Stability behavior of non-surfactant water-in-diesel emulsion fuel using microscopic observation

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Abstract. Water-in-diesel emulsion fuel (W/D) is considered to be a potential alternative fuel that can reduce nitrogen oxides (NO\textsubscript{x}) and particulate matter (PM). W/D is normally produced with addition of surfactant to prolong its stability. However, the dependency on surfactant leads to higher production cost. A concept that can eliminate dependency on the surfactant was introduced by strong mixing concept and direct supply the non-surfactant W/D to a diesel engine. Therefore, the objective of this study is to investigate the stability behavior of non-surfactant W/D using microscopic observation. Water and diesel fuel were mixed using a combination of high-shear mixer and an ultrasonic transducer. The amount of water injected into the system are 5% and 10% by volume, which were labelled as E5 and E10 respectively. The produced non-surfactant W/D was then placed into a petri dish for microscopic observation. The results showed that the average diameter of water droplets observed in E5 and E10 at sedimentation stage were 15.38 and 22.41 \(\mu\text{m}\) respectively. The stability period of E5 and E10 before it sediment were 25 and 67 seconds correspondingly. Overall, it is observed that microscopic observation is a reliable approach to determine the stability behavior of non-surfactant W/D.

1 Introduction

The interest in water-in-diesel emulsion fuel (W/D) for diesel engine application has been growing worldwide as it is regarded as an effective way to reduce the harmful pollutants such as nitrogen oxide (NO\textsubscript{x}) and particulate matter (PM) simultaneously. Moreover, it also improves the combustion efficiency [1–4]. An emulsion is a mixture of two or more liquids that are immiscible in nature where one present as droplet, or the dispersed phase throughout another liquid known which acts as the continuous phase [3]. Emulsion is produced with the assistance of some external means, either by mechanical or ultrasonic agitation; and eventually, with an addition of surfactant to help bind both diesel and water together as one solution [5–8]. The mechanical homogenizing machine is the most used emulsifying device in emulsion preparation; it stirs up the mixture of continuous and...
dispersed phase with the presence of surfactant [8]. Furthermore, there are also applications using ultrasonic agitation to produce emulsified fuel. The ultrasonic waves cause cavitation which creates alternating positive and negative pressure waves to form a strong mixing effect. It has been proven that the combination of mechanical and ultrasonic components in producing W/D yields small water droplet size [7].

The surfactant is required in the formation of W/D to lower the interfacial tension between the dispersed phase and the continuous phase while altogether to stabilize the water droplet phase within the diesel fuel phase to avoid coalescence [9]. Surfactant, also known as surface-active agent is a substance that contain both polar and non-polar groups in their molecules. Hydrophilic surfactants have more attraction to polar liquids such as water, in contrast to lipophilic surfactants which are more soluble in nonpolar liquids such as oil [8]. In W/D, the most commonly used surfactants by researchers are SPAN 80 and TWEEN 80, which are added to a certain percentage to achieve emulsion stability [10]. M.T. Ghannam et. al [9] reported that the separation time of water and diesel (i.e. destabilization of W/D) is dependent on water percentage. The increase of water percentage reduces the W/D stability, in other words, the separation time of water droplets phase from emulsion decreases. The stability of W/D relies on two factors which are the existence of surfactant around the surface droplets and coalescence mechanism [11]. In diesel engine, if the W/D is destabilized during storage or while the engine is in operation, it may cause engine failure and damage other components.

Utilizing the surfactant to a great large scale W/D production is not viable as the price of the surfactant is very high. This hinders the commercialization of W/D in the real-world applications. Therefore, with the motivation to utilize the W/D without using surfactant or so called non-surfactant W/D, a novel concept of a mixing system was introduced in previous study [12,13]. The idea of the system was to focus on the production of W/D through an in-line mixing system by continuously mixing diesel fuel and water, then directly supplies the produced W/D to the engine. This concept of mixing system is known as Real-Time Emulsion Fuel Supply System (RTES). Through this concept, the need of W/D with extended stability period is not relevant anymore, and the addition of the surfactant in W/D can be omitted. The W/D produced from the RTES is thermodynamically unstable, but it needs to stay stable before it is injected into the engine. In previous studies, there are several methods of determining emulsion stability have been proposed such as droplet size analysis, measurements of the physical properties of emulsion [14], accelerated test [15], turbidity ratio measurement [16] and bottle test method. With the latter, the prepared emulsion is placed into beaker/bottle and the time for the emulsion to destabilize is recorded (layer of diesel and water is observed) [9,15]. However, none of the researchers use the microscopic approach to explaining the stability behavior of non-surfactant W/D.

Therefore, the objective of this paper is to investigate the stability behavior of non-surfactant W/D produced by RTES in term of droplet size measurement and stability period by microscopic observation. In the experimental work, a detailed analysis of the stability behavior was conducted with a variety of 5 % and 10 % water content.

2 Experimental setup

The experimental procedures comprised of two stages where the first stage was the preparation of the non-surfactant W/D with 5 and 10 volumetric percent of water using a strong mixing system, RTES. The non-surfactant W/D with 5% and 10% of water were labeled as E5 and E10 respectively. The second stage was the stability behavior analysis of the non-surfactant W/D, carried out by using a microscope. In this analysis, observations of the water droplet size and the duration of W/D to sediment were performed. The produced
non-surfactant W/D need to stay intact or in other words, to be stable for at least 3 seconds, which is the time needed for the fuel travel from the pump to reach the engine. Figure 1 below shows the non-surfactant W/D preparation process and the test setup.

**Fig. 1.** Non-surfactant W/D preparation and test setup.

### 2.1 Preparation of water-in-diesel emulsion fuel (W/D)

For the preparation of E5 and E10, Malaysia grade 2 diesel fuel (D2) was mixed with water using RTES, which is a combination of high shear mixer with a constant motor speed of 1400 rpm and ultrasonic transducer with a frequency of 50 kHz. As shown in Figure 1, the diesel fuel flowed from the fuel tank and water was injected into the mixing chamber. Both liquids were mixed with high shear force and acoustic wave to produce W/D. Here, no surfactant was involved in the process. The amount of water (5% and 10%) was regulated by a controller placed in the RTES control panel. RTES then supplied the non-surfactant W/D emulsion continuously and instantaneously. Table 1 below shows the properties of diesel fuel, used in the experiment.

**Table 1.** Specifications of Malaysia grade 2 diesel fuel [2].

| Properties                |       |
|---------------------------|-------|
| Cetane index              | 54.6  |
| Viscosity at 40°C, cSt    | 4.642 |
| Density at 15°C, kg/L     | 0.8538|
| Carbon, wt %              | 84.1  |
| Calorific value, MJ/kg    | 45.280|
| Total sulphur, mass%      | 0.28  |
| Flash point, °C           | 93    |

In the sampling process, the non-surfactant W/D was transferred into a graduated beaker. Then, a small portion of the fuel was placed into a petri dish for observation and
measurement. While the behavior of the fuels were recorded by a microscope, the destabilization process of E5 and E10 emulsion process were also taken in video format with a duration of two minutes. For this purpose, a super resolution digital microscope with a magnification range of 100 to 1000 times was used. The specifications of the microscope are listed in Table 2.

Table 2. Specifications of the digital microscope.

| Properties                          | Value                                           |
|-------------------------------------|-------------------------------------------------|
| Model                               | KEYENCE VHX-200E digital microscope, equipped with VH-Z100R zoom lens |
| Magnification range                 | 100 to 1000 times                               |
| Image size (pixels per image)       | 1600 x 1200                                     |
| Video size (frames per second)      | 15                                              |

3 Results and discussion

As shown in Figure 2, the water droplet size gradually increased proportionally with time. At 10 seconds, the average diameter of water droplet size in E10 was 15.38 μm and the water droplets were moving freely at this stage. An emulsion is characterized as thermodynamically unstable; thus, the repulsive force between the droplets become weaker as time elapses. Ghannam et al. defined that the stability of the W/D by measuring the amount of water separated over time [9]. However, in this analysis, the stability of W/D ended when water layer began to appear at the bottom of the beaker. In other words, the W/D is described as destabilize when the sedimentation process occurs. As illustrated in Figure 2, after 25 seconds, the droplets became more attracted to each other, and the average droplet diameter reached 24.41 μm, which was bigger than previous droplet size, at 15.38 μm. The larger water droplet started to sink to the bottom of the beaker, which signified the sedimentation process to begin after the 25th second.

Figure 3 shows the sequence of sedimentation process of the same E10 emulsion, recorded after two minutes of mixing. It was evident that the size of water droplets become bigger with time. In the destabilization process of W/D emulsion, initially, the droplets underwent a flocculation process, where the water droplets move toward each other. This was due to the weaker repulsive bond between the droplets. They then formed three dimensional (3-D) clusters in which multiple droplets were bounded and separated only by a thin-film. The thickness of the thin-film was then slowly receding as it dictated by the Van der Waals force and when it reached its critical limit, the film broke, and the droplets formed larger water droplet (i.e. coalescence). Finally, followed by the sedimentation process, where large water droplets sank to the bottom of the beaker due to the density difference between water and diesel fuel. The process continued until the water was completely separated from diesel fuel and the W/D turned into two distinct layers.

The stability experiment for E5 was conducted under the same experimental condition as E10. Similar behavior and pattern with E10 was observed, where at the initial stage, the droplets were freely moving in the emulsion continuous phase. After 67 seconds, the sedimentation process occurred. Here, the average water droplet size observed in E5 is 9.54 μm which was smaller compared to the droplet size found in E10 (22.41 μm). This was because the amount of water in E5 injected into the system was less than in E10. In contrast, the sedimentation process in E5 takes 2.68 times longer than in E10. While in E5, it occurred at 67 seconds, in E10, it took place at 25 seconds. This implied that non-surfactant W/D with higher water percentage reached sedimentation stage faster compared
to one with lesser water percentage. Figure 4 shows the water droplets of E5 after 15 seconds of mixing, at a magnification of 1000x.

Fig. 2. The sequence of water droplet size in E10 emulsion (1000x magnification).

Average droplet size diameter: 15.38 μm

Average droplet size diameter: 24.41 μm

Fig. 3. The sequence of the water droplets destabilization behavior in E10 after 2 minutes of mixing. (100x magnification).
Fig. 4. Water droplet in E5 after 15 seconds of mixing.(1000x magnification).

4 Conclusions

This study highlights the stability behavior of non-surfactant W/D produced by a strong mixing system, RTES. Based on the experimental results, the average droplet size diameter of E5 and E10 at sedimentation stage was 9.54 \( \mu m \) and 22.41 \( \mu m \) respectively. Higher water percentage influence the droplet size and the stability period of non-surfactant W/D. From the observation, the stability period of E5 before reach the sedimentation stage was 67 seconds, while in E10, it was 25 seconds. This shows that both E5 and E10 are stable and exceeded the time it takes for the fuel from the pump to reach the engine.

Overall, it is observed that the evaluation of non-surfactant W/D stability behavior using microscopic observation technique is a reliable method. The characteristic of non-surfactant W/D that has been analyzed make it a potential alternative fuel which can be used in diesel engine.

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