and 578 nm, with a power ratio of 2:1. To filter out the light at 578 nm for this monochromatic light extinction, two interference filters at 510 nm and 515 nm with 10 nm full width at half maximum (FWHM) achieving a 5 nm FWHM were used. The interference filters also served to block the visible soot luminosities, though the intensive soot emission, especially at high ambient temperature cases, may still contribute to the signal gain and raise noises in the determination of the liquid penetration. The scattered light emitted from the fiber was condensed by an aspheric condenser lens and then reflected via a mirror of 6 mm diameter placed in front of the condenser lens that could be considered as from a point source before entering the chamber. A schematic drawing of the setup is shown in Figure.2. The camera was triggered to start the recording by the injection signal and was set to record for a duration long enough to cover the entire duration of combustion. The spatial resolution of the camera was typically 0.108mm/pixel.

Shadowgraphs based on the diffraction index variation for vapor-air localization has been used for quite a long history. The use of this technique, usually involved two optical windows installed inline on a test chamber, was motivated against elastic scattering because of the difficulties involved in discriminating the border between the vaporized fuel and surrounding air in reacting environments as pointed out by a few researchers [29,31]. In the present study, a similar principle based on the reflection index variation instead of diffraction index variation has been adopted since only one optical accessible window is installed on the top of the chamber. The raw images obtained from each complete injection sequence were first corrected by the first image of the respective sequence which was taken right before the fuel injection. The histogram equalization was then performed to enhance the contrast of each image and minimized the effect of the illumination intensity variation due to the ambient temperature difference and light degradation from case to case. It is also found that this procedure eliminate the bulk noise of the background which later makes easier the determination of both the liquid penetration and cone angle. As the camera will capture stronger reflection signal of the laser beam from spray, the liquid penetration length can be defined as the distance between the injector tip and the first pixel above a preset threshold along the jet centerline. The determination of the threshold has been discussed by a number studies. [31,32] In a most recent study of et al [29], both the centerline intensities and the derivatives have been used to divide the spray jet into continuous liquid core, droplets and fuel vapors. After performing a similar analysis, the author found the determination of the droplets penetration and vapors penetration could be very challenging and subjected to inconsistency due to the aforementioned soot luminosity noise in the background. Therefore, only one threshold was chosen in the present study and was referred as the liquid penetration. It is also worthwhile to mention that penetration is not merely decided by "one" pixel touching the threshold, but rather a 3x3 pixel arrays whose value are all above the threshold, such that the impact of the noise can be minimized. Once the liquid penetration was determined, the cone angle can be measured by finding the farthest 3x3 pixel array above the same preset threshold perpendicular to the jet centerline in the similar fashion as the liquid penetration determination. All the results were averaged from at least five shots for a statistical base.

## 3. Results and discussion

### 3.1 Fuel stability

After 14-day standing, the separate layers occurred in the tubes of HLB equal to 6, 7, and 8, which were 8, 16, and 20% volume of total emulsion, respectively. Therefore, nearly no separate layer, milky white liquid of emulsion indicated that the HLB = 5 is the relatively suitable surfactant composition to the diesel/water interfacial condition. However, both blending duration time and water content did not show any significant effect in the appearance of emulsion because of that all the O/W/O diesel with different water contents and operation times stayed in a stable one crystalline phase. Thus, the more micro-scale observation becomes important.

The bubble size distribution and SMD could also grade the homogeneities of different fuels. Additionally, the smaller droplet diameter leads to the greater reaction surface per volume of fuel, thus promoting more complete combustion [7,8]. Fig. 2a to 2d showed the bubble appearances, sizes and homogeneities of the 5, 10, 15, and 20 vol.% water-containing emulsified fuels under a 400x microscope.

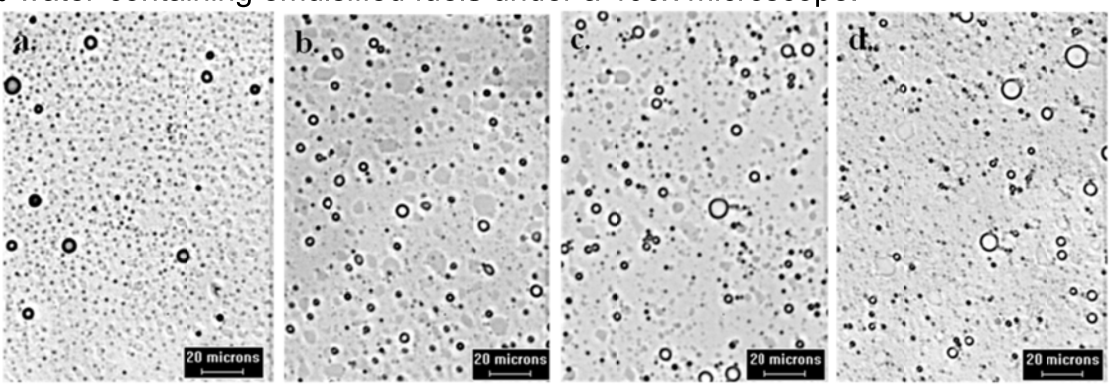



Figure 2. O/W/O bubbles of water-diesel emulsion with (a) 5%; (b) 10%; (c) 15%; and (d) 20% water contents under 400x microscope

Obviously, the 5 vol.% water emulsified diesel (W5) had the smallest and most homogeneous bubble distribution while the big bubbles increased with the water contents. For the quantitative analysis, Fig.3a shows the probability density function (PDF) of the O/W/O bubble sizes. The PDF curves displayed the W5 and W10 had relatively higher fractions of small bubbles around 2 μm while W15 and W20 had lower peak value at the smaller diameter region. Additionally, all of the W10, W15 and W20 had an extra peak close to 4 μm of diameter which means more non-homogeneous distribution then W5. The volumetric density function (VDF) was defined as the volume ratio (vol.%) of (bubble with specific diameter) / (overall bubble volume) in Fig.3b. VDF could amplify the contribution of those huge bubbles with small number, which could not be shown in PDF graph. According to the VDF, the specific bubbles with relatively longer diameter were found around 17~21 μm and 24~30 μm in W15 and W20 curves, respectively. The above results reveal that the destabilizing tendency increased with the increasing water content even W20 still stay in a stable milky emulsion after 14-day standing.

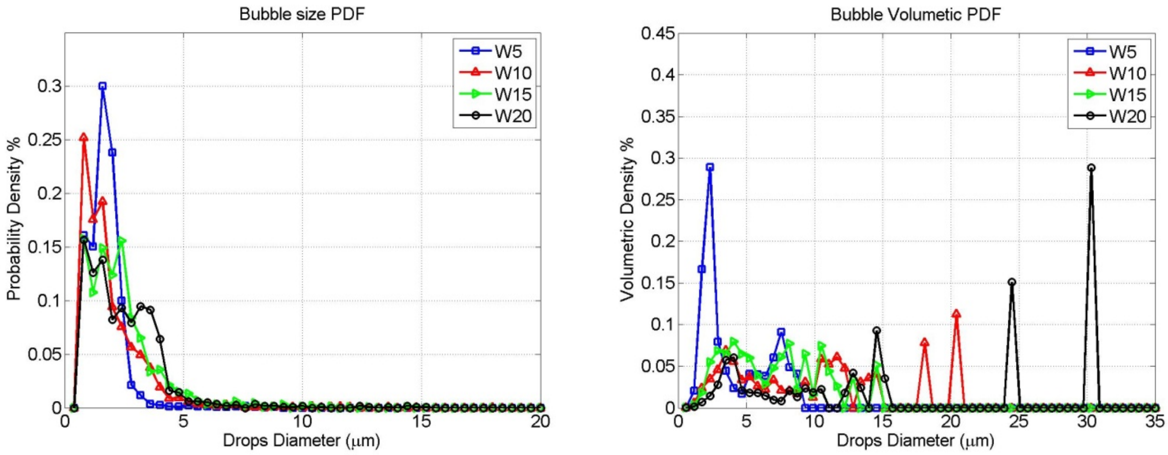



Figure 3. (a) Probability density functions of various W/O droplet diameters; (b) volumetric density fractions of various W/O droplet diameters

In addition, the SMD calculation showed that the extension of emulsification time did not work while the SMD of W20 with 5, 10, 20, 30 minutes operation durations were 30.2, 29.8, 30.1, and 29.8 m, respectively. Therefore, 10 minute was practically used for its efficient and sufficient property. For grading the stability of different water additions, SMD of W5, W10, W15, and W20 were derived as 2.57, 5.91, 10.2, and 29.8 m, respectively. This result again indicated the instability of higher water fraction in emulsion which would flocculate, coalesce, and form cream after a longer time which supported by the aforementioned VDF graphs. However, Fu et al [22] has reported that the micro-explosion strength has a maximum value around 40~60 vol.% of water and decreased in either lower or higher region. The storage energy of nucleation will be small and lead to a weak micro-explosion when the water content was small; when water ratio was large, more water needed to evaporate for keeping on an oil membrane formation, which will lead to small water remained in dispersed bubble [22]. Nevertheless, the micro-explosion strength reduced with the increasing diameter of the dispersed bubble had been investigated. Both multi-component and emulsion bubbles had been verified [37, 38]. Consequently, the W10 and W20 were chosen for their higher tendency and strength of micro-explosion while the storage time should be restrained in two weeks in current study.

## 3.2 Spray Studies.

### 3.2.1 Liquid penetration and cone angle

The evolution of the spray for a single shot for the three tested fuels under different ambient temperatures with a injection pressure of 89 MPa are illustrated in Fig.4-6 while the averaged quantitative measurement of liquid penetration and spray cone angle are shown in Fig.4 and Fig.5 respectively.

Each curve was averaged over at least five shots and shot-to-shot variation was typically within 5%, similar error analysis was also applied to integrate natural flame luminosity as will be discussed later. Under low ambient temperatures, all the tested fuels presented longer liquid penetration due to the lower evaporation rate, which is consistent with some previous studies. [29] This proves the trend keeps the same for emulsified diesel with different water content and different injection pressure. The benefits from low ambient temperature combustion such as better fuel/air mixing and larger portion of premixed burn will also be hold. As seen from Figure 4, the penetration reached a peak rapidly after the injection and gradually shortened once the combustion was started due to the hot gases pouring back into the spray jet together with the radiation from the soot emission, enhancing the vaporization. In comparison the penetration under high ambient temperature were shorter and reached a quasi-steady state immediately after the injection due to the shorter ignition delay and the rigorous diffusion flame swallowing the liquid jet spray. Both W10 and W20 were featured with longer liquid penetration, especially under low ambient temperatures, which can be attributed by the low volatility of the water.

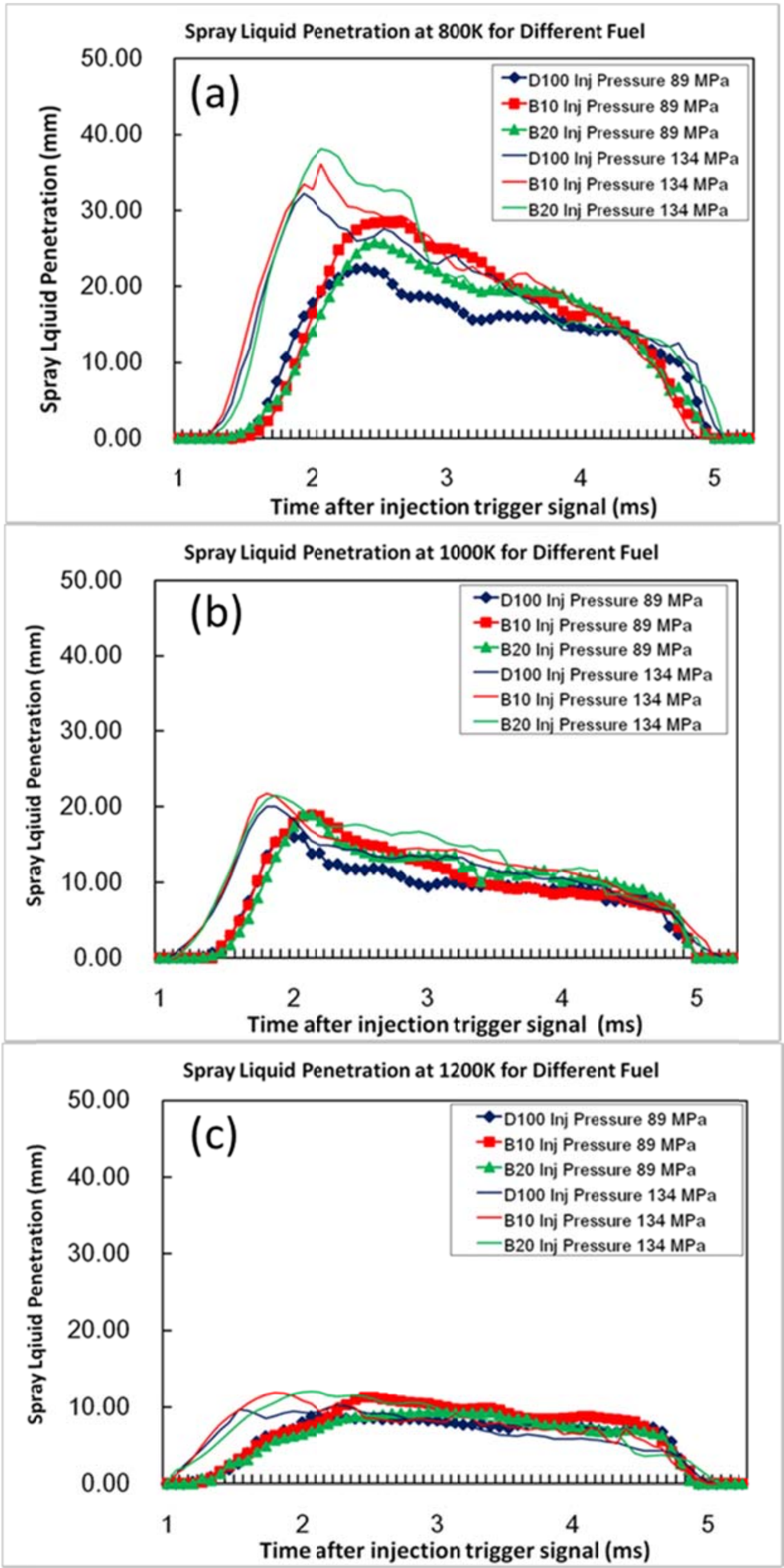


Figure 4. Liquid jet penetration for three tested fuels under ambient temperature of a) 800K, b) 1000K, c) 1200K

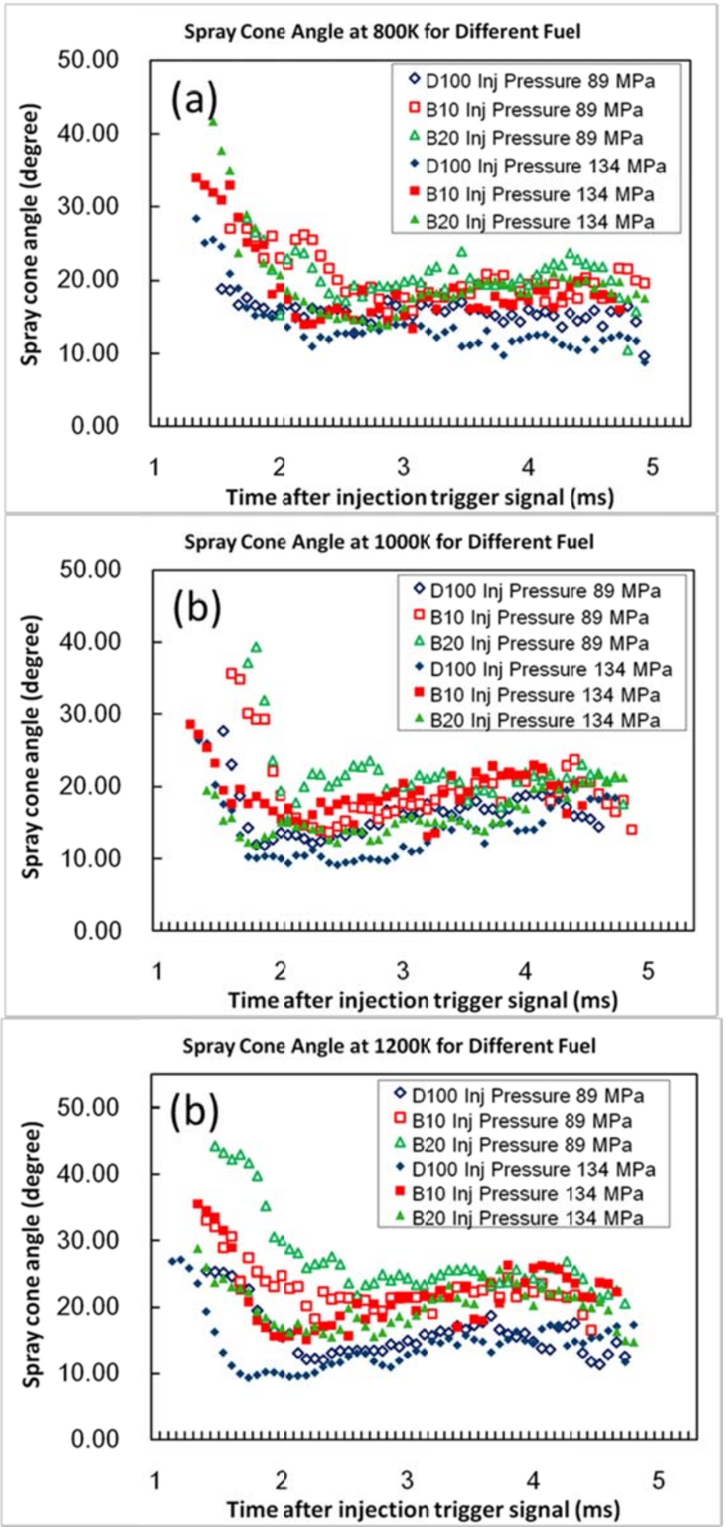



Figure 5. Spray cone angle for three tested fuels under ambient temperature of a) 800K, b) 1000K, c) 1200K

Such variation became less apparent as the ambient temperature increased which can be explained by a few factors. First, the emulsified fuel has higher viscosity and surface tension than regular diesel fuel which is likely to be more resistant to shear and break up [20], as both the ambient temperature is elevated, both property will decline and favors the atomization process, thus makes the penetration comparable to that of the pure diesel. Micro-explosion, as will be further discussed in the later section, could also enhance the atomization process. It is interesting to notice that as the water content increase, W20 actually exhibit a slightly shorter liquid penetration with a injection pressure of 89MPa, indicating the penetration is a competition result from the low volatility of water and a better atomization of the water emulsified diesel.

For all the tested fuels, the spray had a relatively larger cone angle at the very beginning of the injection and narrowed down afterwards. After around 0.4~0.8 ms, it reached a quasi-steady state though fluctuations can still be observed. The fluctuation was mainly resulted from the instability along the periphery of the spray jet and possible background noise. The spray cone angles for water emulsified diesel were generally larger than those of the diesel and the difference was more remarkable under high ambient temperature. To explain the observation, the snapshots for a single spray give more intuitive insights on the spray structure, as it can be clearly seen some abrupt areas raised along the periphery of the spray jet body especially at the early stage of the spray evolution and at relatively high ambient temperatures (>1000K) for the water emulsified fuel which made the spray cone angle larger. Such observation was never seen with the pure diesel under all circumstances indicating it has to be due to the presence of water, or the phenomena of micro explosion.

Regarding the impact of the injection pressure, it is apparently that liquid penetration reached the peak value or the quasi-steady state much faster with elevated injection pressure due to the higher jet velocity. It is also observed that the liquid penetration peak increased with elevated injection pressure under low ambient temperature, though such variation was negligible at high ambient temperature indicating the smaller fuel droplet sizes induced by higher aerodynamic shear evaporated faster and compensated the longer penetration caused by the high jet velocity. The impact of the injection pressure on the spray cone angle is more pronounced, as lower injection pressure resulted in larger cone angle. This is due to the fact that with lower jet velocity, the spray has more time to adjust to the surroundings and less constrained to expand.

## 4. Conclusion

The spray characteristics of water emulsified fuel with different blending ratio were experimentally investigated in a constant volume combustion chamber with different injection pressures and under various ambient temperatures. The bubbles' size of the water phase has been measured using microscope and stability tests have been conducted for all the prepared emulsions. All emulsified fuels tested were stable within a range of two weeks. The fuel was later injected and combusted in the constant volume chamber. Compared with some of the previous studies, both the ambient temperature and injection pressure has been widened to a larger range to see if the trends are still hold for the emulsified fuels in light of the spray penetration and cone angle. It is shown that both W10 (10% water by volume) and W20 were featured with longer liquid penetration, especially under low ambient temperatures, which can be attributed by the low volatility of the water. Notable increased cone angles were observed for emulsified fuel at the beginning stage of injection indicating the occurrence of micro-explosion.

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