

An Aerosolization Method for Characterizing Particle Contaminants in Diesel Fuel

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ABSTRACT

Diesel fuel injection systems are operating at increasingly higher pressure (up to 250 MPa) with smaller clearances, making them more sensitive to diesel fuel contaminants. Most liquid particle counters have difficulty detecting particles $<4\text{ }\mu\text{m}$ in diameter and are unable to distinguish between solid and semi-solid materials. The low conductivity of diesel fuel limits the use of the Coulter counter. This raises the need for a new method to characterize small ($<4\text{ }\mu\text{m}$) fuel contaminants. We propose and evaluate an aerosolization method for characterizing solid particulate matter in diesel fuel that can detect particles as small as $0.5\text{ }\mu\text{m}$. The particle sizing and concentration performance of the method were calibrated and validated by the use of seed particles added to filtered diesel fuel. A size dependent correction method was developed to account for the preferential atomization and subsequent aerosol conditioning processes to obtain the liquid-borne particle concentration. Finally the method was used to evaluate a diesel fuel sample after filtration through a laboratory grade filter to demonstrate the practical application of the method.

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INTRODUCTION

Diesel engines offer better fuel economy than gasoline engines mainly due to higher compression ratio and reduction of air pumping loss [1]. More stringent emission limits in all major diesel markets in the world [2, 3] have resulted in manufacturers implementing major changes to diesel engines, especially the fuel systems, to reduce emissions and improve performance [4, 5]. High pressure common rail (HPCR) fuel injection systems enable higher injection pressure and more precise injection timing control, which leads to better engine efficiency and lower emissions. Today, the operating pressure of HPCR systems is up to 250MPa, more than four times the pressure of high pressure hydraulic systems [6]. Clearances between moving parts are often smaller than $5\text{ }\mu\text{m}$ [6]. The high pressure and reduced clearances make the fuel system more sensitive to contaminants.

Particulate matter contaminants found in diesel fuel are divided into two major groups based on volatility: semi-solid and solid particles. Semi-solid particles consist mainly of asphaltene and microorganisms, which come from the refining process and storage [7]. Semi-solid particles plug fuel filters, thus reducing the service life [8], but are not as harmful to the fuel injection systems due to their deformability. Solid particles found in diesel fuel are typically composed of inorganic material such as SiO_2 , Al_2O_3 and Fe_2O_3 [9], which are abrasive and detrimental to the fuel injection system. A major functional impact of the solid contaminants is larger injector leakage, which increases the backflow, sometimes as high as 400% more than the initial value [9]. This will make the high pressure pump unable to deliver sufficient fuel to ensure the required rail pressure and in extreme cases could lead to a system shut-down command from the engine control unit. Excessive fuel injection leakage

also increases engine out particulate matter emissions [9]. These issues have raised the awareness of fuel quality and the need to update fuel quality standards.

There are several concerns with current diesel fuel particle contaminant specifications. They are mostly mass based. This allows a large number of small size, low mass particles to enter fuel injection system [9]. Current specifications don't specifically set limits for contaminants $<4\text{ }\mu\text{m}$, because traditionally these particles are not considered to be harmful in hydraulic power systems [10, 11, 12]. However, with increasingly higher pressure of fuel injection systems there is emerging demand to regulate fuel contaminants $<4\text{ }\mu\text{m}$ due to their relatively higher concentrations [4, 13]. Finally, fuel injection system manufacturers express the need for specific specifications that limit the allowable concentration at the outlet of the distribution pump fuel filter rather than simply specifying a filtration efficiency because of the large variation in pump fuel quality [9]. Moreover, it has been reported that the peak particle size distribution becomes smaller after filtration [9], which requires that the concentration of $<4\text{ }\mu\text{m}$ contaminants be determined after filtration. These concerns raise the need for a particle characterization method in diesel fuel that can provide a high resolution size distribution covering a wide range of sizes, especially for particles smaller than $4\text{ }\mu\text{m}$.

Common ways to measure liquid-borne particle concentration include the gravimetric method, optical and electron microscopy, electric zone sensing, and various radiation scattering/absorption systems [14]. The gravimetric method uses a filter to collect the particles and a balance to measure mass [15]. This method is less accurate when dealing with clean liquids containing mostly small particles [16]. The microscopic method is time-consuming and not suitable for routine filter testing [17]. The extensive sample preparation, removal of liquid phase and high vacuum needed may distort the particles of interest [14]. Electric zone sensing (e.g. Coulter counter) measures the electric resistivity and is not applicable to non-conductive liquids like diesel fuel [18]. Radiation scattering (e.g. light scattering) is one of the mostly used methods for characterizing liquid-borne particles [19, 20, 21], especially for diesel fuel contaminants [11]. However, the performance of the light scattering method becomes poor for particles smaller than $4\text{ }\mu\text{m}$ in diesel fuel due to its opacity. According to Dombrovsky [41], the index of absorption of diesel fuel for 500 nm wavelength of light is around 1×10^{-6} which is about 1000 times higher than the value of water (around 1×10^{-9} [42]). The relatively high refractive index of diesel fuel [41] also limits the amount of light scattered by contaminants needed to distinguish them from the diesel background. Thus, there is a lack of a method that enables rapid measurement of particle concentration for particles smaller than $4\text{ }\mu\text{m}$.

Airborne particle characterization methods for submicron particles are well developed, and able to detect and characterize particles $<4\text{ }\mu\text{m}$ in size. The objective of this

study is to develop and characterize a new aerosolization method for characterizing solid particles in diesel fuel $<4\text{ }\mu\text{m}$ in size. The size distribution and total concentration of solid particle contaminants ranging in size from 0.5 to $4\text{ }\mu\text{m}$ are determined in doped and undoped diesel fuel. The technique is applied to evaluate a laboratory grade filter to demonstrate the practical application of the technique.

METHODOLOGY

Many devices, e.g., compressed air nebulizers, ultrasonic nebulizers, spinning disc and electrosprays, have been developed to aerosolize liquid suspensions [23]. Commercial electrospray generators are generally limited to producing particles $<0.15\text{ }\mu\text{m}$. Commercial spinning disc aerosol generators usually produce droplets between 20 and $30\text{ }\mu\text{m}$, but are not suitable for generation of particles $<3.5\text{ }\mu\text{m}$ [24]. Compressed air atomizers are able to provide droplets ranging in size from 1 to $6\text{ }\mu\text{m}$ and are widely used in biological and clinical application as well as aerosol research [25, 26, 27]. By atomizing diesel fuel the background for the particle contaminant is changed from the opaque diesel fuel to transparent air, thus enabling the use of aerosol instruments, which may offer greater sensitivity than a liquid particle counter. After evaporation of the fuel droplets and organic material, only solid, non-volatile particle contaminants survive to be detected by the aerosol instruments. The measured aerosol size distributions are used to calculate the liquid-borne values by applying a proposed conversion factor that is discussed later.

Experimental Apparatus

Figure 1 shows the apparatus used for the aerosolization method to detect particle contaminants in diesel fuel.

A syringe pump (Cole-Palmer Model 7802200, Vernon Hills, IL) is used with a constant output atomizer (TSI Model 3076, Shoreview, MN) to atomize the liquid diesel fuel. The liquid sample is forced into the atomizer by the syringe pump. This non-recirculating configuration generates a more stable aerosol output than the recirculating configuration, in which a continuous increase in the solute concentration and shift in the size of the atomized aerosol is observed because of evaporation of the solvent [28]. To achieve a stable output concentration for the particles in the size range of interest (0.5 to $4.0\text{ }\mu\text{m}$), the inlet pressure of the atomizer is set to 69 kPa. The liquid feed rate is set as 60 ml/hr. The jet of air breaks up the filament of liquid to form droplets of various sizes. More than 90% of the total droplets at this point will have sufficient momentum to impact the wall of the atomizer and go down to the exhaust liquid bottle. The smaller droplets containing contaminants are transported by the air flow to the outlet of the atomizer and exit as an aerosol. Figure 2 shows the droplet size distribution (volume weighted) measured at the outlet of the atomizer by an aerodynamic particle sizer (APS, Model 3321, TSI Inc., Shoreview, MN). The aerodynamic particle sizer sizes particles from 0.5 to $20\text{ }\mu\text{m}$

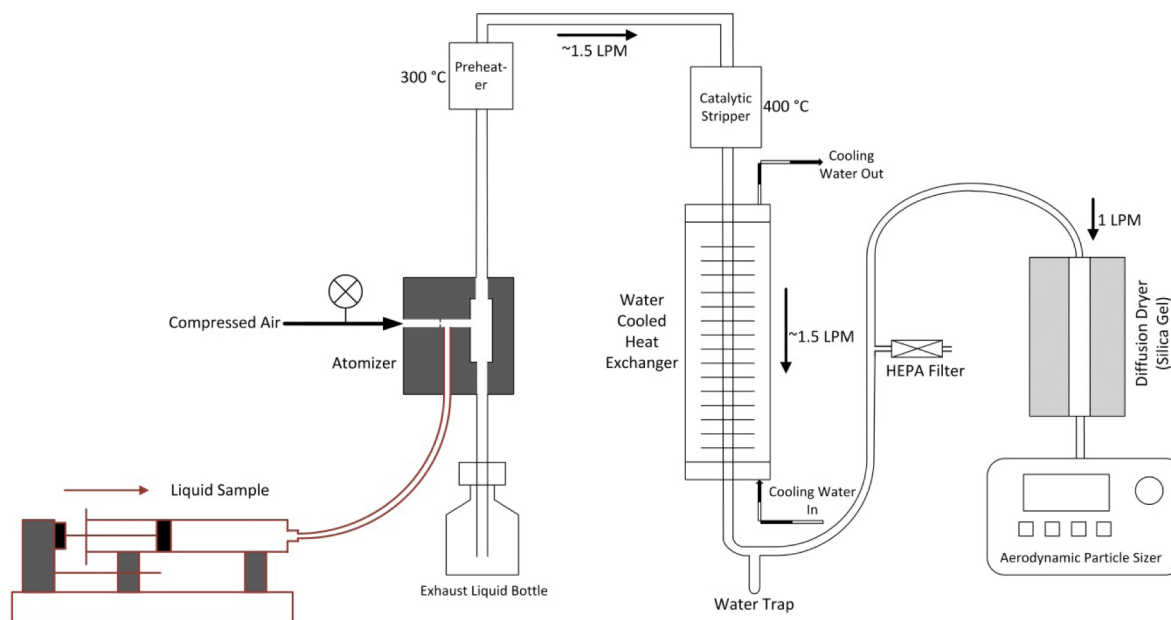


Figure 1. Schematic diagram of the system

using a time-of-flight technique that measures the velocity of particles in an accelerating air flow through a nozzle and converts that to the particles' aerodynamic diameter in real time [32]. The technique is unaffected by index of refraction or Mie scattering property of the particle.

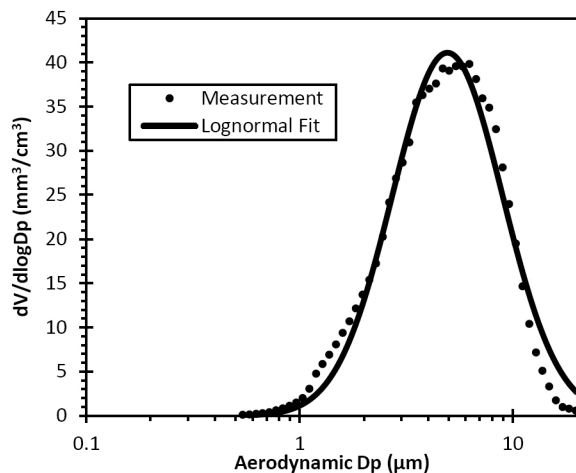


Figure 2. Fuel droplet size distribution (volume weighted) atomized by constant output atomizer

The diesel fuel aerosol contains a large amount of fuel droplets that must be removed before the solid particle contaminants are quantified. A catalytic stripper, described in detail elsewhere [29, 30, 31], is used to remove volatile and semi-solid material. A preheater is placed before the catalytic stripper to preheat the aerosolized fuel sample to around 200 °C to facilitate the evaporation of the droplets in the catalytic stripper that is operated at a flow rate of 1 L/min and a temperature of 400 °C to ensure the complete evaporation and oxidation of the organic material. Stenitzer [31]

estimated the particle penetration as a function of size and reported that there is a trade-off between particle loss and hydrocarbon removal efficiency in the catalytic stripper. In the current design, hydrocarbon removal efficiency is high and as a result, the loss of nanometer-sized particles is high. However, for the size of interest in this study (0.5 to 4 μm), the loss is relatively independent of size. This is important to preserve the relative shape of the size distribution of the solid particle contaminants.

Since the temperature of the aerosol exiting the catalytic stripper is about 400 °C, a water cooled heat exchanger is used to cool the sample prior to entering the aerodynamic particle sizer. There is thermophoretic loss during the cooling process but the loss is relatively small (~25%) and independent of particle size which will be discussed in more detail in the results section and shown in Figure 7. During cooling a significant amount of water condenses to droplets, which are collected by a water trap placed under the heat exchanger. To remove the water condensed onto the particulate contaminants and reduce the vapor content in the aerosol, a silica gel diffusion dryer is placed after the water trap.

To validate the proposed method a highly filtered diesel fuel sample was spiked with solid particles of known size and concentration. Ultra-low-sulfur diesel fuel containing no biodiesel was first filtered using a cascade filtration system with 3 filters with pore sizes of 1 μm, to 0.45 μm, and 0.2 μm. The fuel was then seeded with silica particles to simulate contaminants that are known to adversely impact fuel filters and fuel injectors [33, 34]. In fact, silica itself is a major component of solid diesel fuel contaminants, along with Al₂O₃ and Fe₂O₃. Three sizes (0.73, 1.80 and 3.60 μm) of monodisperse silica particles were used to cover the size