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Effect of base oil on the nanostructure and oxidation characteristics of diesel particulate matter

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Keywords

Diesel particle; Base oil; Nanostructure; Oxidation characteristics; Transmission electron microscopy

Abstract

Effect of base oil on the characteristics of emitted particulate matter from a four-cylinder turbocharged diesel engine was investigated. This engine was lubricated with two kinds of base oil (characterized as 150 and 350). The viscosity and flash point of 350 base oil are higher than 150 base oil's. Transmission Electron Microscope, Raman spectroscopy and Thermogravimetric analyzer (TGA) were employed to analyze the particle diameter distribution, nanostructure, graphitization degree and its oxidation characteristics. The primary particles diameter distribution conformed to Gaussian distribution, whereas the mean diameter of 150 base oil was larger than 350 base oil's at 1200 rpm and 2000 rpm. The layer fringe length of 350 base oil was shorter than 150 base oil's, while its separation distance and fringe tortuosity were larger. As to the graphitization degree of 150 base oil, the ratio of the D₁ band to G band areas (A_{D1}/A_G)

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decreased from 1.4162 to 1.0945 at 1200 rpm, indicating that the particle structure of 150 base oil was more ordered than 350 base oil's. According to the TGA results, the elemental carbon mass fraction of 150 base oil was larger than that of 350 base oil, while the volatile fractions were less.

1. Introduction

Diesel engine has been widely used in both stationary and mobile applications for its higher thermal efficiency and fuel economy. However, while its carbon monoxide and hydrocarbon emissions are smaller than that of gasoline engines, diesel engine has also become one of the major sources of the fine and ultrafine particles. [1] According to the relevant research [2-4], these discharged particulate matter (PM) can be harmful to the environment and human health. The detailed investigations on the physical and chemical properties of PM are necessary to provide fundamental knowledge to address these issues.

To date, most research attention has been on how to reduce the particle emissions by different methods, including fuel quality [5], alternative fuel [6, 7], fuel injection system [8, 9] and after treatment system [10, 11]. Diesel particles are composed of spherical or nearly spherical primary particles, and the physical properties of the particles mainly include particle size, morphology and nanostructure. The PM is a kind of complex pollutant composed mainly of dry soot, soluble organic fractions (SOF), sulfates, ash and moisture. A large number of research works have been done all over the world on comparing the particle size distribution, the fractal dimension and nanostructure of the diesel particles by using the Transmission Electron Microscope (TEM) [12-16]. The characteristics and formation mechanism of diesel particles have

been well understood.

There are also many studies focused on the effects of lubricating oil on diesel particle emission. From the works done by Cartellieri and Tritthart [17] and Cartellieri et al. [18], about 90% SOF and 70% of PM mass in diesel particles were caused by lubricating oil. Taylor [19] studied the effect of lubricating oil volatility on diesel emissions and found that with the removal of light fractions of the lubricating oil below 200 to 260°C, the amount of particles decreased. Froelund and Yilmaz [20] found that the impact of lubricating oil to engine-out particulate for contemporary US produced heavy-duty diesel engines ranged from 20% to 30% of the total engine-out particles by introducing sample data. Kahandawala et al. [21] found in a shock tube that dry soot adsorbing oil as SOF was the major formation mechanism of PM. Christianson et al. [22] hypothesized that the mileage accumulated on the lubricating oil would affect PM emissions and the results indicated that the PM mass emissions potential increased as lubricating oil ages. Even though different literatures gave different values of the contribution ratio of lubricating oil on PM emissions, their values are reasonable because they are closely correlated with the method, test bench and work conditions adopted in these studies. There is no doubt that the impact of lubricating oil on diesel PM emissions is considerable. In order to further reduce the PM emission and meet the stringent emission regulations, it is necessary to understand how the lubricating oil influences the particle physical properties.

Lubricating oil is composed of base oil and several additives. For the diesel engine oil, base oil generally accounts for about 90% by mass. The properties of base oil have a great impact on lubricating oil performance [23, 24]. The objective of this study is to find out the effect of lubricating base oil on the nanostructure, graphitization degree and oxidation characteristics of

diesel particles. Two different base oils were used as lubricating oil and particles sample were collected through themophoretic system and quartz filter. Primary particle diameter distributions, nanostructure, graphitization degree, and oxidation characteristics of diesel particles from different base oils were analyzed and compared.

2. Experiment Setup

2.1 Test Engine and Lubricating oil

This study was conducted on YN4100QB, a 4-cylinder direct injection diesel engine, which was equipped with turbocharged system. The details of the engine are shown in Table 1. A hydraulic dynamometer was connected to the engine so as to adjust and measure the engine speed and torque. Experimental data were recorded and stored by the EMS2020 SYSTEM. During the experiment, the engine was operated at low (1200 rpm) and medium (2000 rpm) speeds under 50% load (80 Nm and 120 Nm). For each operating condition, the engine was allowed to run for a period of time until the cooling water temperature and the lubricating oil temperature have attained steady-state values and then began to collect the particle samples.

Two different base mineral oils, 150N and 350N, were compared in this study. Considering the complex composition and characteristics of base oil, we chose viscosity and volatility as the main indices of base oil, since viscosity and volatility are the most important factors that influence the consumption of lubricating oil [23, 24]. The chemical compositions of two base oils was studied by gas chromatography-mass spectrometry. 150 base oil contains undecane, tridecane, tetradecane, pentadecane, hexadecane, docosane, tetracosane, nonacosane, 3-methylundecane, 4-methyldodecane, 3-methyltridecane, 3-methyltetradecane,

4-methylhexadecane, 4-methylheptadecane and 3-methyloctadecane and other substances. 350 base oil contains pentadecane, hexadecane, heptadecane, octadecane, nonadecane, eicosane, heneicosane, tetracosane, nonacosane and other substances. These differences between the chemical compositions have a directed influence on the volatility and combustion oxidability, thereby affect diesel emissions. Specifications of these two base oils are given in Table 2. To ensure the accuracy of experiment results, the amounts of two different lubricants added to oil sump were 4 L. The oil filter was replaced and engine was cleaned in order to avoid the influence of previous oil when lubricant was changed.

2.2 Sample Measurement and Collection

Given the different research targets, particles were collected by different systems. The schematic diagram of the particle sampling system is shown in Fig. 1, including themophoretic system and quartz filter.

Diesel particles can be accessed through the small hole drilled at the exhaust pipe. At this small hole, the diesel particles were collected and removed rapidly. Copper 200 mesh grids coated with carbon or silicon monoxide film were used as filters for TEM sample system and this grid was equipped on a fabricated probe with a width of about 3.05 mm. The probe was driven by a self-developed mechanical system, so it could enter into and exit out of the exhaust pipe rapidly. With the control of a digital timer, the residence time ranged from 0.1 to 0.7 s and varied with the engine operating conditions. In this study, images with a magnification of 40 000 times and 400 000 times were used to analyze the primary particle diameter and nanostructure respectively.

Similarly, there were several other holes drilled at the exhaust pipe for particle collection.

47 mm quartz fiber filters (Tissuquartz 2500QRT-UP, PALL) were used in this study to collect the particles with the fabricated filter supporter. Vacuum pumps with a flow rate of 1 L/s were employed for the particle collection. After each measurement, the filter was taken out from the supporter and placed into the sealed dryer for the Raman spectroscopy analysis and TGA analysis.

3. Results and Discussion

3.1 Primary Particle Diameter Distributions

Different operating conditions have a direct impact on the in-cylinder combustion process so as to affect the particle formation and growth. At higher engine speed, the primary particles have shorter time to form aggregates. With the increasing engine loads, more fuel will be injected into the combustion chamber and burnt during the diffusion mode. [15] As for the lubricating oil, the amount of lubricating oil burnt in the combustion chamber varies due to different characteristics mainly referred to the viscosity and volatility.

In this study, about 25 TEM images and 10 high resolution TEM (HRTEM) images were obtained for each operation condition and used to analyze the primary particle diameter distribution and the particle nanostructure. Fig. 2 presents the representative TEM images of the primary particles sampled at different test points. When the engine was lubricated with 350 base oil, the primary particle boundaries tended to be clearer than 150 base oil's. This is mainly because the base oil evaporated into the combustion chamber and took part in the combustion process. The viscosity of 150 base oil is lower than 350 base oil's and the flash point is also lower, which means that oil consumption of 150 base oil is more than that of 350 base oil. Thus,

more base oil burned in the combustion chamber and more organic fractions adhered to the surface of the primary particles.

Figs. 3 and 4 present the diameter distributions of the primary particles at 1200 rpm and 2000 rpm respectively. The primary particles of agglomerates are approximated by a normal distribution. For 150 base oil, the mean primary particle diameters are 24.854 and 23.286 nm at 1200 and 2000 rpm respectively. For 350 base oil, the values are 23.807 nm and 22.736 nm at 1200 and 2000 rpm.

First, the mean primary particle diameters are about 24 nm, 23 nm, 23 nm and 22 nm under different conditions. Zhu et al. [13] investigated the particle emissions of a light-duty diesel engine and calculated the mean particle diameter at the range of 19-33 nm. Lu et al. [15] studied the effects of engine operating conditions on the size of diesel particles and calculated that the primary particle diameter changed at the range of 23-30 nm. Under different engine types and operating conditions, the mean primary particle diameters were in the range of 19 nm to 33 nm. Previous studies that mainly considered the engine operation conditions also measured the primary particle diameter and concluded the normal distribution of primary particles [13, 15, 25]. Uncertainties will definitely exist during the image analysis process but errors should be within 1 nm.

Second, the mean diameter of 150 base oil is larger than 350 base oil's. This result can be explained by the fact that the base oil is mainly composed of macromolecules, thus the particles were larger when more lubricating oil was burned. When the same engine was operated with blended fuel containing 0.5% by weight of lubricants, lubricants also lead to an increase in primary particle diameters [25]. That lubricants involved in fuel combustion was not only base oil, but also many additives which contain metallic content and sulfur. The sulfur and metallic

content of lubricants contributed significantly to the engine-out PM yield [26, 27], but the main compositions of base oil are organic macromolecules. The particles from base oil are larger than those formed during the combustion of diesel fuel. Viscosity and volatility are the main factors that determine the quantity of oil burnt in the combustion chamber. The higher the viscosity, the less lubricating oil will be pumped into the combustion chamber. The higher the flash point is, the less lubricating oil burnt in the combustion chamber.

3.2 Particle Nanostructure

In this study, about 10 HRTEM images were obtained for each test point and used to analyze the nanostructure of the diesel particles. Fig. 5 presents the representative HRTEM images of the primary particles sampled at different test points. Both the particles show the typical inner core-outer shell nanostructure, which is similar with other studies [28-30]. The inner core with diameter 4-6 nm is surrounded by ordered layer planes. The fringe length is defined as the linear distance traversed by the atomic carbon layer planes, which can also reflect the physical extent of the atomic carbon layer planes. Fringe separation is the mean distance between the adjacent layers, which also represents the possibility for oxygen to access the edge areas of the graphite layer. The tortuosity is the ratio of fringe length to the straight distance between the two endpoints. These three parameters are the primary parameters to quantify the particle nanostructure. Based on the statistical analysis, the values of fringe length, separation distance and tortuosity are given in Table 3.

Effects of base oil on the fringe length of diesel particles under the different operating conditions are presented in Fig. 6 (a) and (b). It can be seen that the fringe length distributions of different base oils under various operation conditions are similar, which mainly range from 0.30 nm to 2.4 nm. Fringes which are shorter than 0.3 nm or longer than 2.4 nm were treated as

artifacts in the image analysis process [31]. It can be found that the fringe length firstly reaches its maximum value around 0.55 nm, and then gradually decreases. Statistical works show that nearly 85% fringes of the particulate matter are between 0.3 nm and 0.9 nm. Only 2% was selected for fringes with length larger than 1.8 nm ranges. The fringe length when using 150 base oil is longer than 350 base oil's at the same engine condition. A long fringe length corresponds to a large layer, which contains low reactivity carbon atoms at the basal plane. This result indicates that the particles of 150 base oil have a more graphitic structure and lower reactivity than 350 base oil's.

Fig. 6 (c) and (d) show the particle fringe separation distance. As for the researched range, we mainly consider the value from 0.3 nm to 0.56 nm. The left data are treated as artifacts during the image analysis process. Although not very regular, the distributions are mainly concentrated in a small range of 0.32 nm to 0.50 nm. Based on the analytical data from Table 3, it can be concluded that the mean fringe separation distance of 150 base oil is less than 350 base oil's. The particles of 350 base oil have larger interior separation distance layers. Thus, oxygen have a greater possibility to access sites on the layer's edge and the particles are easier to be oxidized. According to the measurement uncertainty of fringes, results can be treated to be same. Differences of the fringes length, separation distance may not dominate the influence of soot reactivity [31].

Fig. 6 (e) and (f) show the tortuosity of particles is mainly distributed in the range of 1.0 to 1.7. Tortuosity larger than 2.2 was discarded in the image analysis process. The tortuosity distribution shows unimodal distribution and the peak is concentrated between 1.1 and 1.6. According to the statistical calculation shown in Table 3, the mean tortuosity of 150 base oil and

350 base oil are 1.3703 and 1.3784 at 1200 rpm, while the values are 1.3750 and 1.3814 at 2000 rpm. A large value of tortuosity indicates that more disordered nanostructure occurs when the engine was lubricated by 350 base oil.

3.3 Graphitization Degree

Because the combustion process of diesel engine is short and intense, the generated particles usually have low graphitization degree. In order to characterize the effect of different base oils on particles graphitization degree, the relevant experiments and Raman spectra were studied.

In this study, the structure characteristics of particles are researched by Raman first-order spectrum analysis. The most obvious feature of particle Raman spectra is the D bands and G bands. In line with the findings of previous researches [32-34], the peaks are nearby 1330 and 1580 cm^{-1} respectively. The intensity ratio of G to D bands depends on the proportion of distorted graphene planes. With the decrease of the carbon structure order degree, the intensity of D bands is increased and that of G bands is weakened. The ratio between their integral intensities (I_D/I_G) is related to the structural defects in the basal plane of individual grapheme layers and is used to evaluate the degree of graphite crystal.

In the data analysis process, four curves (D_1 , D_2 , D_3 , G) can better fit the Raman spectrum curve. Fig. 7 shows the Raman spectrum of diesel particles between 800. From the curves, D_1 band peaks are around 1350 cm^{-1} and G band peaks are around 1580 cm^{-1} . According to intensity analysis, conclusions are that the intensity of D_1 band is less than that of G band. For fitting curve, the corresponding results are shown in the Table. 4, which records ratio ($R=I_{D1}/I_G$) between the intensities of D_1 and G bands. It can be obviously found that the values of R when the engine was lubricated by 350 base oil are larger than 150 base oil's at 1200 rpm and 2000 rpm. It means that

the particles of 350 base oil become less ordered and the graphitization degree becomes lower than 150 base oil's.

3.4 Oxidation Characteristics

TGA was used to distinguish between the volatile fraction and solid fraction of the collected particles. The total mass loss is considered to determine the mass fractions of the volatile fraction and elemental carbon fraction. The volatile fraction part is further divided into high volatile and low volatile fraction based on temperature range. TGA has been performed twice for every particle sample. TGA plots for the particles under 1200 rpm and 2000 rpm are shown in Fig. 8. Detailed information of the separation method is shown in Fig. 9. Significant differences in the values of volatile fractions and elemental carbon fraction were measured.

The base oil can influence the organic carbon composition in emitted diesel particles. The results in Fig. 9 reflect that high volatile fraction increased from 14.0% to 59.8%, while from 64.8% to 23.9% for the elemental carbon at 1200 rpm when the engine was lubricated with 350 base oil. The values at 2400 rpm are from 48.3% to 77.3% for high volatile fraction and from 45.2% to 15.4% for elemental carbon when 350 base oil was used as lubricating oil. As was suggested by Kahandawala et al. [21], dry soot adsorbing oil as soluble organic fractions was the major formation mechanism of PM. The base oil may mainly contribute to the elemental carbon. Since the viscosity and flash point of 350 base oil is higher, the amount of lubricating oil burnt in the combustion chamber is less. The base oil influence the combustion process and increases the elemental carbon fraction and thus the volatile fraction will proportionately decrease.

4. Conclusions

In this study, the effect of base oil on the characteristics of exhaust particle was investigated. Two kinds of base oil were adopted and compared in this experiment. Conclusions can be drawn as followings:

- (1) When the lower viscosity and lower flash point base oil was used as lubricating oil, the primary particle boundaries tend to be more irregular and the mean primary particle diameter is larger. The layer fringe length increases, while its separation distance and tortuosity decrease. The Raman spectrum analysis indicates that the graphitization degree of diesel particles increases.
- (2) The base oil can influence the organic carbon composition in emitted diesel particles. PM emissions from high viscosity base oil have higher volatile fraction, mainly because of the reduction of elemental carbon formation.

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Table 1

Diesel engine specifications.

Model	YN 4100QB
Engine type	4-cycle, water cooling
Fuel injection system	Mechanical pump
Suction type	Turbo
Bore×stroke (mm)	100×115
Compression ratio	17.5: 1
Rated power (kW)	80 at 3200 rpm
Max torque (Nm)	290 at 2000-2200 rpm

Table 2

Properties of base oil.

	150 base oil	350 base oil
Condensation point (°C)	-30	-24
Flash point (°C)	190°C	222°C
Viscosity index	108.27	99.69
Kinematic viscosity at 100°C (mm ² /s)	5.15	8.38
Kinematic viscosity at 40°C (mm ² /s)	28.88	64.10

Table 3

The particles nanostructure of different base oils

	150 base oil		350 base oil	
	1200-50%	2000-50%	1200-50%	2000-50%
Fringe length	0.680	0.671	0.674	0.662
Separation distance	0.3836	0.3844	0.3854	0.3862
Tortuosity	1.3703	1.3750	1.3784	1.3814

Table 4

Ratio of D1 and G area concentration.

sample	I _{D1} /I _G (S)	sample	I _{D1} /I _G (S)
150-1200 rpm	1.0945	350-1200 rpm	1.4162
150-2000 rpm	1.1230	350-2000 rpm	1.4388

Fig. 1 Schematic layout of the particle dilution and sampling system

Fig. 2 Representative TEM images of primary particles: (A) 150 base oil at 1200 rpm; (B) 350 base oil at 1200 rpm; (C) 150 base oil at 2000 rpm; (D) 350 base oil at 2000 rpm

Fig. 3 Primary particle diameter distributions for 1200 rpm

Fig. 4 Primary particle diameter distributions for 2000 rpm

Fig. 5 Representative high resolution TEM images of primary particles (A) 150 base oil at 1200 rpm; (B) 350 base oil at 1200 rpm; (C) 150 base oil at 2000 rpm; (D) 350 base oil at 2000 rpm

Fig. 6 Histograms of particles. Fringe length (a and b), separation distance (c and d), tortuosity (e and f)

Fig. 7 Raman analysis of particle for different base oils used.

Fig. 8 Thermogravimetric analyses of particles under 1200 rpm and 2000 rpm

Fig. 9 Elemental carbon and volatile fractions as determined by TGA for different base oils.