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ORIGINAL PAPER

Influence of non-thermal plasma after-treatment technology on diesel engine particulate matter composition and NO_x concentration

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Abstract The effect of non-thermal plasma technology for particulate matter removal and nitrogen oxide emission reduction from diesel exhaust has been investigated. A sample of exhaust was cooled to the ambient temperature and passed through a dielectric barrier discharge reactor. This reactor was employed for producing plasma inside the diesel exhaust. A range of discharge powers by varying the applied voltage from 7.5 to 13.5 kV (peak–peak) at a frequency of 50 Hz has been evaluated during the experiments. Regarding the NO_x emission concentration, the maximum removal efficiency has been achieved at energy density of 27 J/L. Soot, soluble organic fraction and sulphate components of diesel particulate matter have been analysed separately, and the consequence of plasma exposure on particle size distribution on both the nucleation and accumulation modes has been studied. Plasma was found to be very effective for soot removal, and it could approach complete removal efficiency for accumulation mode particles. However, when applied voltage approached 12 kV, the total number of nucleation mode particles increased by a factor of 50 times higher than the total particle numbers at the reactor inlet. This increase in nucleation mode particles increased even more when applied voltage was set at 13.5 kV.

Keywords Dielectric barrier discharge · Diesel emission reduction · Nitrogen oxides · Non-thermal plasma · Particulate matter

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Introduction

Diesel engines emit a lot of pollutants such as carbon monoxide (CO), unburned hydrocarbons (HC) nitrogen oxides (NO_x) and particulate matter (PM) due to the incomplete combustion of diesel fuel. Among different components of diesel emission, diesel particulate matter (DPM) is the most complex component. DPM is essentially composed of a solid fraction (soot), soluble organic fraction (SOF), which is heavy hydrocarbon derived from fuel or lubrication oil and sulphate particulates derived from sulphur. The solid fraction generally consists of elemental carbon while it has not formed any chemical bond with any other elements. Furthermore, metallic ash is another component of the solid fraction, and it is basically composed of metal oxides, sulphates, phosphates and iron oxides derived from the corrosion of exhaust manifold (Merkel et al. 2001). Sulphate particulates are formed in the process of heteromolecular nucleation, where sulphuric acid chemically reacts with the water fraction presented in the exhaust gas (Morawska et al. 2008). Sulphate particulates are considered to be separated from carbon particles, and they are found as nucleation mode particles in the exhaust gas (Maricq et al.

2002). The number of sulphate particles depends on the sulphur content of the diesel fuel directly (Ristovski et al. 2006). The SOF fraction can exist in the form of separated fine droplets in diesel exhaust or can be adsorbed on the surface of carbon particles. The proportion of SOF in the total PM can be different depending on the engine specifications and engine operating conditions (Majewski 2002).

Regarding particle size, diesel engines show a bimodal particle distribution with a combination of nucleation mode and accumulation mode (Kittelson et al. 2006). Nucleation mode particles are mostly consisting of SOF and sulphate particles that are primarily volatile and unstable with a small amount of solid elemental carbon and other materials. On the other hand in accumulation mode, primary carbon particles and other solid materials agglomerate with each other to form a bigger particle and adsorb some SOF fractions and other vapour components on its surface (Majewski 2002). While most of the mass is in the 0.1–1.0 μm "accumulation" size fraction, most of the particle numbers are in the <0.1 μm "nucleation particle" fraction (Seinfeld 1975; Kittelson 1998). Such small particles are inhalable, able to reach the lower human airways and penetrate deep into the lungs where they can enter the bloodstream and even reach the brain (Zhua et al. 2007; Wang et al. 2013). Therefore, there is a considerable concern about the deleterious effect of diesel particles on human health, and DPMs are suspected for a series of adverse effects on the environment (Ramanathan 2007) and human health (Ristovski et al. 2012; Seaton et al. 1995; Sydbom et al. 2001; Omers et al. 2004).

Given the likely health effects of diesel emissions, there is a clear need to consider new technologies to reduce the emissions of diesel engines. Non-thermal plasma (NTP) is believed to be a promising candidate for different emission reduction applications (Zhang et al. 2013; Zhu et al. 2011, 2009). Plasma is the fourth state of matter composed of free electrons, ions, radicals, atoms and molecules in various states of excitation, which have a tendency to remain overall electrically neutral over the large length scales (Inan and Gołkowski 2010). Plasma is divided into thermal or hot plasma and non-thermal or cold plasma. In the thermal variety, the kinetic energy (temperature) of charged particles and the kinetic energy (temperature) of the background gas are similar, while in non-thermal plasma, electrons have a kinetic energy higher than the energy corresponding to the random motion of the background gas molecules (Majewski 2004). Non-thermal plasma contains many kinds of chemically active species generated by many chemical reaction mechanisms such as electron attachment, dissociation, ionisation and excitation. For example, O, O_3, N, N*, N_2^+ and OH can be generated by the dissociation and ionisation of the ambient gases caused by the impact of energetic electrons (Akiyama et al. 2007).

Both gaseous and particle emissions of exhaust gases undergo chemical changes when they are exposed to plasma. The effectiveness of NTP at low temperatures for emission reduction has been mentioned as one of the advantages of this technique in the literature since it can be used in cold start condition and also in catalyst position in which the exhaust temperature is around 150 °C where the catalysts are not so effective (Rajanikanth et al. 2005; Sato et al. 2011). Furthermore, emission reduction in diesel engines at low temperature is important in some special applications of diesel engines such as mining industries where exhaust cooling occurs for safety reasons. In comparison with the numerous publications on NO_x treatment by plasma (Lebouvier et al. 2011; Vinh et al. 2012; Arai et al. 2004; Jolibois et al. 2012; Mohapatro and Rajanikanth 2011; Chae 2003; Yoshida et al. 2009), a limited amount of research has investigated particulate matter (Ye et al. 2005; Thomas et al. 2000; Babaie et al. 2013) and most of it has emphasised PM mass reduction only (Yao et al. 2006; Fushimi et al. 2008). In this paper, the effect of plasma technique on particulate matter constitutes (soot, SOF and sulphates) and NO, NO_2 and N_2O emission concentrations have been studied. A dielectric barrier discharge (DBD) reactor was employed for producing plasma inside the diesel exhaust, and a range of discharge power by varying the applied voltage from 7.5 to 13.5 kV has been discussed. The values of applied voltage refer to peak–peak voltage measured by the oscilloscope during the experiments. Moreover, PM size distribution for a different range of voltages was deliberated. PM size distributions have been considered in conjunction with PM constitute outcomes to obtain a better insight into the plasma effects on diesel particulate matters' structure. This research has been carried out at the Department of Mechanical System Engineering, Gunma University, Japan, in 2013.

Materials and methods

A schematic diagram of experimental set-up is shown in Fig. 1. Experiments were conducted on a 0.4-L, two-cylinder, four-stroke, indirect injection diesel engine with a swirl combustion (Kubota, Z-402E). All the experiments have been conducted under steady-state conditions at a constant engine load of 2 kW. The exhaust gas flow rate passing through the reactor was kept constant at 3 L/min for all experiments. The heat is removed from the exhaust gas to cool it down to room temperature before entering the reactor via a water trap. Condensate water (liquid) has been removed by the water trap from the exhaust gas before

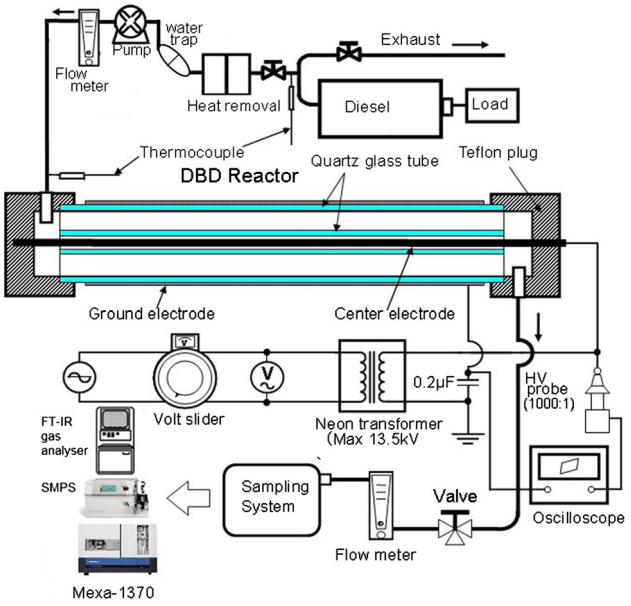

Fig. 1 Schematic diagram of experimental set-up and sampling system

entering the reactor to avoid the blockage and risk of electrocution.

A dielectric barrier discharge (DBD) reactor was used to create plasma inside the exhaust. DBD is an excellent source of energetic electrons with 1–10 eV and high density (Xu 2001). It consists of two concentric quartz tubes, each with wall thicknesses of 2 mm. The outside diameter of the inner tube is 12 mm and inside diameter is 8 mm. For the outer quartz tube, the inside and outside diameters are 22 and 18 mm, respectively. Exhaust passes through the gap between these two quartz tubes. Based on predesigned geometry, the discharge gap is 3 mm. Both ends of the reactor tubes are stoppered by heat-resistant Teflon caps. The centre electrode is a stainless steel rod, having a diameter of $do = 6.0$ mm (SUS304), which is passed through the centre of the inner glass tube. Aluminium foil is used on the outer periphery of the outer glass tube to provide the ground electrode. The effective length of the barrier discharge device in this reactor is 250 mm. AC voltage obtained by boosting a neon transformer (LECIP Inc., 100A-15UHCS) as a commercial power supply (up to 13.5 kV) at 50 Hz is applied between the electrodes to form the plasma. For the experiments, a range of voltages from 7.5 kV up to 13.5 kV has been applied at a constant frequency of 50 Hz. As shown in Fig. 1, a 0.2-μF capacitor is used between the ground and the outer peripheral electrodes to provide the discharge power between these two electrodes.

To observe the effect of plasma on particle composition before and after introducing plasma, quartz-fibre filter papers were utilised to collect PM samples at the reactor outlet. At first, the power supply was turned off and the applied voltage was zero. Then voltage was increased up to 7.5 kV, which is just higher than the required breakdown voltage of the proposed configuration for initiating the plasma. Four more voltage levels (9, 10.5, 12 and 13.5) have been applied to produce stronger plasma inside the plasma reactor. Ten different samples have been collected for each voltage level, and PM components such as dry soot, soluble organic fraction (SOF) and sulphate are measured using a combustion-type PM analyser (Horiba, MEXA-1370PM). On the other hand, the size distributions of particles sampled before and after the DBD reactor are measured by a scanning mobility particle sizer (TSI, SMPS-3034). PM size distribution was measured at reactor inlet and reactor outlet without applying any voltage to find the particle deposition rate inside the reactor. Then, the plasma was introduced inside the DBD reactor at the different aforementioned voltage levels. Size distribution and PM composition results have been analysed in conjunction, to obtain a better understanding of plasma effects on PM composition. Moreover, the effect of NTP on different nitrogen oxides has been considered. To find out the effect of NTP on NO_x concentration, NO, NO_2 and N_2O have been measured by the Fourier transform infrared (FTIR) Horiba Mexa-4000 FT in the reactor inlet and outlet at different voltage levels.

Results and discussion

Emission characterisation of diesel engine

Before studying the effect of plasma on diesel engine exhaust, the gaseous emissions have been measured at different engine loads. CO, CO_2, H_2O, O_2 and C_2H_4 results are shown in Fig. 2. As can be seen, after a rapid fall in CO concentration at 0.5 kW, there is a gradual decrease in CO concentration while engine load increases to 2.4 kW. On the other hand, CO_2 concentration shows the opposite trend and it enlarges with the engine load increase. H_2O, O_2 and C_2H_4 concentration patterns are somehow similar. As it is shown, after a drop in concentrations at 0.5 kW, the concentrations of H_2O, O_2 and C_2H_4 remain almost constant by changing the engine load. Figure 3 shows the results of the nitrogen oxide concentrations on diesel exhaust gas by changing the engine output power. It was confirmed that NO_2 and NO increased along with the increase in the engine output power while N_2O did not change a lot. Also,

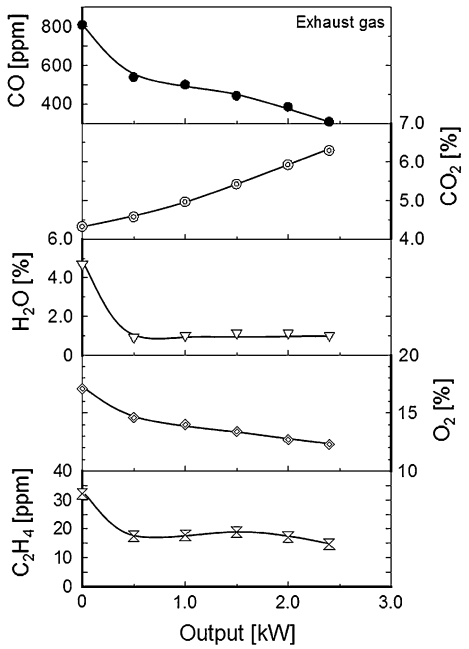

Fig. 2 CO, CO_2, H_2O, O_2 and C_2H_4 concentrations in exhaust gas at different engine output powers

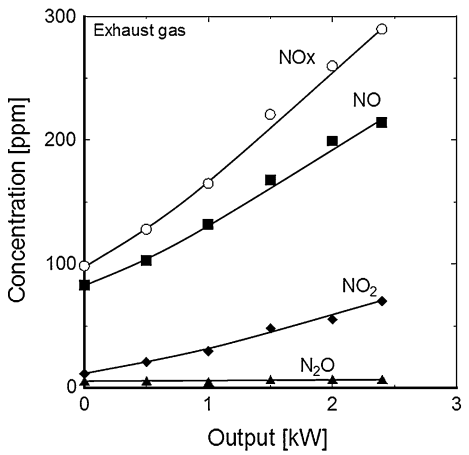

Fig. 3 NO_x concentration in diesel exhaust gas at different engine output powers

nearly 80 % of the emitted NO_x (NO + NO_2) is for NO in this diesel engine at all engine loads.

Discharge power characteristics

The AC voltage obtained from boosting the neon transformer (LECIP Inc., 100A-15UHCS) at 50 Hz as a commercial power supply was applied between two electrodes of the DBD reactor to form the plasma inside the exhaust gas. The supply power source goes through two transformers before reaching the reactor. The primary

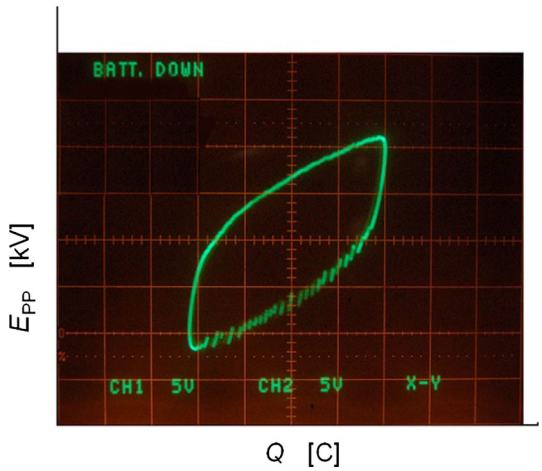

Fig. 4 *E–Q* Lissajous on oscilloscope

transformer, also a volt slider, is used to adjust input applied voltage. The secondary transformer (Neon Transformer, ratio 1:150) converts the input voltage into high voltage in the range from 5 to 15 kV. A high-voltage probe, Iwatsu HV-P30, with ratio 1000:1 is inserted into circuit to minimise load on the oscilloscope. A 0.2-μF capacitor was set up between the ground and peripheral electrode to define the stored charge in DBD reactor. Charge–voltage Lissajous technique is used to measure the discharge power inside the DBD reactor (Hui et al. 2013).

Figure 4 illustrates the charge amount Q [C] of the capacitor and the applied voltage between the electrodes E_{pp} [V] obtained from a KENWOOD, C-S8010 oscilloscope. The area of the Q–E_{pp} Lissajous figure represents the electrical discharge energy per cycle [W (J/cycle)], and the average discharge power [P (J/s = W)] can be calculated by multiplying the energy per cycle [W (J/cycle)] and the frequency [f (1/s)]. The voltage was 7.5 kV for the first experiment, and it is increased up to 13.5 kV. The Lissajous figures for all voltages have been obtained by taking photographs from the oscilloscope screen. Then, by the use of image processing, the discharge power has been calculated for all voltage levels. The result is plotted in Fig. 5. When voltage is 7.5 kV, the discharge power is just 0.02 W, which means at this condition, plasma is very weak inside the exhaust gas. However, when the voltage goes up, the discharge power increases, especially after 10.5 kV. As it can be seen, the discharge power jumps to about 1.4 W at 13.5 kV. At this point, plasma is quite powerful, and we can expect a lot of free radicals and ions and a lot of electron impact reactions inside the exhaust.

Particle size distribution under plasma discharge

The effect of plasma at different voltage levels is shown in Fig. 6. As mentioned before, first the particle size

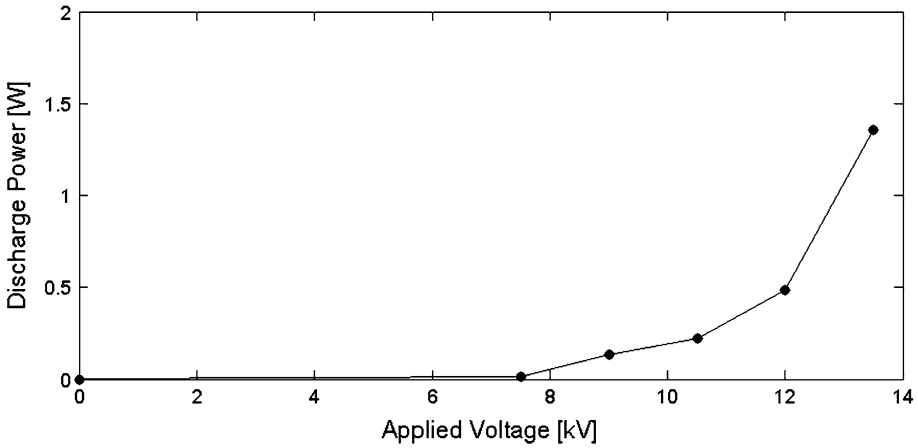

Fig. 5 Average power discharge versus applied voltage for plasma DBD reactor

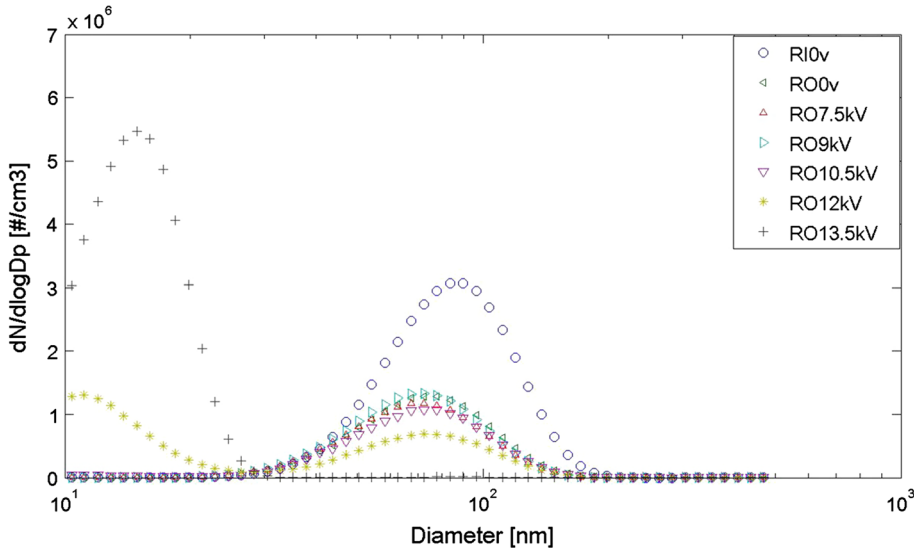

Fig. 6 SMPS particle size distribution at different voltage levels

distribution has been considered at the reactor inlet and outlet without any plasma and then the power supply was turned on. Comparison of particle size distributions at reactor inlet (RI0V) and reactor outlet (RO0V) while plasma has not been formed indicates the tendency of particles to adhere to the walls due to the deposition (precipitation) effects. Therefore, deposition of particulates was found to be an important parameter, which has been considered in all experiments.

For the first case, the applied voltage was 7.5 kV and the particle size distribution is measured at reactor outlet (RO7.5 kV). The voltage was increased up to 13.5 kV (named RO13.5 kV on Fig. 6) by 1.5 kV voltage steps. As discussed in Fig. 5 before, the discharge power and therefore plasma are weak at 7.5 and 9 kV. The former is in agreement with Fig. 6 which shows that the particles at these two voltages are not ionised sufficiently to effect the particle size distribution. We thus conclude that at these two voltage levels the plasma is not powerful enough for removing particles, and most of the PM removal is just due to the deposition.

When the voltage increases to 12 kV, there is a substantial decline in particle number in accumulation mode compared with PM numbers at the reactor inlet. For particles with diameters bigger than 30 nm, the DBD plasma reactor showed 77 % removal efficiency of particle numbers. This is a promising phenomenon, which is obtained by a conventional DBD reactor and can be improved by reactor modifications in future plasma applications. However, when it goes to particles smaller than 30 nm, an increase on nucleation mode particles is observed. When the voltage gets to 13.5 kV, almost all of the particles larger than 30 nm have been removed. On the other hand, high numbers of particle concentrations in nucleation mode were found at this voltage level. PM size distribution peaks to 5.6×10^6 particles, which is 1.8 times greater than the maximum of particles

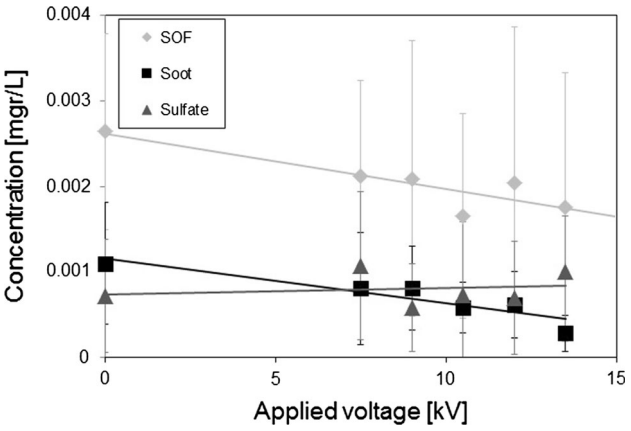

Fig. 7 Effect of non-thermal plasma at different voltages on PM components

number at reactor inlet. These nucleation mode particles can be formed due to the condensation of SOF derived from the accumulation mode particles after oxidisation of their embedded elemental carbons and also due to the condensations from the gas phase.

Effect of non-thermal plasma on diesel particle composition

The effect of non-thermal plasma on soot, SOF and sulphate is shown in Fig. 7. PM samples were collected on QR-100 Silica filters (gas collection efficiency of 99.99 % for 0.3 μm DOP) at the reactor outlet and then analysed by Horiba MEXA 1370PM. Regarding the filter's collection efficiency, most of the collected PMs on Silica filters can be assumed to be from the accumulation mode. When the voltage was zero, no plasma was formed inside the exhaust, and SOF, soot and sulphate content of the emitted particles were 0.0026, 0.0011 and 0.0007 mg/L, respectively. Therefore, particles emitted from the test engine appear to be as wet particles because the SOF content is considerably higher than the soot and sulphate contents. As expected, the sulphate fraction is very low at the reactor outlet measurements, due to the low sulphur content of employed diesel fuel, and remains almost unchanged under plasma treatment.

A continuous decrease in soot concentration of PM by increasing applied voltage can be found in Fig. 7. Soot concentration at the reactor outlet when there is no plasma is 0.0011 mg/L, and it decreases to 0.0003 mg/L when the voltage increases up to 13.5 kV. This is equivalent to about 73 % soot mass reduction at this voltage level when the plasma is formed strongly inside the exhaust. Since the soot basically can be considered as elemental carbon, this effect is expected due to the oxidative potential of plasma. Ozone, NO_2 and other active oxygen species (such as O_2+, O_2-, O, O+, O− and ionised ozone) produced by plasma are the most important suspicious for oxidising the soot component of emitted particles (Okubo et al. 2008). This finding indicates a good potential of non-thermal plasma applications for carbonaceous particle removal. The main reactions for oxidation of carbon based particles in plasma state can be considered as follows (Okubo et al. 2004):

$$C + 2NO_2 \rightarrow CO_2 + 2NO$$
$$C + NO_2 \rightarrow CO + NO$$
$$C + O_3 \rightarrow CO_2 + {}^1/_2O_2$$

Despite some fluctuations in SOF data, the general trend of an increase in SOF concentration can be found in Fig. 7. A major fall about 37 % in SOF concentration is observed at 10.5 kV; however, with the increase in voltage to 12 and 13.5 kV, the SOF ascends a bit. This outcome is in agreement with the results of Fig. 6, which shows a bimodal size distribution at 12 and 13.5 kV. While at these voltage levels the total particle number in nucleation mode increases, PM numbers on accumulation mode particles decline significantly. This increase in SOF content and decrease in PM numbers in accumulation mode can be explained by some kinds of rearrangements on PM structure. At these voltage levels, a considerable decrease in PM numbers and the soot component in accumulation mode are found, so we can assume that some SOF constitutes removed particles adhere on the surface of the remaining particles in accumulation mode and form some more wet particles. This assumption should be studied in more detail in the future.

Effect of energy density on PM removal efficiency

Energy consumption is an important challenge in plasma applications for PM removal. Energy density is usually employed for evaluating the discharge energy and defined as the ratio of discharge power to the exhaust flow rate. The magnitude of energy density affects the removal efficiency on plasma applications. The effect of energy density on removal efficiency of total particulate matter is shown in Fig. 8. As discussed previously, the PM numbers were increased in nucleation mode particles at 12 and 13.5 kV, so the PM removal has been considered for particles larger than 30 nm.

As can be seen from Fig. 8, there is a direct relationship between discharge power and removal efficiency. At first, the removal efficiency for the particles larger than 30 nm is about 59 %, and it rises to 76 % at

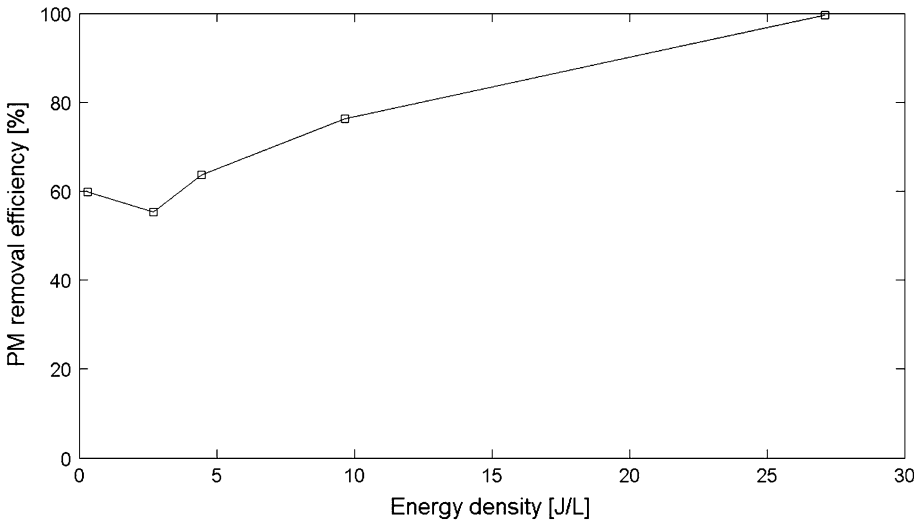

Fig. 8 Effect of energy density on PM removal efficiency

an energy density of 10 J/L. Nearly all particles larger than 30 nm can be removed when the discharge power is about 27 J/L.

Effect of non-thermal plasma on NO_x concentration

The NO_x reduction reactions by using NTP technology can be considered in two main categories (Talebizadeh et al. 2014):

- NO_x removal reactions and
- NO–NO_2 conversion reactions.

In the first category, NO_x molecules can be changed to N_2 and O_2 molecules under plasma state through the following reactions:

$$N_2 + e \rightarrow N + N + e$$
$$NO + N \rightarrow N_2 + O$$
$$O_2 + O \rightarrow O_3$$
$$N_2 + e \rightarrow N_2(A) + e$$
$$N_2(A) + NO \rightarrow N_2 + N + O$$
$$N_2(A) + N_2O \rightarrow 2N_2 + O$$
$$NO_2 + N \rightarrow N_2 + O_2$$

where $N_2(A)$ represents N_2 metastable state.

In the second category, the dominant reaction is oxidation of NO to NO_2 by the following reactions:

$$O_2 + e \rightarrow O + O + e$$
$$NO + O \rightarrow NO_2$$
$$O_2 + O \rightarrow O_3$$
$$NO + O_3 \rightarrow NO_2 + O_2$$

The effect of non-thermal plasma on nitrogen oxides has been studied during the experiments as well. The samples are collected by a Tedlar bag and analysed by a Fourier transform infrared spectroscopy exhaust gas analyser (Horiba Co., Ltd, MEXA-4000FT) at the reactor outlet. Applied voltages have been changed from 7.5 to 13.5 kV, the same as in previous experiments, and NO, NO_2, N_2O and total NO_x ($NO + NO_2$) concentration have been considered. The results are summarised in Figs. 9 and 10. As can be seen, the change in concentration has been started after 9 kV. Therefore, plasma has not been powerful enough to make any significant variation in nitrogen oxide concentrations before this point. It was found that NO is oxidised to NO_2 due to the existence of different active oxygen species and ozone as it is reported on previous studies (Mohapatro and Rajanikanth 2011; Arai et al. 2004; Fitzsimmons et al. 1999). NO concentration decreased by 72 % and NO_2 concentration increased by 77 % by applying maximum voltage at 13.5 kV during the experiments. On the other hand, a small increase in N_2O concentration has been found during the experiments. The initial concentration of N_2O was about 13 ppm when there is no plasma inside the exhaust, and this concentration increased to about 18 ppm when applied voltage was 13.5 kV. This increase can be due to the reaction of NO_2 with produced N radicals during the plasma process by the following reaction scheme (Vinh et al. 2012):

$$NO_2 + N \rightarrow N_2O + O$$

The effect of non-thermal plasma on NO_x ($NO + NO_2$) emission reduction is shown in Fig. 10. The maximum of energy density is limited by applied voltage at 13.5 kV, the same as in previous experiments. By increasing the energy density, a continuous increase in NO_x removal efficiency has been observed. NO_x removal efficiency is not very high, while the energy density is changing up to 10 J/L. However, a significant increase in NO_x removal efficiency has been observed when energy density has been increased to 27 J/L. For the given configuration, the maximum NO_x

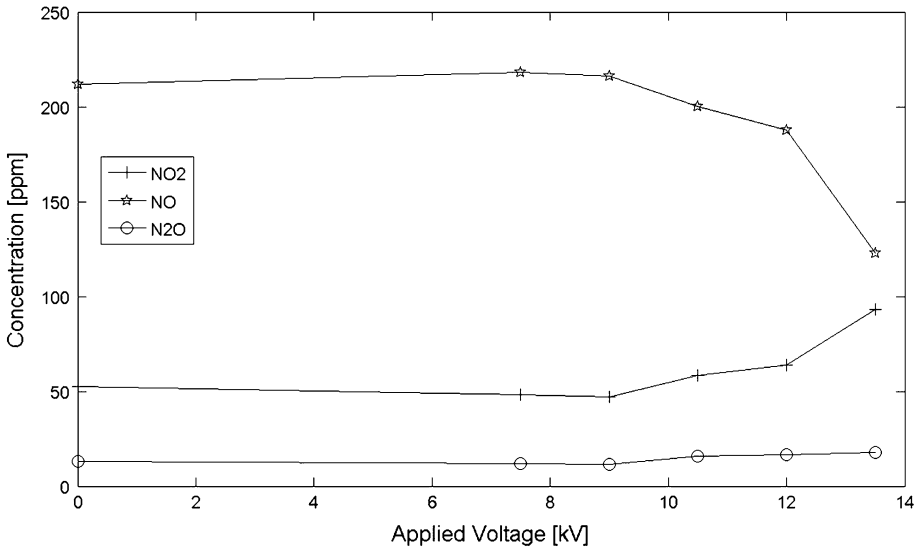

Fig. 9 Effect of non-thermal plasma on different nitrogen oxides

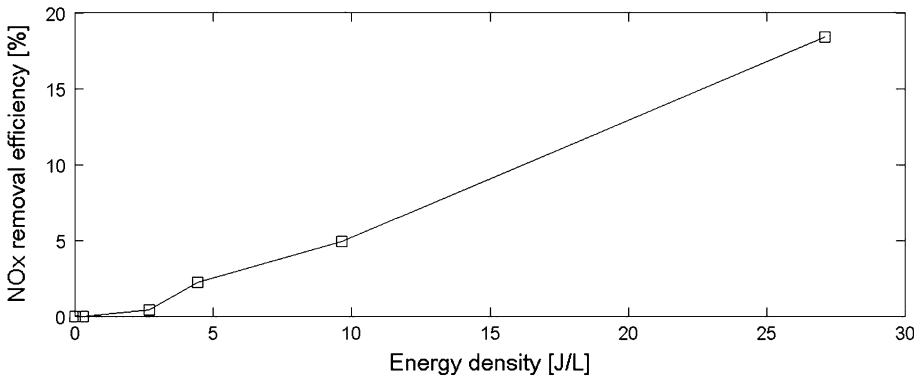

Fig. 10 NO_x removal efficiency at different energy densities

removal efficiency of about 18 % has been achieved when energy density was about 27 J/L. It should be noted that total NO_x reduction cannot be concluded from considering the effect of plasma on NO, NO_2 and N_2O only. Other nitrogen oxides such as NO_3 and N_2O_5 can be formed during the process, and they should be considered in future applications (Fitzsimmons et al. 1999).

Conclusion

In this study, the effects of NTP on PM structure and NO_x emission of real diesel exhaust at different voltage levels (which corresponds to different discharge powers) have been investigated. Nitrogen monoxide (NO) was found to be oxidised to nitrogen dioxide (NO_2) during the experiments. This result was in good agreement with previous studies in this field. NO_x removal efficiency increased by increasing the discharge power, with its maximum being about 18 %. Moreover, the effects of NPT on PM components and PM size distribution have been studied simultaneously to obtain a better insight into PM removal mechanism. NTP was found very effective for PM removal, especially for soot reduction. A maximum reduction of 73 and 37 % at energy density of 27 J/L has been found for soot and SOF mass concentration, respectively. The effect of NTP on PM size distribution has been studied by using SMPS data. Despite a considerable increase in nucleation mode particles at 13.5 kV, NTP showed a good potential for PM reduction in the range of 10–500 nm. Regarding the PM removal efficiency, energy consumption and PM size distribution on nucleation mode, running the DBD reactor at 12 kV can be considered as the optimum operating condition for the given configuration. Since the obtained results show a promising potential of NTP application for PM and NO_x reduction at room temperature, further study is required at higher temperatures which has more value being conducted for research and development.

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