SOOT PARTICLE MEASUREMENT IN ENGINE CYLINDER: A REVIEW

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Abstract

This review article describes a list of most common techniques of soot measurement, particularly for in-cylinder diesel engine soot. The techniques presented in this paper are Laser Induced Incandescence (LII), Light/Laser Extinction Measurement (LEM), Light/Laser Scattering Measurement (LSM), Emission Tomographic (ET) and Thermophoretic Sampling. All techniques are briefly elaborated and their principles of operation, as well as their applications and current issues, are covered. It is commonly acknowledged that non-invasive techniques feature some advantages and are more preferable compared to invasive techniques. Besides, more modern technique such as ET is said to be most widely applied in the future due to its high level of accuracy. However, there are possibilities that both invasive and non-invasive methods can complement each other to obtain more accurate soot measurements.

Keywords: Soot, in-cylinder, soot formation, diesel engine, engine emission

Graphical abstract

1.0 INTRODUCTION

In the context of diesel engine emission, soot can be defined as small spherical carbonaceous particles that formed in a fuel-rich region of incomplete fuel combustion. This solid phase element which nucleates from the vapour phase, is one of the compounds of diesel particulate matters (PM). Diesel PM consists of three main components; carbonaceous solid (known as soot), heavy hydrocarbon condensed or absorbed...
on the soot, known as a soluble organic fraction (SOF) and sulfates (SO₄) as can be visualized in Figure 1.

![Figure 1 Components in the diesel particulate matter](image.png)

**Figure 1** Components in the diesel particulate matter [1]

The adverse impact of soot and another PM components to human life is not a new story and has already been known decades ago. Continuous efforts by various governments or corporate institutions have been exercised to overcome the problems. Some of the pioneer countries in emission control enforcement such as the USA, Europe, Japan and China have become role models for other developed countries to emulate [2]. South East Asian countries, for example, have taken appropriate actions to introduce stricter emission regulations in their countries as summarized in Table 1 (example given is for light duty diesel powered vehicle or passenger car, GVW ≤ 2500 kg).

Impacts of soot to the environment, human health and engine performance are undeniable and well-proven by numerous researchers. These unwanted particles that are emitted into the atmosphere will cause global warming, acid rain, smog, odors and other health hazards [3-4]. Numerous studies also successfully show that these particles contribute to the toxicity for human health [5-8]. Moreover, these particles will also reduce the overall engine performance by decreasing the mean surface temperature [9], damaging the metal surface [10] and increasing the influence to engine wear [11-12].

Recognizing those critical impacts, various efforts to reduce soot emissions have been continuously carried out by researchers. Even a tiny improvement in emission control method will give a great contribution to the pollution and economy [13-14]. Some researchers recommended to develop cleaner diesel engine by some modification on engine hardware such as Diesel Particulate Filter (DPF) and NOx reduction system [15] and to reduce soot by using microwave heating [16]. Wei and Na [17] in their review, summarized that the emission control techniques include the improvement of diesel fuel, implementing supercharger and intercooler, using four-valve techniques, improve spraying method, using exhaust gas recirculation (EGR) and also improving the post-treatment of exhaust emission.

To achieve successful emission control regulations, governments should view the problem in a more holistic perspective whereby all related agencies should work together in developing more eco-friendly vehicles. From an academic point of view, soot emission could possibly be eliminated from its source, which is inside the cylinder, hence making its size and distribution in engine cylinders as key parameters to be further investigated. This article will discuss in general some of the common techniques available for measuring soot, particularly soot that is formed in internal combustion engine cylinder.

<table>
<thead>
<tr>
<th>Country</th>
<th>Current Standard Implemented</th>
<th>Planned Standard to be Enforced</th>
<th>Date to be implemented</th>
</tr>
</thead>
<tbody>
<tr>
<td>Malaysia</td>
<td>Euro 2</td>
<td>Euro 4</td>
<td>January 2016</td>
</tr>
<tr>
<td>Singapore</td>
<td>Euro 5</td>
<td>Euro 5</td>
<td>January 2014</td>
</tr>
<tr>
<td>Thailand</td>
<td>Euro 4</td>
<td>No information</td>
<td>No information</td>
</tr>
<tr>
<td>Indonesia</td>
<td>Euro 2</td>
<td>Euro 4</td>
<td>No information</td>
</tr>
<tr>
<td>Brunei</td>
<td>Euro 1</td>
<td>Euro 4</td>
<td>2016</td>
</tr>
<tr>
<td>Vietnam</td>
<td>Euro 2</td>
<td>Euro 4 and Euro 5</td>
<td>2017 and 2022</td>
</tr>
<tr>
<td>Philippine</td>
<td>Euro 2</td>
<td>Euro 4</td>
<td>January 2016</td>
</tr>
</tbody>
</table>
2.0 SOOT FORMATION AND PROPERTIES

Soot particle size and distribution investigation can contribute to the overall measures of reducing the environmental and health problems. To achieve that, soot characteristics and their properties during engine operation need to be explored [20]. To understand the process, the conceptual model of spray combustion integrated with chemical kinetics is shown in Figure 2. The image indicates the region where pre-combustion, soot and NOx formation takes place.

Initial soot formation started with pyrolysis in high-temperature condition [21] before it grows to be a poly-aromatic hydrocarbon (PAH) [22]. Larger PAH molecules formed by reactions of PAH-PAH grow into precursors for soot nucleation [23]. Then, nucleation or inception process from gas-phase species condensation will form bigger three-dimensional soot particles [22-24]. Concurrently during the end of nucleation, there will be surface growth process occurs. Then, the bigger particles during the previous process will collide and combine to form a single spherically shaped particle during coalescence (or coagulation) stage, thus reducing the particle numbers. It is followed by agglomeration process in which the particles settled together to form bigger clusters of primary particles [21]. By taking into consideration of soot oxidation, it will take places at anytime during the whole soot formation process when the temperature is above 1300 K, where the carbon or hydrocarbons are converted to combustion products. As a conclusion, understanding the whole soot process is important to reflect the most suitable measurement techniques that should be applied.

3.0 SOOT MEASUREMENT TECHNIQUES

Since decades ago, numerous experimental setups have been developed to measure soot parameters such as mass distribution, particle number, surface concentration, volume fraction, density and particle size. Each system introduced different measurement principles which will lead to different outcomes. Some of the common experimental techniques will be discussed in this subsection.

With recent technologies in aerosol measurement, it is possible to have more exact and reliable data [26]. However requirement to keep improvising the current system will always arise [1]. Even though typical experimental setup in the laboratory will be highly preferred [27], present trends show that portable emission measurement systems (PEMS) is capable of taking over the measurement function especially for in-situ and real driving condition measurements [28].

There are various types of measurement techniques proposed by researchers and manufacturers, particularly for the in-cylinder arrangement. Chun et al. [29] categorized the measurement techniques into invasive and non-invasive measurement techniques. While Giechaskiel et al. [30] arranged the techniques by more detailed categories which were based on different methods (i.e. filtering, chemical analysis, optical, microbalance, charging and combination of size distribution). This article will describe various systems or methods used to measure particularly in-cylinder soot particles properties.

3.1 Laser Induced Incandescence (LII)

LII is one of the most popular methods used to measure the soot particles since significant numbers of research articles were found reporting the use of this method. The energetic laser beam is used to radiate the soot particles so that particular soot are heated up through absorption of the laser pulse to very high temperatures (~4000 K). The increasing temperature of the soot particles will emit increased levels of a black body (Planck) radiation, called incandescence. Signal intensity of the incandescence is proportional to the soot volume fraction (SVF), and can be measured by a proper calibration. After being heated up by the laser, the cooling behavior of the particles will reflect the particle size distribution (PSD) with respect to the spectral characteristics and intensity of their blackbody radiation – smaller particles cool faster than larger particles due to the larger surface/volume ratio. Therefore, time-resolved LII (TIRE-LII) can be applied to calculate the averaged particle diameters in the measurement volume [29]. Figure 3 shows the common arrangement of the LII system.

Previous researchers reported that LII is one of the best tools in characterizing the primary particle size, volume fraction and any other characteristics of soot particles [31–34]. Some comparison with recent measurement techniques e.g. TEM also shows a good agreement in defining soot particle process [35] and
as a calibration-independent technique for soot measurement [36]. Other researchers are interested in using LII technique to verify the effect of fuel injection i.e. pressure, timings and injection styles (single or multiple) to the soot measurement [37–39].

However, a number of unresolved challenges mostly for particle sizing using time-resolved LII that have been drawn recently, certainly need special attention. All the parameters which significantly affect the analysis of the results, namely excitation and detection wavelengths, laser radiation and spatial beam profile, temporal detection issues and calibration methods need to be further improved [40–42].

While previous works by Seifert and Desjardins [48], and also by Yunkers [49] successfully proved that this method can also be used in determining the weight concentration of soot in used diesel engine oil without special reference oils or standards to perform the analyses [48–49]. However Karatas and Gülder [50] reported that this method requires radially resolved soot volume fraction when soot is investigated in a case of elevated pressures, so it is hardly used in soot studies [50].

3.3 Light/Laser Scattering Measurement (LSM)

Dissimilar to the LII and LEM method, the LSM method applies laser beam from the light source that will be diffracted by a lens so that the light scatters through a dispersed particulate sample. The angular variation in the intensity of the scattered beam will then be measured. The principle is, by relative to the laser beam, large particles will scatter the light at small angles while small particles will scatter the light at large angles.

By using the Mie theory of light scattering, angular scattering intensity data that create the scattering pattern is then analyzed to calculate the size of the particles. The particle size is reported as a volume equivalent sphere diameter. This method also can be combined with LEM [34] and LII [35] to measure soot particle size but with much more complexity. Figure 5 below shows the common arrangement and how LSM system works.

Figure 3 Laser Induced Incandescence [31]

3.2 Light/Laser Extinction Measurement (LEM)

This measurement system needs a light source, such as laser, arc lamp or a set of spectroscope and photodiodes [43]. But the laser is most commonly used due to its advantages of producing monochromatic light with high brightness, excellent collimation and a high degree of linear polarizability [34]. Figure 4 shows the common arrangement of the LEM system. Firstly, a beam of light will be passed through a beam expander, a set of oscillating diffusers, condenser lens and a collimator lens before it encounters the soot-laden flame. The extinction occurs as a sum of absorption and scattering effect [44]. Then, the light passes through a relay lens, band pass filter and neutral density filters. Lastly, the light is recorded by a CCD camera. Each pixel associated with a corresponding digital image records the intensity of the particles.

Research work done by Arana et al. [45] and Tree and Dec [46] were successfully applied light extinction measurement to measure soot volume fraction distributions in laminar flames, but this technique has the drawback of measuring a line-of-sight average that is not suitable for turbulent flame studies [47].
3.4 Thermophoretic Sampling Technique

Thermophoretic sampling technique adopts thermophoresis principle for collecting soot particle in flames. Principally, the thermophoretic mass transfer will cause the small particles to travel from the higher to the lower temperature region because of the temperature gradient in flames. Via this technique, a very thin in-thickness probe is inserted into the hot flame to capture a soot sample by thermophoretically deposition onto the grid. The probe exposure time should be long enough to capture a significant soot sample but short enough to present a cold surface to the flame-born particles. This cold surface will freeze various reactions of the particles that are already captured to prevent changes in the soot morphology after the particles have impacted on the grid. The soot samples collected on the grid are then inspected by electron microscopy either by Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM). Figure 6 shows the arrangement of the system that is commonly used.

![Figure 6 Thermophoretic Sampling Technique](image)

Earlier research by Eisner and Rosner [53], and also by Köylü et al. [54] successfully measured the concentration and morphology of each type of soot separately by using a thermocouple. While recent research works done by few researchers demonstrates the potential of implementing this method in particle size measurement as it shows an increasing trend in research report number. Kook et al. [55] studied the morphology of soot particles within reacting diesel jet to prove that the soot particles appear not to be sensitive to the exposure duration of the grid to the sooting flame. While Yamaguchi et al. [56] explored the behavior of soot clouds around different axial location, either with or without sampler. Pressure histories and heat release rates of diesel combustion with and without the sampler did not show significant differences. Zhang et al. [57] studied the comparison between wall deposited soot versus in-flame soot characteristics and they found that amount of soot particles deposited on the wall were far less than those soot collected inside the flame. Merchan-Merchan et al. [58] who conducted the research to compare between diesel and biodiesel found that biodiesels have smaller soot particle diameters compared to diesel. Works done by Lutic et al. [59] shows that soot particles can also be detected using resistivity sensors by employing thermophoresis technique as the particle deposition mechanism. Resistance variation was observed after certain accumulation of the soot in surface in accordance with filtration theory. Another important research work was done by Lee et al. [60] which reported the analysis of the design of thermophoretic probe. It was concluded that three factors that influence the probe vibration were (a) damping cylinder, (b) orientation of probe insertion, and (c) geometry of probe.

3.5 Emission Tomography (ET)

ET is typically used to calculate distributions of temperature and soot volume fraction from the emission spectrum of the non-uniform flame. A spectrometer or CCD image sensor is used in order to attain spectrum radiation figures from different directions by scanning a cross section of flame. The radiation received either in visible or in infrared spectrum mostly comes from soot particles [57]. Figure 7 shows the simple arrangement of ET system.

![Figure 7 Emission Tomographic](image)

Snelling et al. [61] applied this method to spectrally resolved measurement of flame radiation to determine soot temperature and concentration, which will be priceless for the generation of flamelet libraries and for the knowledge enhancement of soot formation mechanisms especially in laminar diffusion flames. Ayranci et al. [62] found that this particular method to be especially powerful in the near-infrared range for accurate prediction of flame properties where the spectral variation of optical constants is significant. Weikl et al. [63] used the same technique to determine the soot temperatures in an axisymmetric flame and was validated against coherent anti-Stokes Raman spectroscopy (CARS). Stereoscopic tomography technique was proposed by Huang et al. [64] in order to obtain the same parameters. While Zhao et al. [65] applied a 2D tomography principle to develop a new optical diagnostic method, named Cone Beam Tomographic
Three Color Spectrometry (CBT-TCS) to measure the planar distributions of temperature, soot particle size, and soot volume fraction in a co-flow axis-symmetric laminar diffusion flame. Zhou et al. [66] who analyzed the recent method of measurements in a coflow, diffuse ethylene-air flame by visible image processing, found that ET was one of the new techniques appropriate to measure soot volume fraction and temperatures in that particular environment.

### 3.6 Soot Measurement Summary

Overall, soot measurement principle, system, process and their characteristics can be summarized in Table 2 below:

<table>
<thead>
<tr>
<th>Measurement principle</th>
<th>Measurement system</th>
<th>Sampling / Source</th>
<th>Extraction / Classification</th>
<th>Diagnose / Characterization</th>
<th>Soot Characteristic</th>
<th>Advantages / disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Light absorption</td>
<td>LII</td>
<td>Laser pulse to the flame</td>
<td>Blackbody gives different radiation level</td>
<td>Measure particle diameter based on signal intensity</td>
<td>SVF and particle size</td>
<td>Considered as a non-intrusive technique, so able to provide high degrees of temporal and spatial resolution. But high soot concentration will cause attenuation of signal intensity.</td>
</tr>
<tr>
<td>Light extinction</td>
<td>LEM</td>
<td>Laser beam to the flame</td>
<td>Extinction occurs as a sum of absorption and scattering effect</td>
<td>Measure a particle’s shadow (areas where light is extinct) as it passes through the viewing region</td>
<td>SVF</td>
<td>Considered as a non-intrusive technique, so able to provide high degrees of temporal and spatial resolution. But only able to give limited spatial resolution as it is applied a line-of-sight technique</td>
</tr>
<tr>
<td>Light scattering</td>
<td>LSM</td>
<td>Scattering light through a dispersed particles</td>
<td>Measure the angular variation of the scattered beam</td>
<td>The intensity carries information about the particle mass, while the angular dependence carries information about the size of the particle</td>
<td>Particle size (equivalent sphere diameter)</td>
<td>Considered as a non-intrusive technique, so able to provide high degrees of temporal and spatial resolution. But it is difficult to suppress scattered light from other sources.</td>
</tr>
<tr>
<td>Thermophoresis</td>
<td>Thermophoretic sampling</td>
<td>Capture soot by entering hot flame</td>
<td>Collected onto grid probe</td>
<td>By electron microscopy either by SEM or TEM</td>
<td>Particle size and mass</td>
<td>Considered as an intrusive technique, so possibly disturb the combustion zone during sampling.</td>
</tr>
<tr>
<td>Tomographic principle</td>
<td>ET</td>
<td>Scan a cross-section of flame</td>
<td>Obtain spectrum radiation information from different directions</td>
<td>Monochromatic radiative intensity from soot</td>
<td>SVF and particle size</td>
<td>High level of accuracy, can also be used in condition where optical techniques based on laser are not suitable</td>
</tr>
</tbody>
</table>

### 4.0 DISCUSSION AND RECOMMENDATION

Basically, aerosol measurement technologies keep on improving since decades ago but the arising level of pollution and health effects due to exhaust emission indicates that current efforts to revolutionize these measurement technologies needs to be heightened. When the first soot measurement technique was
introduced 40 years ago, the interest parameter was only the particle size. Nowadays, other inputs (e.g. distribution, number, composition, volume, surface etc.) which are proven to give the same important effect to the study are already achievable.

As for measurement method specifically in-cylinder combustion, it is widely accepted that invasive techniques have some disadvantages compared to non-invasive techniques. For instance, probe sampling techniques (TEM grid) can distract the combustion region during the sampling which lowers the temporal and spatial resolution. Therefore, the combination of probe sampling techniques with the conjunction of optical methods such LEM, LII and LSM are preferable in order to reduce the temporal and spatial resolution problems. In some cases, ET is said to be the most widely applied soot measuring method in the future due to its advantages of high level of accuracy, simultaneously determination of 1-D/2-D distributions of temperature and soot volume fraction, and its ability to also be used in a condition where optical techniques based on laser are not suitable.

5.0 CONCLUSION

This paper offers a review of most common methods available for in-cylinder soot measurement. All methods such LII, LEM, LSM, Thermophoretic and ET are explained in a way that the basic principle of the methods together with their applications and issues are presented respectively. In general, all methods presented can be divided by two main categories; invasive and non-invasive.

Overall, non-invasive method is more frequently used due to various advantages over the other. But realizing that one method is not the panacea for all soot measurement requirements, there could be some possibility for a few methods to be used in combination in order to have a better solution and result. By doing so, almost all important soot measurement processes which include sampling, extraction, characterization and analysis methods will be more efficient and reliable.

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