Measurement of Electrical Charge on Diesel Particles

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Nanoparticles ($D_p < 50$ nm), which are formed as diesel engine exhaust cools and dilutes, constitute minority of total particle mass but majority of total particle number. There are several different theories to explain the nucleation of nanoparticles from diesel exhaust. The two main theories are homogeneous binary nucleation of sulfuric acid and water, and ion-induced nucleation. This study examined the ion-induced nucleation theory. In order to test the ionic nucleation theory, the charged fraction of the diesel particles were measured as a function of particle size using regular diesel fuel in this study. A very small amount of charge was found for the diesel nanoparticles in the nuclei mode, whereas there was a large charged fraction for the diesel particles in the accumulation mode. If ion-induced nucleation were the dominant mechanism for the nucleation of nanoparticles from diesel exhaust, one would expect a significant charge on the nuclei mode particles. The results from this study suggest that ion-induced binary nucleation is at least not a dominant mechanism for the nucleation of diesel exhaust when using regular diesel fuel.

This study also examined the influence of metal additives on nucleation and particle charging. The metal additives examined are of the type used to enhance particle oxidation in diesel particulate filters. When used, the additives led to a large increase in the concentration of solid particles in the nuclei mode, and significantly raised the level of particle charge for particles of all sizes. When additives were used, some of the solid particles in the nuclei mode carried a charge. We believe that these metal related particles form early enough in the combustion process to be charged by ions present during and shortly after combustion.

INTRODUCTION

With great emphasis being placed on the health effects and environmental impact of diesel particles, concerns about possible increases in the engine nanoparticle emissions have been growing (Dockery et al. 1993; Pope et al. 1995). Bagley et al. (1996) suggests that modern low mass emission engines might emit higher concentrations of ultrafine particles ($D_p < 100$ nm) and nanoparticles, as compared to older engine designs. Current regulations on the emissions of particulate matter (PM) from

engines are based on mass, which is measured by filter sampling of the PM. The contribution of nanoparticles to the total PM mass is very small, as shown in Figure 1 (Kittelson 1998). Recent studies (e.g., Brown et al. 2000) on the health effects of nanoparticles have led to an increased interest in other methods of measuring PM emissions, such as surface area or number, as well as an increased awareness of the importance of characterizing nanoparticle emissions.

This study was performed in order to gain a better understanding of the nucleation of diesel nanoparticles. Because of the highly complex and nonlinear characteristics of the nucleation process, the process is difficult to simulate in the lab and is thus poorly understood. In the absence of metal additives, most diesel nanoparticles ($D_p < 50$ nm) are formed during dilution through the gas-to-particle conversion processes, as the diesel exhaust cools and dilutes in the atmosphere (Abdul-Khalek et al. 1999; Baumgard and Johnson 1996; Shi and Harrison 1999). In contrast, carbonaceous diesel particles ($D_p > 50$ nm) are formed early in the engine cycle. Therefore, the formation of these carbonaceous particles is insensitive to the dilution condition (Abdul-Khalek et al. 1998, 1999; Kittelson 2001). Ziemann et al. (2002) found nuclei mode particles ($D_p < \sim 30$ nm) to be mainly composed of volatile organics coming from lubricating oil and/or unburned fuel. Sakurai et al. (2003) measured the volatility of particles in the nuclei mode. They found out that there are two kinds of PM in the nuclei mode, more volatile PM and less volatile PM. More volatile PM is mainly composed of volatile organics, whereas less volatile PM is composed of carbonaceous particles with volatile organics coated on the surface. Their study shows that, as particle size gets smaller, the fraction of more volatile PM increases in the nuclei mode. There are several theories to explain the nucleation of nanoparticles from diesel exhaust. Among those theories are the homogeneous binary nucleation theory and the ion-induced nucleation theory. Diesel nanoparticle formation through binary nucleation of sulfuric acid and water has been proposed (Abdul-Khalek et al. 1999; Baumgard and Johnson 1996; Shi and Harrison 1999). Abdul-Khalek et al. (1999) and Khalek et al. (2000) have suggested that this nucleation is followed by absorption or condensation of heavy hydrocarbons. Yu (2001) proposed ion-induced nucleation involving water and sulfuric acid in combination with ions. He attempted to explain the inconsistencies between predictions of the binary nucleation
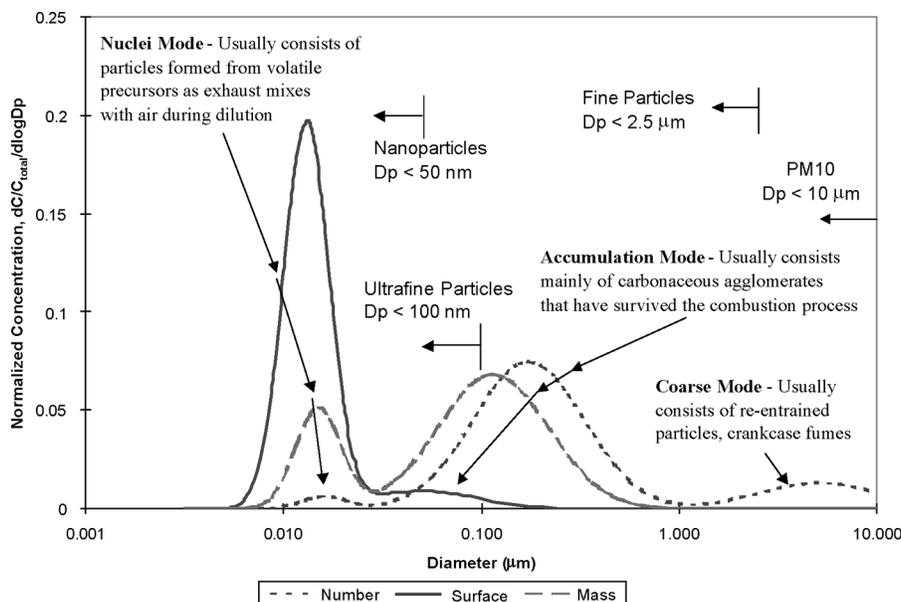
FIG. 1. Typical diesel particle size distributions weighted by number, surface area, and mass.

theory and the nuclei mode formation as observed by Abdul-Khalek et al. (1999) and Shi and Harrison (1999), and did so using the ion-induced nucleation theory. Under the ion-induced nucleation theory, it is believed that charged molecular clusters can grow significantly faster than corresponding neutral clusters. This leads to the clusters' ability to achieve stable, observable sizes (Yu and Turco 2000, 2001). If ionic nucleation is important, nuclei mode particles should have a significant charge.

For this study, charged fractions of diesel particles were measured as a function of particle size, in order to examine the possibility of ionic nucleation. This was done using both regular diesel fuel, and diesel fuel dosed with metal additives. The size of the nuclei mode present in the diesel exhaust increases in the presence of metal additives (Kittelson et al. 1978; Kytö et al. 2002; Matter and Siegmann 1997; Mayer et al. 2003). There is an increased interest in putting metal additives in diesel fuel, because doing so lowers the ignition temperature of particles collected in DPFs (Diesel Particulate Filters), and thus enhances regeneration of DPFs (Lahaye et al. 1996; Stanmore et al. 1999). Skillas et al. (2000) and Jung et al. (2003, 2005) found that the nuclei mode increases while the accumulation mode ($D_p > \sim50$ nm) decreases in the presence of metal additives.

EXPERIMENTAL

Diesel Fuel, Cerium Additive, and Lubrication Oil

The charged fractions of the diesel particles were investigated using various diesel fuel mixtures. Standard EPA No. 2 on-road diesel fuel containing 300–500 ppm sulfur was used as a base fuel. Rhodia Electronics and Catalysis provided the cerium ad-

ditive, the product name of which is EOLYS DPX-9. A dosing rate of 100 ppm (by weight) cerium in the fuel was used. Heavy duty diesel engine lubricating oil (SAE 15W-40) was used in this study as calcium containing fuel additive. This oil has calcium content of $\sim$2500 ppm by weight. Two percent (by volume) of the oil was mixed with the base fuel to provide $\sim$50 ppm of calcium (by weight) in the fuel mixture. The fuel was stirred completely, using a motor-driven stirrer after the additives were added to the base fuel. This was done to ensure that the fuel was well mixed.

Engine, Sampling, Dilution System, and Apparatus

The engine used in this study is a medium-duty, direct injection, 4 cylinder, 4 cycle, mid-90s turbocharged diesel engine (John Deere T04045TF250) of 4.5 L displacement, with a peak power output of 125 HP (93 kW) at 2400 RPM, and a peak torque output of 400 N · m at 1400 RPM. The output of the engine was coupled with a dynamometer for load control. The engine was operated at 1400 RPM under 10, 50, and 75% load (300 N·m). The air to fuel ratio measured was 27 for base regular fuel at 1400 RPM, under 75% load.

Figure 2 shows a schematic diagram of the overall experimental setup, including the engine bench, sampling system, dilution tunnel, Electro Static Filter (ESF) and Scanning Mobility Particle Sizer (SMPS). As shown in Figure 2, the exhaust was sampled 25 cm downstream of the turbocharger exit, and was diluted in a two-stage-air-ejector dilution tunnel similar to one described by Khalek et al. (1999). Samples for this study were taken after the second stage of dilution in order to prevent the saturation of the CPC (Condensation Particle Counter), which can be caused by high concentrations of the particles in the nuclei
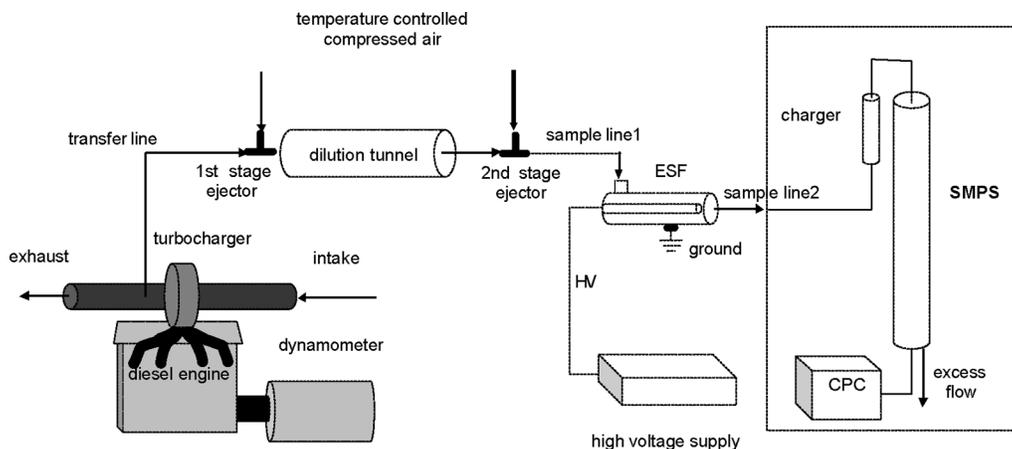
FIG. 2. Experimental setup.

mode. Residence times for the aerosol flow were estimated in Table 1. It took ~0.6 seconds from the exhaust sampling point to the end of dilution. The total residence time of the aerosol flow for the measurement, using the ESF, was ~16.5 seconds.

The dilution ratio for the first stage of dilution was measured as 16:1 for 10% load, and 23:1 for 75% load at 1400 RPM. This was done by comparing the NOx concentration in the exhaust gas and in the diluted exhaust gas after correcting for the background NOx concentration. The second stage was operated at a constant dilution ratio of 31:1. Thus, the overall dilution ratio after the second stage ranged from 500 to 710:1. The first stage dilution air temperature was controlled to maintain a temperature of $32°C$ at the exit from the first dilution stage.

The SMPS measures mobility equivalent particle diameter for the diameter range of 7 to 300 nm during a 4-minute (2 minutes up, 2 minutes down) scan time. This particle measurement system consists of a neutralizer, a mobility section (long column DMA similar to TSI model 3934), a CPC (TSI model 3010) and a computerized control and data acquisition system with a sampling flow rate of 1 LPM and a sheath flow rate of 10 LPM. This system is described in more detail by others (Abdul-Khalek et al. 1999; Khalek et al. 2000)

The ESF was used, in combination with the SMPS, to determine charged fraction as a function of particle diameter. The ESF is composed of an aluminum cylinder with a concentric rod at the center, as shown in Figure 2. Diluted sample aerosol sent to the ESF passes through the annular region of the ESF with a flow rate of 1 LPM. Charged particles were precipitated on the wall of the ESF when high voltage (7 kV) was applied between the center rod and the outer wall of the ESF.

RESULTS AND DISCUSSION

Size Distribution Measurements

Figure 3 shows number weighted size distributions of the diesel particles measured under 10, 50, and 75% engine loads at 1400 RPM, using regular diesel fuel. All number concentrations are corrected for the dilution ratio. The diesel particles at high engine load (75%) are mainly agglomerates for all size ranges, and are mostly found in the accumulation mode, whereas diesel particles at light engine load (10%) are composed of both nuclei mode and accumulation mode particles. While the accumulation mode forms during combustion by soot formation process, the nuclei mode forms as exhaust gas supersaturated during dilution process (Shi and Harrison 1999).

Figure 4 shows the number size distributions of the diesel particles for the base fuel and the fuel mixtures for the 1400 RPM, 75% load engine operating condition. The size distributions for

TABLE 1
Residence times of the aerosol flows

	Transfer line	Dilution tunnel	Sample line 1 and 2	ESF	SMPS
Residence time (s)	.0085	0.52	1.9	~12	~2[1]
Inner Diameter (cm)	0.64	N/A[3]	0.64	1, 3.7[2]	N/A
Length (cm)	5	N/A[3]	50	20	N/A
Flow (LPM)	11.3[4]	221	1.0	1.0	1.0

[1]The residence time was estimated using SMPS software from TSI. This is the time from the sampling inlet to the light detector in the CPC.
[2]ESF is in concentric shape. $D_{inner1} = 1$, $D_{inner2} = 3.7$.
[3]Detailed dimensions, measurement and calculation for residence time can be found in Ph.D. thesis by Abdul-Khalek (1999).
[4]Calculation is based on average primary dilution ratio of 19.5 using cold flow values.
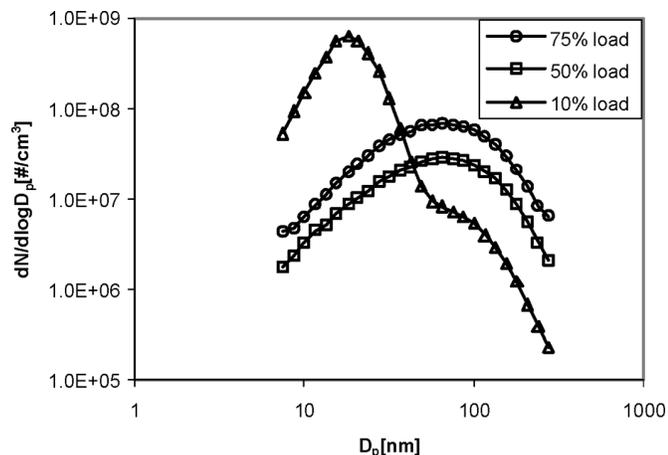
FIG. 3.   Size distributions at different engine loads when using regular diesel fuel under different engine loads at 1400 RPM.

the doped fuels show two noticeable changes compared to that of the base fuel. First, the concentration of the particles in the accumulation mode decreased significantly, presumably due to enhanced oxidation rates, and second, a large nuclei mode appeared. This is consistent with other recent work dealing with metal additives (Jung et al. 2003, 2005; Mayer et al. 2003; Skillas et al. 2000). The addition of metal additives to fuel increases the concentration of volatile metal compounds [see e.g., Abdul-Khalek et al. (1998)'s calculation on the formation of nuclei mode particles from Ca in the lubricating oil], which must undergo gas to particle conversion during the expansion stroke of the engine, while at the same time decreasing the accumulation mode, and thus the available surface area onto which gas phase material can condense or adsorb. This makes nucleation of metal compounds more likely.

Although the bimodal distributions for the light load condition using regular base fuel, as shown in Figure 3, and the heavy load condition using metal additive dosed fuel, as shown in Figure 4, look amazingly alike, there are significant
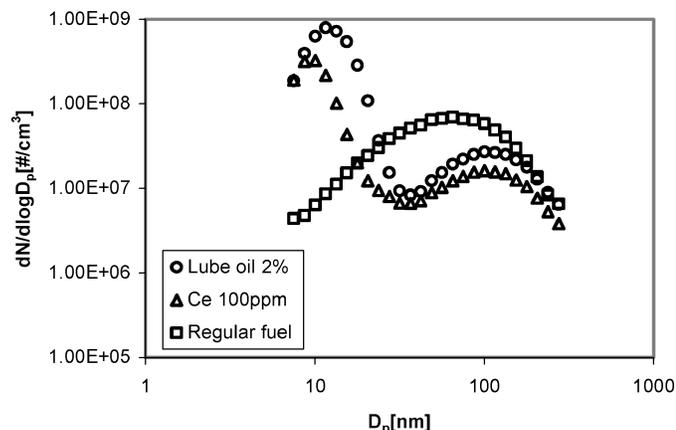
differences in their formation processes and composition. As described above, nuclei mode particles, at light engine load with regular diesel fuel, are formed during dilution and consist mainly of volatile organics. Other investigators have separated volatile materials from diesel particles by employing either a thermal denuder (Burtscher et al. 2001), an active carbon trap (Mayer et al. 1996) or a catalytic stripper (Abdul-Khalek and Kittelson 1995; Ng 2002). The thermal denuder and the active carbon trap remove the volatiles by evaporation/adsorption while the catalytic stripper removes volatiles by evaporation/oxidation. Undoped diesel particles from 10% engine load at 1400 RPM were sent to the catalytic stripper in previous studies (Abdul-Khalek and Kittelson 1995; Abdul-Khalek et al. 1998; Ng 2002; Stenitzer 2003). Most of the volatile organics and nuclei mode particles were completely removed, although Abdul-Khalek et al. (1998) found some residue (which was ash core) in the nuclei mode.

The nuclei mode particles that appeared under the heavy load with metal additive dosed fuel are formed as diesel exhaust cools down during the expansion process as illustrated by Kittelson et al. (2005). Skillas et al. (2000) collected nuclei mode particles on filters and performed material analysis with 100 ppm cerium dosed fuel. He discovered that nuclei mode particles are mainly composed of cerium compounds. For this study, we compared the size distributions before and after the catalytic stripper. Diesel particles with 100 ppm cerium dosed fuel were sent to the catalytic stripper operating at 300°C. Figure 5 shows that the nuclei mode particles with the 100 ppm cerium dosing rate are only partially removed by the catalytic stripper. This removal is mainly due to diffusion in the nuclei mode size range. After the correction for losses, the size distributions with and without the catalytic stripper are essentially the same, indicating that both

FIG. 4.   Size distributions using metal dosed fuel compared to using regular fuel under 75% load at 1400 RPM.
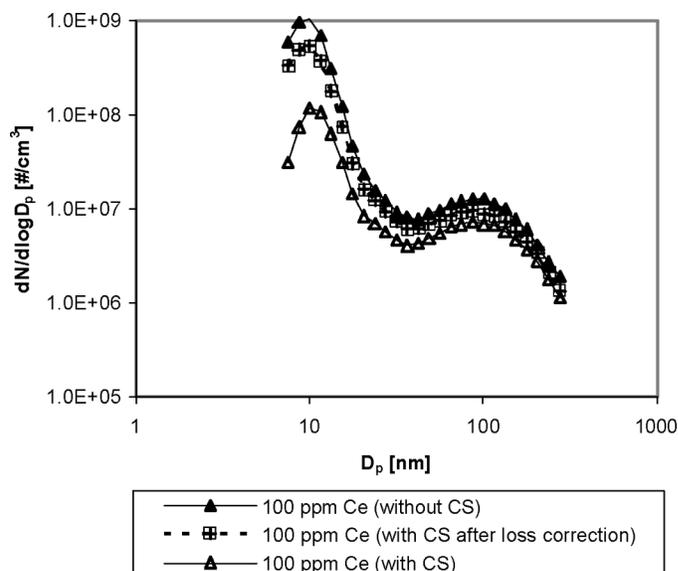
FIG. 5.   Size distributions with and without the catalytic stripper using 100 ppm ceria dosed fuel under 75% load at 1400 RPM.

nuclei and accumulation mode particles are mainly solid (Jung et al. 2005).

Measurement of the Electrical Charge on the Diesel Particles

In order to determine the charged fraction of diesel particles, number size distributions were measured consecutively with the ESF turned on and off. Figures 6a, b, and c, compare number size distributions with the ESF turned on and off. Figures 6a and b show results for standard fuel at 1400 RPM, 10 and 75% load, respectively, while Figure 6c shows the results for the doped fuels at 1400 rpm, 75% load.

The charged fraction is defined as 1-R, where R is the ratio of the concentration of particles of a given size with the ESF on to that with the ESF off. Charged fractions measured here as well as results from a previous study (Moon 1984) and calculated Boltzmann equilibrium charge distribution (Hinds 1982) at different temperatures are plotted against particle size in Figure 7. Particles produced at the heavy load (75% load, 1400 RPM) have higher charged fractions than those produced at the light load (10% load, 1400 RPM). The charged fractions for the heavy load condition show a trend similar to Moon's (1984) data, which were obtained using an older diesel engine at 90% load, 1200 RPM. The particles produced at 75% load, which are mainly composed of solid carbonaceous material, showed ~60 to 80% charged fraction over the size range. The accumulation mode particles produced under 10% load had lower, but still significant, charged fractions of 45 to 70%. The metallic additives led to an increase in charged fraction throughout the size ranges, but especially in the accumulation mode region.

Above about 150 nm the measured charged fractions for the regular fuel at 10 and 75% load drop slightly. This is likely due to incomplete removal of larger, less mobile particles by the ESF. Under given conditions (geometry and voltage), and assuming the flow is a turbulent plug flow in the ESF, the penetration efficiencies for the ESF were calculated as 1 and 12% for singly charged particles 170 and 300 nm in diameter, respectively. This agrees qualitatively with a falloff of measured charged fractions observed for the regular fuel case. The falloff of the charged fraction was not observed for the metal dosed fuel, presumably due to increased charges per particle, resulting in more effective precipitation under the given condition.

Nuclei mode particles under 10% load ($D_p < ~30$ nm) showed a very small charged fraction. The nuclei mode particle size distributions using regular fuel are very sensitive to the engine operating parameters. This causes greater uncertainty in the size distribution measurement, as shown in Figure 6b, compared to that of the accumulation mode, as shown in Figure 6a. The charged fraction fell sharply below 40 nm, lower than that of the Boltzmann distribution at room temperature. This sharp drop occurs as volatile, freshly nucleated, nucleation mode particles become much more prevalent than accumulation mode particles.

(a)

(b)

(c)
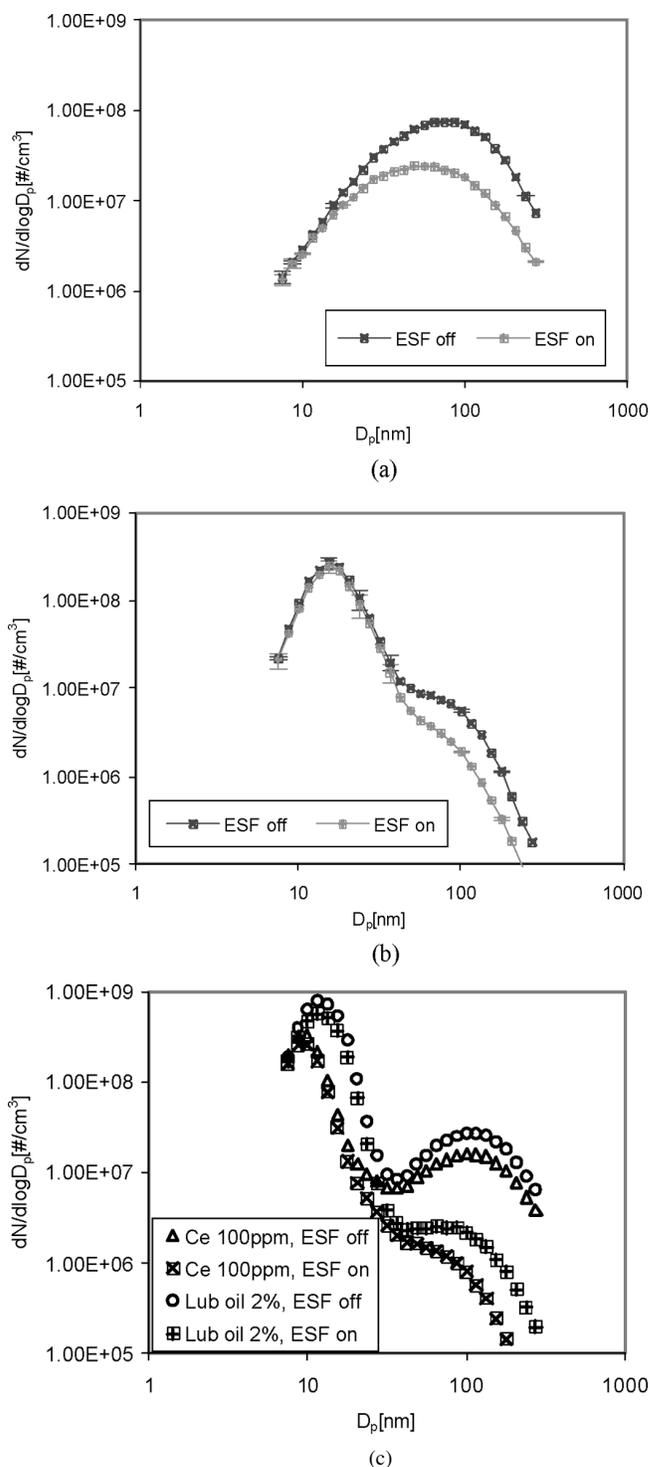
FIG. 6. Size distributions with the ESF on and off. (a) 75% load at 1400 RPM using regular fuel. (b) 10% load at 1400 RPM. (c) 75% load using metal dosed fuel.

DISCUSSION

The nuclei mode particles at the 10% engine load were formed during the dilution process, with regular base fuel being used. If ion-induced nucleation were the dominant mechanism for the
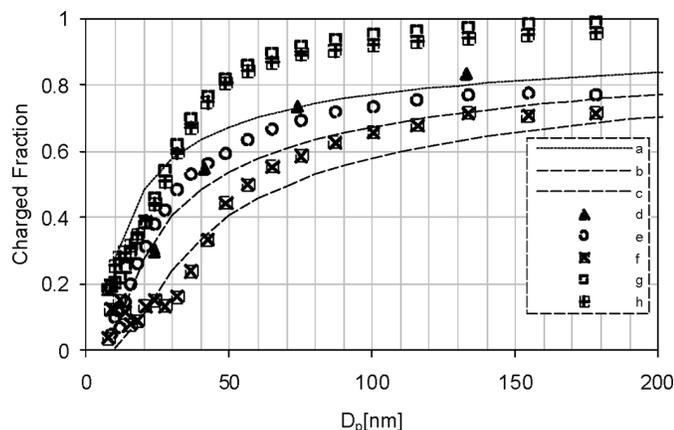
FIG. 7. Charged fraction as a function of diameter for various fuels in comparison with prior study. (a) Boltzmann equilibrium charge distribution at 1000 K, (b) Boltzmann equilibrium charge distribution at 500 K, (c) Boltzmann equilibrium charge distribution at 300 K, (d) Regular fuel at 90% load, 1200 RPM by Moon (1984), (e) Regular fuel at 75% load, 1400 RPM, (f) Regular fuel 10% load at 1400 RPM, (g) 100 ppm Ce doped fuel at 75% load, 1400 RPM, (h) 2% lube oil doped fuel at 75% load, 1400 RPM. Note: (e), (f), (g), and (h) are results of this current study.

nucleation of nanoparticles from diesel exhaust, one would expect a significant charge on the nuclei mode particles. The results of this study suggest that ion-induced binary nucleation is at least not a dominant mechanism for the nucleation of diesel exhaust when using regular diesel fuel.

In related work, nuclei mode size distributions were measured with an ion trap capable of removing all ions placed in the sample stream immediately before dilution (Ma et al. 2005). Removing upstream ions had no significant influence on the nucleation mode suggesting that ionic nucleation did not play an important role (Ma et al. 2005) also calculated ion concentration histories, accounting for recombination and attachment during expansion stroke in diesel combustion. These calculations indicate ion concentrations too low to account for nucleation mode formation. Futhermore (Ma et al. 2005) estimated the time scale for neutralization of charged nuclei and found it too slow compared to the residence time in our system to account for the absence of charge observed here.

On the other hand, a homogeneous binary nucleation model of H_2SO_4-H_2O showed qualitatively good agreement with measurements (Abdul-Khalek et al. 1999, Shi and Harrison 1999); however, that model predicts much lower nucleation rates than measurement (Shi and Harrison 1999). Khalek et al. (2000) and Shi and Harrison (1999) suggested that hydrocarbon or some trace gas found in diesel exhaust may play a role in causing higher than predicted nucleation rates.

The higher charge levels observed in the presence of metallic additives is likely due to the formation of metal ions during combustion. Collings et al. (1988) suggested that adding metals to diesel fuel can increase the exhaust ion concentration, either by slower recombination coefficients of metal ions, or by pro-

longing the overall combustion. The addition of metals also may increase the peak ion concentrations during combustion. Higher ion concentrations would result in higher charged fractions on the particles, especially since the metals suppress the formation of the accumulation mode particles that provide surface for ion removal by attachment

It can be seen from Figure 7 that even nuclei mode particles carry some charge when metal additives are used. We do not believe that the charge is due to ionic nucleation. The charged fraction observed was small, only about 20% at 10 nm and was likely acquired in the same way as for accumulation mode particles, since these metallic nuclei are formed relatively early in the expansion process (Abdul-Khalek et al. 1998) when ion concentrations are still high (Collings et al. 1988, Moon 1984)

The higher charge levels found in the presence of metal additives might have a practical application. Attempts have been made (Thimsen et al. 1990, Wadenpohl and Löffler 1994) to use the natural charge on diesel particles to allow their collection in low power electrostatic precipitators without a corona charger. One of the problems encountered in this work was large size and low collection efficiency. It is possible that the higher charge levels associated with metallic additives might make such a device more feasible, although it would not work well for the smallest particles.

CONCLUSIONS

Charges on the diesel particles were measured with and without metal additives in the fuel. A very small amount of charge was found for the diesel nanoparticles in the nuclei mode, whereas 60 to 80% of the particles in the accumulation mode were charged when using regular diesel fuel. The lack of charge on the particles in the nucleation mode suggests ion-induced nucleation is at least not a dominant mechanism in the formation of nuclei mode particles during exhaust dilution when using regular diesel fuel.

Metal additives increase the charged fraction of diesel particles, especially for the accumulation mode particles. This increase in charged fraction is likely associated with the formation high concentrations of relatively stable metallic ions that attach a reduced concentration of accumulation mode particles.

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