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Effects of oxidized biodiesel on formation of particulate matter and NO_x from diesel engine

Hyungkyu Kang*,, Hoyoung Song*, Jonghan Ha*, and Byung-Ki Na**,†**

*Korea Petroleum Quality & Distribution Authority, 33, Yangcheong 3-gil, Ochang-eup, Cheongwon-gu, Cheongju, Chungbuk 28115, Korea
**Department of Chemical Engineering, Chungbuk National University, 410 Seongbong-ro, Heungduk-gu, Cheongju, Chungbuk 28644, Korea
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Abstract−A test was conducted to investigate the effect of pure biodiesel without additives on formation of particulate matter (PM) and nitrogen oxide (NOx) in the exhaust gas of a diesel engine. Pure biodiesel from waste cooking oil without adding any additive was used. The biodiesel was oxidized at 110 °C for 10 days and blended with commercial automobile diesel oil distributed in the market as a testing fuel. Blended fuels were produced by adding 10% of oxidized biodiesel and un-oxidized biodiesel to automobile diesel oil, respectively. Material properties such as density, kinematic viscosity, oxidation stability, and cetane number were tested. Emission tests were conducted using a large diesel engine of direct injection type, inline six-cylinder, 4 stroke, turbocharger and intercooler. The oxidized and unoxidized biodiesel blends did not show any difference in density and kinematic viscosity. The oxidation stability of the oxidized biodiesel blends was lower than that of the unoxidized biodiesel blends. In the emission test, the two blends showed almost no difference in the total number of concentration of the micro-particles, and also showed almost no difference in particle size distribution such as nucleation mode and accumulation mode. On the other hand, the oxidized biodiesel blends showed less PM and NOx emission than the unoxidized biodiesel blends.

Keywords: Particulate Matter, Total Particle Number, NO_x, Hydroperoxide, Fatty Acid Methyl Ester, Lower Heating Value

INTRODUCTION

The particulate matters (PMs) of exhaust from a diesel engine are comprised of soot, soluble organic fraction (SOF), and very small amount of sulfate. The SOF can adhere to the surfaces of carbon particles in the process of dilution and cooling or can be atomized into smaller particles [1,2]. While particulate matter of diesel engine is regulated based on the weight, several countries added particle number to the EURO 5 vehicle emission regulation.

Many studies have been conducted on the number and size distribution of the particles emitted from diesel engines. According to these studies, particle number distribution of a general diesel engine is classified into three forms of nucleation mode, accumulation mode, and coarse mode. Nucleation mode is mostly comprised of carbon material and sulfur component, and of the particles of diameter between 5 nm and 30 nm. Particulate matters account for most (90% or more) of the total number, and their effect on the mass is rather small (1-20%). Accumulation mode is related to soot and volatile materials adsorbed into it, and the particle diameters are between 30 nm and 1.0 μm. Although the particle number of accumulation mode is smaller than that of nucleation mode, its effect on the particle weight is bigger. Coarse mode is comprised of the particles deposited in the engine cylinder or the vehicle exhaust emission system, and PMs of 1.0 μm diameter or bigger account for about 5-20% of the total weight.

A wide range of studies have been conducted on biodiesel and blended diesel fuel using diesel engines. While the amounts of PM emitted from both biodiesel and blends are reduced in the estimation by the gravimetric method, the total particle numbers have shown a conflicting result in several studies [3-7]. In particular, Tsolakis [4] reported that the amount PM emitted from the combustion of biodiesel is smaller, but the numbers of NOx and micro-particles are bigger. Tinsdale et al. [7] reported that the number of micro-particles emitted from the combustion of biodiesel was found to be reduced by up to 16%.

There are many methods to measure the particle number and particle size distribution of a diesel engine. Scanning mobility particle sizer (SMPS) shows very good particle distribution resolution, but its moment particle measurement resolution is insufficient. Electrical low pressure impactor (ELPI) shoes good moment particle measurement resolution, but its particle distribution resolution is lower than that of SMPS. Condensation particle counter (CPC) is used to measure particle number at a certain velocity or cycle, but it does not show particle size distribution. Engine exhaust particle sizer (EEPS) spectrometer can be used for transient cycle to determine particle size distribution. Though its particle distribution resolution is lower than that of SMPS, its high collecting capacity can measure not only engine exhaust gas in real-time but also

†To whom correspondence should be addressed.
E-mail: nabk@chungbuk.ac.kr

most of the particles (size between 5.6 nm and 560 nm) emitted from a diesel engine.

Many studies have been conducted on the effect of particulate matter (PM) and nitrogen oxides (NOx) by the biodiesel [8-12]. Experiments conducted by Schumacher and Borgelt [9] running on 100% biodiesel in a pickup have shown increases of 13% in NOx, while PM decreased in 20%, compared to pure diesel. Similar results were found by Chang et al. [10], using blends of diesel with methyl and isopropyl esters of soybean oil. On the other hand, Peterson et al. [11,12], in experiments conducted in the same way, found a decrease of about 12% in NOx emission, while PM emissions increased about 10% relative to pure diesel [8].

In this study, we investigatee the effect of formation of PM and NOx in the exhaust gas generated from a diesel engine by using the fuel mixed with the pure biodiesel with no additive and oxidized biodiesel. Specifically, the main subject of this study was to (a) analyze the physical/chemical property of fuel mixed with biodiesel including no additive and oxidized biodiesel, (b) investigate the size distribution of PM in the exhaust gas, (c) measure the particulate matter (PM), particle number (PN) and nitrogen oxide (NOx).

EXPERIMENTAL METHOD AND DEVICE

1. Testing Fuels

The biodiesel used in this study was pure biodiesel produced from waste cooking oil without adding any additives, and was used after being oxidized for ten days in an unsealed steel container at 110 °C. It is the same temperature condition of EN14112, which is the test method of evaluating oxidation stability of fatty acid methyl esters (FAME). The oxidized biodiesel and unoxidized biodiesel were blended with automobile diesel of sulfur content of 10 ppm or lower distributed in the market of which the content of biodiesel was about 2% and it was used as the testing fuel.

2. Testing Engine

A large diesel engine (12 Lt) of direct injection type (common rail), inline six-cylinder, 4 stroke, turbocharger and intercooler was used as the testing engine. The detailed specification is shown in Table 1. The testing engine with a fuel supply system and a cooling water supply was designed to be controlled by the automatic control system of AVL, which was connected to an engine dynamometer of AC type with the maximum control output of 440 kW.

3. Emission Analysis

The exhaust gas from the diesel engine was directly collected from the vent pipe. The collected exhaust gas was diluted using a rotating disk thermo-diluter, and the particle number and distribution of PM were measured using an EEPS device of TSI. PM was measured using a SPC 472 of AVL. NOx was measured using a MEXA 7200DEGR of Horiba, and chemiluminescence detector (CLD) method was applied.

4. Testing Procedure

Vehicle emission tests were conducted in the order of automobile diesel, unoxidized biodiesel blends (BD10) and oxidized biodiesel blends (OxiBD10). Test conditions were set at six conditions by applying 10%, 50% and 100% of load conditions at 1,800 rpm, which was the maximum output speed, and at 1,200 rpm, which was the maximum torque of the test engine, respectively. The device was operated for a minimum of five minutes in each test condition and the exhaust gas data was continuously collected from the exhaust pipe. To minimize uncertainty of test, particle number and distribution were measured continuously for five minutes, and particle distribution, particle number, and gaseous emission were estimated by averaging the data collected for the last two minutes in each test condition. The particle number collected from the exhaust pipe was converted to #/kWh using the following formula:

PN : total particle number (#/kWh)
PNC (#/cc) : average particle number concentration
G_{exh} (kg/hr) : average flow rate of exhaust gas in the exhaust pipe
DF : dilution factor
ρ : density of exhaust gas (1.293 kg/m^3)
P (kW) : actual power

The mass of the particulate matter was converted to g/kWh using the following formula:

PM : particulate matter (#/kWh)
M_{tot} (g) : amount of the diluted exhaust gas that has passed the collection filter
G_{exh} (kg/hr) : average flow rate of exhaust gas in the exhaust pipe
M_f (mg) : mass of the PM collected by the collection filter
M_{sec} (g) : amount of the air diluted second time
P (kW) : actual power

Table 1. Specifications of testing engine

Model	D6CB
Type	Turbocharged DI, 4-cycle
Cylinder arrangement	6 Cylinders, in line
Bore and stroke (mm)	130 and 155
Displacement (cc)	12,344
Maximum power (ps/rpm)	440/1800
Maximum torque (kgf·m/rpm)	206/1200
Injection system	Common-Rail
Emission regulation	EURO 3

RESULTS AND DISCUSSION

1. Analysis of Biodiesel and Blended Diesel Fuel

Blends were produced by adding 10% of oxidized biodiesel and unoxidized biodiesel to automobile diesel, respectively. It was used for material property tests to investigate the physical/chemical characteristics of testing fuel and the result is shown in Table 2.

The oxidation stability of the unoxidized biodiesel was shown to be 1.63 hours, and that of the oxidized biodiesel was 0.62 hour. As no additive (including antioxidant) was added to the unoxidized biodiesel, it did not satisfy the quality standard. As the reactivity of the peroxy free radical generated in the oxidation process of biodiesel is superior, it reduces the induction period [13]. Due to such an effect, the oxidized biodiesel showed lower oxidation stability than that of the unoxidized biodiesel. There are many researches related to the oxidation of biodiesel.

The oxidation process of biodiesel is classified into initiation,

Table 2. Properties of pure biodiesel and biodiesel blends

	Test method	BD100 (unoxidation)	BD100 (oxidation)	EN1421 4	BD10 (unoxidation)	BD10 (oxidation)	EN 590
Density (15 °C, kg/m^3)	KS M ISO 12185	882	883	860-900	832	833	820-845
Kinematic viscosity (40 °C, mm^2/s)	ISO 3104	4.199	4.210	3.5-5.0	2.605	2.594	2.0-4.5
Cetane number	ASTM D6890[a]	56.00	61.00	-	52.84	53.25	-
	EN ISO 5165[b]			51 min			51 min
FAME content (%v/v)	EN 14078	-	-	-	8.38	8.45	-
Total acid number (mg KOH/g)	KS M ISO 6619	0.1717	0.264	-	0.048	0.0957	-
Sulfur (mg/kg)		1.867	1.190	10	2.605	2.594	10
Oxidation stability (110 °C, hr)	EN 14112[c] (EN15751)[d]	1.63	0.62	6 min	13.95	3.14	20 min

[a]ASTM D6890: Standard Test Method for Determination of Ignition Delay and Derived Cetane Number& Ipar; DCN & rpar; of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
[b]EN ISO 5156: Petroleum products-Determination of the ignition quality of diesel fuels-Cetane engine method
[c]EN 14112: Fat and oil derivatives-Fatty acid methyl esters (FAME)-Determination of oxidation stability (accelerated oxidation test)
[d]EN 15751: Automotive fuels. Fatty acid methyl ester (FAME) fuel and blends with diesel fuel. Determination of oxidation stability by accelerated oxidation method

propagation and termination reactions [14], and hydroperoxide (ROOH) was generated in the initiation reaction. Generation of ROOH from biodiesel was known to be related to increase in cetane number [15]. The generated ROOH was decomposed into aldehyde such as hexenal, heptenal and propane [16], and polymer might be generated through oxidation polymerization [17]. The oxygen content of the oxidized biodiesel was reported to be about 21.8%, which was bigger than that of the oxidized biodiesel of about 11.8%

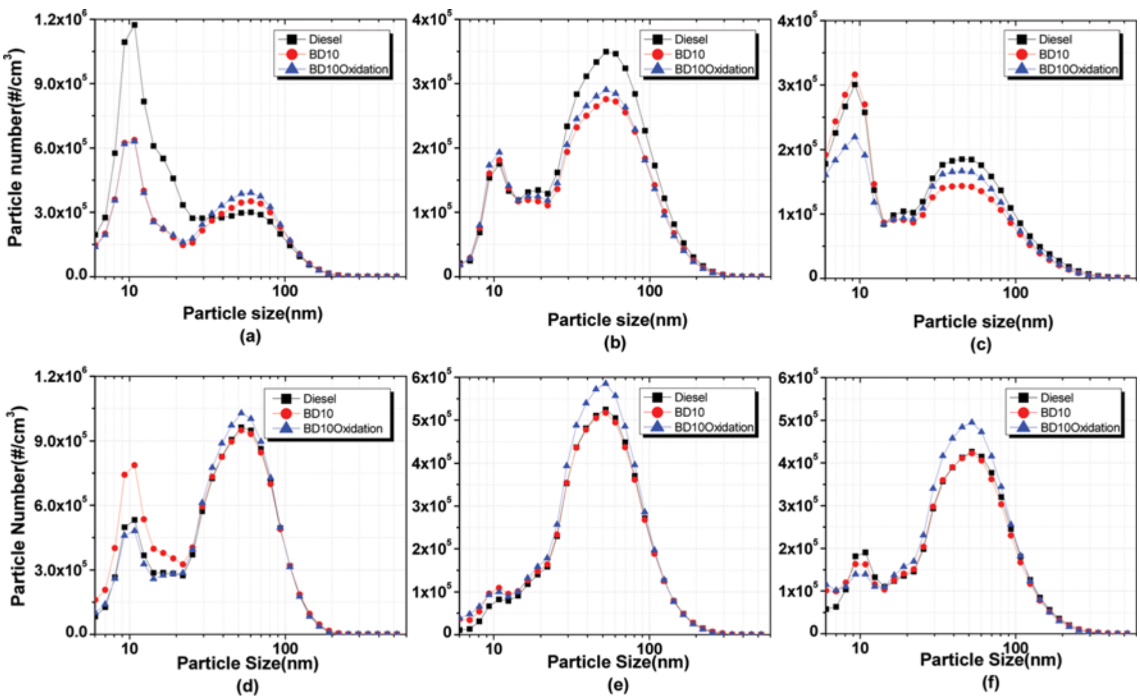

Fig. 1. Particle size distribution for diesel, unoxidized biodiesel blends (BD10) and oxidized biodiesel blends (BD10 oxidation); (a) 10% load, (b) 50%, (c) 100% at 1,200 rpm, (d) 10%, (e) 50%, (f) 100% at 1,800 rpm.

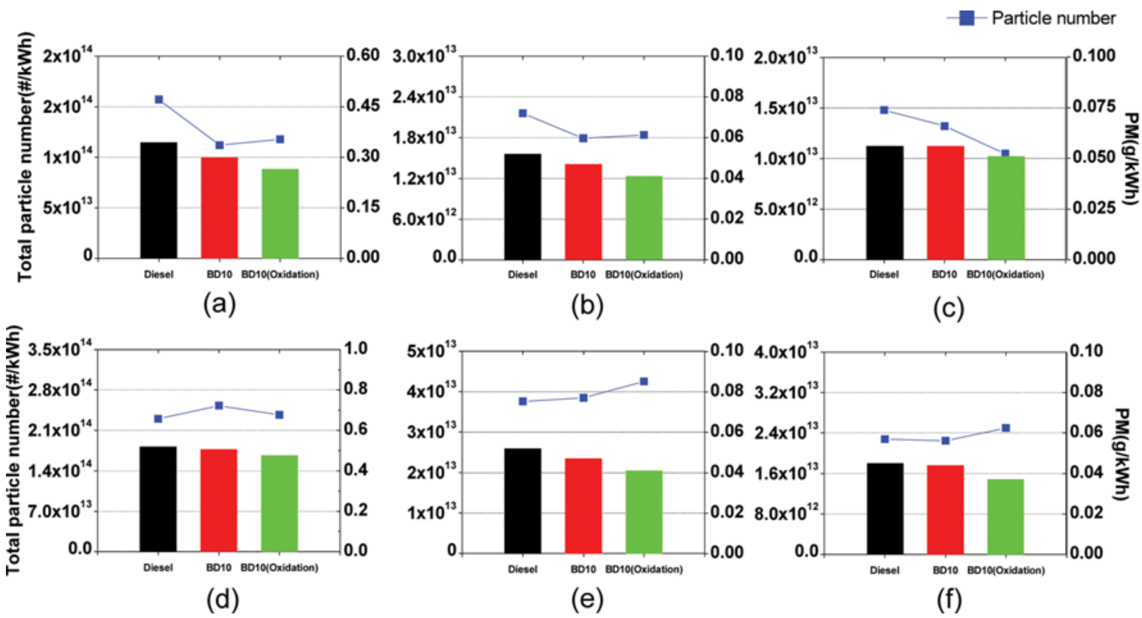

Fig. 2. PM and PN for diesel, unoxidized biodiesel blends (BD10) and oxidized biodiesel blends (BD10 oxidation); (a) 10% load, (b) 50% load, (c) 100% load at 1,200 rpm, (d) 10% load, (e) 50% load, (f) 100% load at 1,800 rpm.

[18]. On the other hand, all other material properties with the exception of oxidation stability satisfied the quality standards of automobile fuel (EN590) and biodiesel (EN14214).

2. Size Distribution of Particulate Matter

The particle size distribution was measured with the six conditions, and the result is shown in Fig. 1. All the testing fuels including the biodiesel blends have shown numinodal lognormal distribution or bimodal lognormal distribution which has nucleation mode of 30 nm or lower and accumulation mode of 30 nm or higher in general.

Particle size distribution in all test conditions showed the highest value of nucleation mode for 10 nm, and the highest value of accumulation mode was formed between 40 nm and 60 nm. The particle sizes with the highest values of nucleation mode and accumulation mode did not show any difference depending on the fuel. Though phenomena that the quantities of nucleation mode particles of OxiBD10 and BD10 emitted at 1,200 rpm in 10% load condition were smaller than that of diesel, that the quantities of accumulation mode particles of OxiBD10 and BD10 at 1,200 rpm in 50% load and 100% load conditions were smaller than those of diesel, and that the quantities of accumulation mode particles of OxiBD10 at 1,800 rpm in all the load conditions were larger than those of other two fuels were observed, the differences were shown to be insignificant. Though reduction in the nucleation mode particles can be explained by the fact that the relatively low carbon and hydrogen content of this oxygenated fuel [19-21] reduces fuel molecules considered as precursor [22] of soot in flames through oxidation and pyrolysis materials, the increase in the accumulation mode particles is not clear. Though insignificant differences appeared in some conditions from above results, the differences were of an ignorable level. Therefore, it is shown that the effect of the fine particles generated by oxidation of biodiesel 10% blended fuel and biodiesel was very small.

3. Measurement of Particulate Matter (PM) and Particle Number (PN)

PM and total particle number concentration were measured applying 10%, 50% and 100% load at 1,800 rpm of the maximum output speed and 1,200 rpm of the maximum torque rotation of the test engine, respectively. The results are shown in Fig. 2.

The quantities of PM emitted from BD10 in 50% and 10% load condition at 1,200 rpm of the maximum torque rpm were smaller than that of automobile diesel by about 9.6% and 13.1%, respectively. It was almost the same level in 100% load condition. The quantities of PM emitted from OxiBD10 were measured to be smaller than that of BD10 in all the load conditions, showing values smaller by 8.9% in 100% load condition, 12.8% in 10% load condition, and 11.4% in 50% load condition.

It is difficult to discuss the difference depending on fuel, as the particle number concentrations of BD10 and OxiBD10 have shown smaller differences of about two times or less from that of automobile diesel.

The quantities of PM of BD10 in 10%, 50%, and 100% load conditions at 1,800 rpm of the maximum output speed showed to have an insignificant difference of about 3% from that of automobile diesel. The emission quantities of PM of OxiBD10 were measured to be smaller than those of BD10 by 15.9% in 100% load condition, by 22.4% in 50% load condition, and by 5.9% in 10% load condition.

The particle number concentrations of BD10 in 50% and 100% load conditions were shown to be similar to those of automobile diesel. Though the particle number concentration in 10% load condition was measured to be higher than that of automobile diesel, the difference was shown to be insignificant. The particle number concentrations of OxiBD10 were bigger than those of BD10 in 10% and 100% load conditions, and it was smaller in 10% load condition.

The particulate matters emitted from a diesel engine are basically comprised of soot generated during combustion and organic materials adsorbed into the soot. As biodiesel is an oxygenated fuel containing less aromatic materials, no raw flour, and less unsaturated fatty acid, it generates less soot. Oxygen structurally contained in the fuel reduces generation of soot by more effectively separating carbon that produces precursor of soot in combustion reaction [23]. Also, the oxygen molecules on the soot surface resulting from oxygenated fuel gradually generate surface combustion on the outermost surface of soot. For such a reason, the soot generated from biodiesel has better reactivity than the soot generated from diesel, and goes through an oxidation process considerably different from that of the soot generated from diesel [24]. Such a phenomenon plays the role of reducing the diameter of the soot particle and reducing the total amount of soot. In this study, the quantities of PM from biodiesel blends were smaller than that from diesel in all the test conditions, and such a result is the same as the findings of the previous studies reported.

Though there is a report that the particle number concentration also increases as the content of biodiesel increases [25,26], such a trend is presumed to change depending on the test conditions. Tan et al. [27] reported their research findings that particle number concentration of BD10 may be lower than that of diesel depending on the engine load, and particle number concentration may considerably increase with a higher content of biodiesel. When diesel was compared with BD10, and OxiBD10 with BD10 in this study, the differences in the total particle number concentrations were low to an extent ignorable.

As mentioned in section 3.1, the content of oxygen in biodiesel increases in the oxidation process. According to the material property test of fuels, the cetane number, which is related to ignition property of compression combustion, increased from 56 to 61. The fact that oxygen content in biodiesel induces reduction in the quantity of PM was verified in previous studies. Therefore, in this study, we found that oxygen newly added in the oxidation process of biodiesel and the delay in ignition shortened due to increase in the cetane number reducing the quantity of PM in the oxidized biodiesel blend.

4. Measurement of Nitrogen Oxide

Nitrogen oxide (NOx) can be divided into nitrogen monoxide (NO) and nitrogen dioxide (NO_2), and most of the nitrogen oxide generated in an engine cylinder is comprised of NO. Fig. 3 and Fig. 4 show the result of nitrogen oxide measured at 1,800 rpm, the maximum output speed, and 1,200 rpm, the maximum torque rotation of the test engine, respectively.

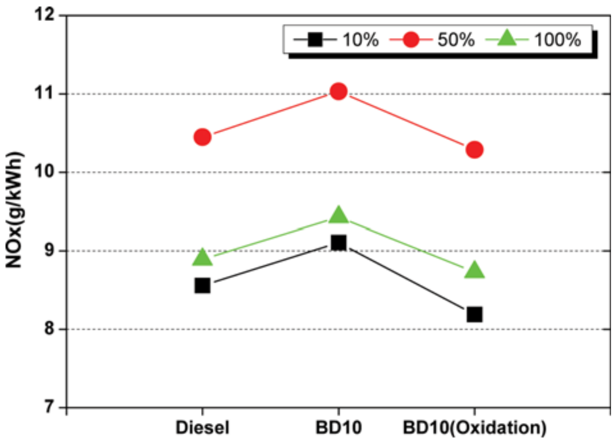

Fig. 3. NOx emissions at 1,200 rpm.

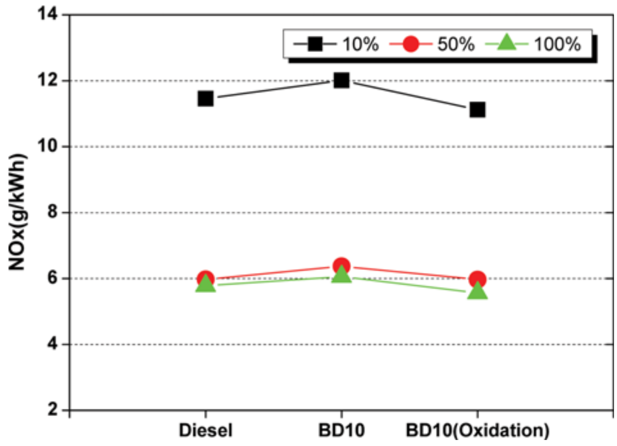

Fig. 4. NOx emissions at 1,800 rpm.

Thermal (Zeldovich), prompt (Fenimore), N_2O pathway, fuel-bound nitrogen and NNH route are the mechanisms involved in generation of NO during a combustion process, among which thermal and prompt are the mechanisms involved in generation of NOx during a combustion process of biodiesel. Thermal NO is generated as the nitrogen in the air is oxidized at the high temperature of 1,700 K or higher, and prompt NO is generated by free radical at the front end of hydrocarbon flame.

According to the exhaust gas test result, BD10 emitted more nitrogen oxide than automobile diesel did by 4.8 to 6.7%, and the increase at 1,200 rpm was shown to be bigger than that at 1,800 rpm. This result showed that biodiesel blends emit more NOx than automobile diesel does as generally known.

Though the reason why NOx increases in a biodiesel blend is known to be because of the molecular structure of biodiesel, diverse opinions are being presented that NOx increases additionally due to adiabatic flame temperature, low radiation of soot, forward movement of injection timing, stoichiometric combustion, oxygenated fuel content [27-29], big size of spray droplets and increased prompt NO.

The NOx emission from the oxidized biodiesel blend was lower than that from the unoxidized biodiesel blend both at 1,800 rpm, the maximum output speed, and 1,200 rpm, the maximum torque rotation of the test engine, respectively. In particular, it showed reduction of 7.4 to 10.1% at 1,200 rpm, and 6.4 to 8.2% at 1,800 rpm. From the results of experiments, it is confirmed that the generation of NOx was reduced by decreasing the combustion temperature and residence time; because the ignition delay time is short due to the effect of the cetane number increased in the oxidation process. These results are consistent with the result of many previous studies [30,31].

CONCLUSION

We investigated the effect of oxidation of biodiesel on formation of particulate matter (PM) and nitrogen oxide (NOx) emitted from diesel engine, and obtained the following conclusions.

(1) From the physical/chemical property test of the fuel produced by mixing with unoxidized and oxidized biodiesel, most analysis items of biodiesel except for oxidation stability were satisfied with the quality standards of automotive fuel and biodiesel. In case of oxidation stability test, the unoxidized and oxidized biodiesel was observed in 1.63 hr and 0.62 hr, respectively. The reason for these results was due to not including the additive like antioxidant.

(2) At the analysis of the size distribution of particulate matter, the distribution by particle size was shown in the 10 nm of the highest level at Nucleation mode and the 40-60 nm of the highest level at accumulation mode, respectively. It means that the particle size distribution from exhaust gas was not to be affected by the oxidation of biodiesel.

(3) At the maximum output and the maximum torque condition, the measurement experiment of particulate matter and particle number was conducted. In case of the measurement of particulate matter (PM), the oxidized biodiesel blend emitted less PM than unoxidized biodiesel blend by 5.9 to 22.4%. It indicated that the particulate matter of biodiesel was decreased by the oxygen generated from forced oxidation process and the shorter ignition delay by the increase of cetane number. In case of the measurement of nitrogen oxide (NOx), when using the oxidized biodiesel blended fuel, the emission of NOx was decreased under the maximum output (1,800 rpm) and the maximum torque (1,200 rpm) conditions. The oxidized biodiesel blend emitted less NOx than unoxidized biodiesel blend by 6.4 to 10.1%. Accordingly, oxidized biodiesel blend was shown to emit less PM and NOx than unoxidized biodiesel in all conditions.

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