the measurement of the size range and number distribution of airborne particles related to automotive sources - a literature study

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ABSTRACT

This report reviews the literature relating to particulate size measurement and the techniques suggested for different applications. Successful measurements have been made using cascade impactors, which give gravimetric analysis of sized fractions and electrical mobility techniques, which give number distributions of sized fractions. It is recommended that this approach be employed in the assessment of automotive particulate emissions. Because the subsequent fate of these emissions are subject to many complex and confounding factors, their measurement "at the tail-pipe" has only a tenuous relationship with air quality particulate inventories.

KEYWORDS

Diesel, emissions, particulates, Pm, particulate size

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SUMMARY

Airborne particulate, to which diesel engines contribute a significant amount, is an issue both with relation to air quality and health concerns. Recent worries have been raised concerning the size range and the number distribution of the particles within the total. All automotive vehicles are believed to emit particles in the 'ultrafine' range (i.e. <1 µm) but the data are very limited and various techniques of uncertain scientific veracity are purported to measure these parameters.

This document reviews the literature relating to particle size measurement and the applications suggested for different techniques. Successful measurements have been made using electrical mobility techniques and it is recommended that this approach be taken for the assessment of automotive particulate.

1. BACKGROUND

Since 1992, legislation has been in place in Europe to control the weight of particulate emitted to the atmosphere from both light and heavy duty diesel vehicles. Legislation has progressively tightened to reduce the allowable mass to 0.08 g/km for light duty vehicles[1] and 0.15 g/kWh for heavy duty vehicles[2] in 1996. Current legislation does not consider particulate emissions from gasoline vehicles.

Particulate has been regarded as a problem associated with diesel vehicles. Gasoline vehicles do emit particulate, although at a very low level (approximately 0.01 g/km). However, as legislation has tightened, and diesel emissions reduced, gasoline particles must now also be taken into account, especially considering the high percentage of gasoline vehicles in the car parc. The UK Particulate Inventory[3] list automotive sources as causing 27% of total UK PM10 (particulate with a mean aerodynamic diameter of 10 µm or less) of which a third is estimated to originate from gasoline vehicles.

There has been continued debate on two aspect of the possible health effects of particulate. First, it has been suggested that diesel particulate is carcinogenic due to the polycyclic aromatic compounds adsorbed on the surface of the particles.[4-7] The International Agency for Research on Cancer (IARC) has reviewed all the available data and classified diesel exhaust as a Group 2A carcinogen i.e. probably carcinogenic to humans.[3]

The second issue related to the effect of particulate air pollution on morbidity/mortality.[8-11] It is known that members of the general population with chronic obstructive pulmonary disease are particularly at risk during episodes of severe particulate air pollution. It is believed that particles with a mean aerodynamic diameter of 10 µm or less (PM10) are primarily responsible for the effects. However, there is growing evidence that particles with a mean aerodynamic diameter of 2.5 µm or less (PM2.5) could be more important in the development of health effects.

In 1974, Miller[12] investigated which size of particulate needed to be considered in order to set a standard for inhalable particles. Recently (1994), Friedlander and Lipmann[13] have addressed the same issue and stress the need for harmonisation of standards.

Recent air quality monitoring in the UK has focused on the mass of particulate below 10 µm and a level of 50 $\mu g/m^3$ has been proposed as an annual standard. In the US a PM10 standard was set by the EPA in 1986, with values of 150 $\mu g/m^3$ (24 hours) and 50 $\mu g/m^3$ (annual PM10). The EPA in the US has now shifted the emphasis from PM10 to smaller particles (PM2.5) and indeed examine the ratio of the two as a measure of air quality. Literature shows that automotive particulate fall predominantly in this smaller size range. Measurement of ambient atmospheric aerosol number concentration was reported by Willeke and Whitby in 1975[15] who suggested that an urban aerosol may reach particle concentrations of $4{,}000{,}000/cm^3$.

Data on the size distribution and the numbers of particles emitted from automotive sources are very limited. Different techniques for particle size measurement are available, each with their individual limitations. It is the aim of this document to review existing methods, their applications in the literature and to recommend a suitable route for the measurement of automotive particulate.

2. DEFINITION OF PARTICLES AND AEROSOLS

An automotive particle consists primarily of a carbon core onto which other chemical species (both organic and inorganic) are adsorbed. The exact chemistry of the particle will depend on fuel type, engine technology, speed and load conditions plus fuel and lubricant quality. The initial carbon particle (the nucleation mode) is probably spherical, but the shape will change as the particle cools and other species adsorb onto it (condensation mode). Finally particles may collide and join together (agglomeration mode), depending on their local environment (e.g. concentration, velocity, composition) before being emitted to the atmosphere as an aerosol. The size of the particle is greatly affected by external influences i.e. temperature, gas/vapour pressures of the hydrocarbons adsorbed and the relative humidity. There is also the possibility of exchange of chemicals between particles and for other species in the air (e.g. volatile organic compounds, sulphates) to adsorb onto them. As a result of these very complex interactions, aerosols are inherently unstable and thus the point of sampling is very important in order that results obtained are relevant for health effects. In the interpretation of particulate effect on air quality, weather has been shown to have the most confounding effect. In addition to the influence of humidity, temperature etc., wind, rain and snow will affect agglomeration, dispersion and deposition rates The sample measured must also be truly representative of the source to be analysed.

For measurement of automotive particles, sampling from the dilution tunnel will reduce further agglomeration and will allow consistent sampling. However, this will undoubtedly give values very different for both size and number from those actually emitted from the engine and possibly also from those encountered in the atmosphere. Work by Whitby[16] shows differences in size distributions based on mass measurements from those calculated from particle counts. Particle counting can count volatile particulates that would be lost during mass measurement techniques.

It is generally accepted that sampling must be carried out isokinetically[17] where the velocity of the sample stream is equal to the velocity of the gas stream being tested. Details of sampling techniques are given by Allen[18] and Fan et al[19] discuss how losses in isokinetic sampling can be avoided.

There are extensive standards in both the UK and US which refer to aerosol monitoring.

The diesel aerosol is polydisperse i.e. it has a wide range of particle sizes but generally appears to have a bimodal distribution[20] with ranges reflecting the nucleation (0.0075 - 0.056 µm) and agglomeration (0.056 - 1.0 µm) modes. Ambient aerosol is also bimodal[21] with peaks below 1µm and between 5-15 µm. The size and number of the particles measured by any technique is also subject to alteration by inconsistent sampling and losses in the instrument used. Biswas[22] describes distortion that can occur in measurements as a result of condensation and evaporation.

3. DEFINITION OF PARTICLE DIAMETER

There is only one true measurement of particle size which is the geometric diameter of a sphere. However, very few particles are spherical and therefore all non-spherical particles are quoted in terms of the diameter of an equivalent sphere exhibiting similar behaviour. The same non-spherical particle can have different diameters assigned to it depending on the way it is measured[113] and likewise differently shaped particles may have the same equivalent diameter assigned to them. There are many different forces acting on a particle in a gas stream, (flow forces - drag, lift; field forces - gravity, electrical, magnetic, centrifugal; inertial forces; inter-particle forces - impact, friction, adhesion; diffusion forces - thermophoretic, photophoretic, Brownian, composition). Thus, any technique used for the measurement of particle size must take into account a thorough understanding of the way the particles behave.

There are several different methods available for the measurement of particle size, each with an optimum size range that can be measured. Great care needs to be exercised both in the interpretation of data and in the comparison of data generated by different techniques. The interpretation of size is based on the diameter definition of the measurement procedure and particulate data can be represented in terms of numbers, volumes or mass. **Table 1** shows the optimum size range of particles measured by the different techniques[84] which are discussed in detail in **Section 4**.

Table 1 Size ranges measured by available techniques.

Operating Principle	Technique	Optimum Size range (µm)	Diameter
Sedimentation/Inertia	Cascade Impactor	0.5-15	Aerodynamic
	Low pressure Impactor	0.05-15	"
	MOUDI[1]	0.05-15	"
	Cyclone/Centrifuge	0.08-15	"
	Aerodynamic Particle Sizer	0.5-30	"
	Aerosizer	0.5-200	"
Optical	Nephelometry	0.3-100	Geometric
Microscopy	Optical	2-100	"
	SEM/TEM[2]	0.01-100	"
Electrical Mobility	Electrical Aerosol Analyser	0.01-1.0	Electrical mobility
	SMPS[3]/DMPS[4]	0.01-1.0	"

1.	MOUDI	Micro Orifice Uniform Deposit Impactor
2.	SEM/TEM	Scanning/Transmission Electron Microscope
3.	SMPS	Scanning Mobility Particle Sizer
4.	DMPS	Differential Mobility Particle Sizer

Owing to the nature of the aerosol the diameters of the particles are not easy to define. As stated, some may be spherical but many are chain agglomerates. There are several definitions of particle diameter that can be applied to the aerosol and the techniques used for its measurement may measure different things.[78]

The first diameter is the *geometric* diameter which can be measured directly by the use of microscopy and optical methods. The other two techniques commonly employed involve imposing a force on the particle,[78] with the nature of the force defining the diameter measured.

The cascade impactors and the cyclones expose the particles to mechanical forces which enables the particles to be classified according to their *aerodynamic* diameter. This is defined as “the diameter of a particle with equal settling velocity to the particle under study but with a density of 1 g/cm^3 ”. The aerodynamic diameter depends on the geometric diameter, the density and the shape of the particle. The collection efficiency of an impactor stage is usually plotted against Stokes number, itself a function of the aerodynamic diameter.[78] The mathematical relationship between the Stokes and the aerodynamic diameters has been discussed in detail by Hering.[79] The *Stokes diameter* is defined as 'the diameter of a sphere which has the same density and the same settling velocity as the particle under study'.[39] For spherical particles the geometric diameter is equal to the Stokes diameter. Using the aerodynamic diameter removes the need to determine the particle density.

The other major category of analyser involves electrically charging the particles to be measured. The diameter measured under such circumstances is called the *electrical mobility* diameter. The electrical mobility diameter as defined as 'the diameter of a spherical particle, which when charged under the same conditions, has the same electrical mobility as the particle under study'.[39] Particles which have negligible inertial mass are usually separated by electrical techniques.

Particles of equal Stokes number that carry the same electric charge will have the same electric mobility. Particles of equal density and equal Stokes diameter will have the same settling velocity i.e. aerodynamic diameter. Particles with the same size and shape but different densities will have the same Stokes diameter but different aerodynamic diameters. It must also be remembered that the Stokes equation is only followed when the particle is in laminar not turbulent flow (i.e. low Reynolds number).

Literature referring to the health effects of particles generally refers to the aerodynamic diameter of the particle. Both Stokes and aerodynamic diameters are important and it is known that a correlation exists between them.[79] However, the electrical mobility diameter is more conveniently and accurately measured.

4. METHODOLOGY FOR MEASURING PARTICLE SIZE AND NUMBER

4.1. SEPARATION BY SEDIMENTATION/INERTIA

This technique includes cascade impactors, cyclones, centrifuges and the Aerosizer. These give gravimetric measurements and also give the potential for subsequent chemical analysis of size separated fractions. Particle sizes are reported as aerodynamic diameter (see **Section 3**) which removes the need to determine the particle density.

4.1.1. Cascade Impactors

Cascade impactors are by far the most commonly used technique for aerosol sampling. The principle of cascade impaction for the size separation of airborne particles was introduced in 1945.[23] Measurements using this technique have been made on a wide range of sources, including automotive.[16, 17, 21, 24-29] The theory of the inertial separation of particles by cascade impaction was developed as early as 1931.[30] Details of the development of the technique and the associated theory are given by Pilat.[17] A book dealing with the subject has been written by Lodge[31] and details of the range of available cascade impactors are available from Graseby Andersen.[32] These consist of a series of plates, each of which is perforated with holes of a certain size (D50 i.e. the diameter at which the collection efficiency is 50%). The size reduces successively with each plate. The aerosol under test is passed through the plates with smaller particles remaining airborne longer to reach lower plates. Details of the operation of a cascade impactor are given in [17]. Problems with particle bounce have been reported and may give rise to false readings. Wang and John[33] and also Marple[34] have reported in detail on this phenomenon and how to overcome it.

Disadvantages of the cascade impactor are that they cannot accurately measure ultrafine particles and take too long to perform measurements which precludes real-time testing.

The size limitations have been overcome by the use of low-pressure impactors and the MOUDI (Micro Orifice Uniform Deposit Impactor) which can collect particles down to 0.05 µm. Details of the evaluation and calibration of this real-time impactor are given in[35] and the application of the technique to automotive measurement described by Venkataraman.[36] Evaluation and calibration of the MOUDI is given in [37]. The technique has been applied to the measurement of aerosols originating from the use of diesel engines in mines with good results.[37, 38] The application of the MOUDI for diesel exhaust is discussed by Kittelson.[39] Lin[14] has used the MOUDI to investigate the rate of deposition of particles as a function of their size.

The Quartz Crystal Microbalance (QCM) is another type of low pressure impactor that can also provide real-time measurement of the smaller particles.

The development of a new type of impactor - the vibrating tube impactor has been described by AEA Technology.[40] This allows different size particles to be simultaneously monitored.

4.1.2. Cyclones and Centrifuges

The first centrifugal device for separating aerosols into a continuous spectrum of sizes was called a conifuge and was developed in 1950.[41] The first attempts were not very successful, but the principle was applied by Stöber[23] who developed the spinning spiral duct. Aerosol centrifuges have good size resolution (down to 0.08 µm) and also give the advantage that subsequent analysis (chemical, microscopic) can be performed on the size separated fractions. Particles are precipitated according to their aerodynamic size in a (usually) conical shaped duct which is lined with either foil or collecting strip, which can be removed, sectioned, weighed and analysed.

For more detailed information, Moore and McFarland[42] give a literature review of cyclone performance and the criteria needed for optimum operation. Work by Lipmann and Chan[43] summarises the use of cyclones for particles <10 µm and remark that aerosol penetration in a cyclone can be used to model respiratory deposition.

4.1.3. Aerodynamic Particle Sizer and Aerosizer

The most sophisticated analytical techniques within this section are the Aerodynamic Particle Sizer (APS)[44, 45] and the Aerosizer, which can measure particles down to 0.05µm. These techniques enable real time analysis of particles, by measuring the time of flight of the particle under study between two laser beams. Particles are accelerated from a nozzle and will lag behind the downstream air to an extent dependent on their aerodynamic diameter. The lasers measure this lag. Clarke[46] believes this technique to be unsuitable for the measurement of diesel exhaust because of their size. The density of the particles is important and corrections need to be applied.[44, 47] Marshall[48,49] has carried out extensive studies on the effect of the shape of the particle on the response of these instruments. The technique can severely undersize both non-spherical and the smaller particles. It was found that larger particles were oversized by the APS and marginally undersized by the Aerosizer. Details on the Aerosizer can be obtained from TSI.[50] Its use has not been recommended for the analysis of automotive particle assessment.

4.2. OPTICAL METHODS/MICROSCOPY

Overviews of the use of optical techniques for the application to diesel particulates are given in [46, 51].

4.2.1. Light Scattering Techniques

Light Scattering has been used to characterise both the size and shape of airborne particles.[52] The amount of light scattered is a function of the particle size, shape, composition and surface and hence interpretation is difficult. Light scattering methods are most effective when the particles measured are in the same order of size as the wavelength of the incident radiation.[18] Clarke[46] considers that the technique is not suitable for very small particles (< 1µm), as the efficiency of the light scattering decreases when the size of the particle falls below the wavelength of light. This can be overcome by using the particles as condensation nuclei for a liquid (e.g. butanol) to increase their diameter. This enables particles down to 0.01 µm to be detected and Heintzenberg[53] has successfully used optical techniques for the measurement of submicron particles in clouds. Condensation Nuclei Counters (CNC) have no size discrimination and have to be used in conjunction with a sizing technique (e.g. a diffusion battery, a technique for separating very small particles by their diffusivity, i.e. Brownian motion), Electrical Aerosol Analyser (EAA) or Differential Mobility Analyser(DMA)). Combinations of this type were investigated by Kittelson in the measurement of diesel exhaust particulates.[39, 54] Detailed product information relating to commercially available CNCs is published by TSI.[55] Further details on the application are given by Agarwal[61] and details on the calibration by Liu and Pui.[57] Further work on the calibration of the particle counter with respect to non-spherical particles has been carried out by Buettner.[58]

Light scattering techniques have been applied by Kievit[59] for the on-line measurement of particle size and also by Tree[60] for the measurement of the size and number of diesel particulates as a function of speed and load.

4.2.2. Microscopy

The use of electron microscopy (both scanning and transmission) enables a detailed analysis of the size and shape of particles. Kittelson[20] has successfully applied the technique to diesel exhaust. SEM can examine particles in the range of 0.01-0.2 µm. A good overview of the technique is given by Lee and Fisher.[61] Care needs to be taken with sampling as collection may cause distortion. Samples can be successfully prepared by electrostatic precipitation onto a filter or grid.[46]

4.3. ELECTRICAL MOBILITY

The most referenced technique with respect to the size analysis of automotive particulate is electrical mobility.[20, 39, 54, 62-65, 114-116] Kittelson[39] describes in detail the use of electrical mobility as a suitable technique, stating that 'the electrical mobility of a singly charged particle is a monotonic function of particle size'. He also comments that along with cascade impactors the use of electrical mobility is the most widely used means of sizing diesel particles.

The principle of the technique involves imparting a charge to the aerosol, which then flows through apparatus of opposing charge and can be separated onto surfaces, or into counters, depending on the size of the opposing charge. The stream of sized particles is then passed through a CNC.

There are two widely used commercial techniques - the Electrical Aerosol Analyser (EAA) and the Differential Mobility Analysers (DMA). The latter are divided into several specialised instruments e.g. Scanning Mobility Particle Sizer (SMPS), Differential Mobility Particle Sizer (DMPS) and Tandem Differential Mobility Analysers. A comparison of the techniques has been performed by Horton[66] who concluded that although the combination of the DMPS /CNC gave the most repeatable answers, the use of the EAA was better for real-time analysis.

The EAA provides a means of obtaining rapid low-resolution measurements of submicron size distributions.[67] The DMPS gives better size resolution than the EAA but has a measurement cycle time about 15 times longer. The new SMPS instrument offers better size resolution and a measurement cycle time of circa 2 minutes which makes it an ideal choice for the measurement of automotive particulates.

4.3.1. Electrical Aerosol Analyser

An early paper by Liu and Pui[68] gives details regarding the detailed operation of the EAA and its limitations. The paper also describes the application of the technique to the measurement of particles in ambient aerosols, rocket exhaust and also automotive aerosols.[69] Mulholland[70] compares the use of the EAA with other techniques. Although the EAA had been used widely for the measurement of monodisperse aerosols (where the particles are all the same size), the application to polydisperse aerosols was less well understood. Although generally good agreement between the techniques was observed, it was considered that the accuracy of the EAA was affected by particle shape. The application of EAA to air monitoring is described by Eldering.[71]

4.3.2. Differential Mobility Analysers

Wang and Flagan[72] describe in detail the use of the Scanning Electrical Mobility Spectrometer. The development of both the EAA and the DMPS has led to electrical mobility becoming the standard technique for the analysis of small particles (<0.1µm). However, measurement of changing aerosols gives problems because of the finite time needed to perform measurements. This restriction has led to the development of the SMPS where the electric field is scanned continuously, rather than in discrete steps. The technique has been used by MIRA[73] to measure the size and number distribution of particles emitted from both diesel and gasoline engines under steady state conditions.

Mayer[74] has used the DMA to investigate the size of particles downstream of a particulate trap, measuring particles down to 20 nm. Flagan[75] has also measured very small particles (2-3 nm) using the DMA whilst monitoring the formation of secondary particulate.

The Tandem Differential Mobility Analyser has been used to measure changes in particle sizes due either to changes in relative humidity[76], or due to evaporation and condensation.[77]

5. CALIBRATION OF INSTRUMENTS

All techniques need careful calibration. If the properties of the aerosol under test differ to those of the calibration medium, the results may be misleading. There are many references relating to the difficulty in the accurate calibration of the various techniques. Calibration of instruments takes place with spherical particles of unit density and it has been shown that sometimes measurement of non-spherical particles is under or over estimated depending on geometric shape.[48] Karg et al[80] describe the effects of relative humidity and gas flow on the size of monodisperse latex particles measured with the DMA. This problem was also assessed by Biswas[22] using a cascade impactor. Fairchild and Wheat[35] describe the calibration of a real-time cascade impactor and Baron[44] goes into detail on the calibration for the Aerodynamic Particle Sizer (APS-300). Calibration and use of the MOUDI is described by Marple et al.[37]

6. AIR QUALITY MONITORING EXPERIMENTS

Air quality monitoring has taken place since the 1960's. Networks are in place in both the US,[81] controlled by the EPA and in the UK[82] as part of the Department of Environment Urban Network. The latter was set up in 1992. Air quality monitoring for particulate originally involved sampling ambient aerosols for Total Suspended Particulate (TSP)[83] or Black Smoke. As health concerns have now focused on smaller particles, more measurements now concentrate on PM10. This means that the cut-off diameter for which the measurement device has a collection efficiency of 50% (D50) is 10µm. Larger particles may still be collected but the collection efficiency is much lower.[84] The US EPA imposed a PM10 standard in 1986. Many different countries do have air quality standards for particulate but as yet there is no uniformity with standards either for Black Smoke, Total Suspended Particles or PM10.

The instrument most commonly used for this measurement is the TEOM (Tapering Element Oscillating Microbalance) which gives a gravimetric total of all PM10. Rupprecht [85] describes the use of the TEOM for this application.

Size measurement of particles in air was referenced by Whitby[16, 21] in the early seventies, where data were collected using an Anderson Cascade Impactor. The cascade impactor was also used by Berner[86] for the same application. There have been a number of detailed references looking at ambient aerosol size distributions using other techniques: EAA[71,87] and MOUDI[88]

Work has now been extended, not only to monitor the size range and number distribution of the ambient aerosol but also to investigate specific particles (e.g. inorganic ions (sulphate, nitrate)[24, 89, 90] hydrocarbon droplets[26, 36, 91, 92]). Milford[93,94] has published reviews on the sizes of inorganic particulates (ions and trace metal) in the atmosphere. Measurements have also been made to understand in more detail the factors that affect particulate size e.g. relative humidity[22, 76, 80, 95, 96] and how their size affects their deposition.[14]

Particles in the air may be either primary (emitted directly from their source e.g. combustion of fossil fuel) or secondary (formed in the atmosphere) and both may originate from either man-made or biological sources.[84] Contributions to the ambient aerosol from fugitive dust are also highly significant. Chemical analysis of particles can give a very accurate 'fingerprint' of the source of the particle and it has been found that particles from specific sources have distinctive ranges of size.[27, 36, 59, 91, 92, 97-100] Extensive work in this area has been carried out by Friedlander[97] as early as 1970 and he claims that "a complete description of the atmospheric aerosol would include an accounting of the chemical composition, morphology, size of each particle and the relative abundance of each particle type as a function of size".

Sampling aerosols and obtaining a range of size distribution data with corresponding chemical analysis can be used by modellers to attribute sources and calculate quantities of particulate emitted to the atmosphere from each source.[101-103] Modelling however may not reflect the true air quality situation, where it has been shown that the major confounding effects on particles are due to the weather conditions. Thus a model provides only a 'snapshot' of the situation.

7. EXPERIENCE IN THE MEASUREMENT OF AUTOMOTIVE PARTICULATE

Early work on the size distribution of the particles in diesel exhaust was carried out by Kittelson et al.[20, 62] Measurements were performed on dilute exhaust using the Electrical Aerosol Analyser (EAA) in combination with a transmission electron microscope. Later work[63] by Kittelson involved the roadway measurement of diesel aerosols, now using a condensation nuclei counter (CNC) with the EAA. Kittelson et al have also reported[39] a comparison of different size distribution techniques (EAA, Diffusion battery with CNC, MOUDI) for the application to diesel exhaust. The MOUDI has also been used[37, 38] to investigate in-mine diesel emissions. Pierson[29] reported on the measurement of particles in tunnel experiments in Pennsylvania based on data collected from an Andersen cascade impactor. Following the collection of the samples, the fractions were subjected to detailed chemical analysis. This paper gives extensive references relating to the size and composition of all automotive particulate. Venkataraman[36] has successfully applied the low pressure impactor to the measurement of automotive emissions.

Other investigations of diesel exhaust particle size distributions have been made by Groblicki,[104] Patschull[105] (using the SMPS) and Rogge.[106] Groblicki reports that there was no consistent effect of speed on particle size although increasing load had a tendency to increase the sizes. Recent work by Kittelson[22] describes how real time measurements of total, volatile and non-volatile particles can be made in vehicle exhaust using the EAA in combination with the CNC. Most of the results available in the literature refer to steady state testing. Lepperhoff of FEV et al[114] describe in detail the size measurement and percentage distribution of particles from four light duty diesel vehicles (also using the EAA) over the Federal test cycle. Samples were collected that were representative of the complete cycle and distributions were found to be very similar for all four vehicles, showing no difference between direct injection (DI) and indirect injection (IDI). Distribution of the particles showed maxima at 13 nm and also (for 3 vehicles) at 237 nm. The high speed direct injection (HSDI) engine was seen to give the lowest number of small particles (<100 nm) but a higher number of larger particles (>400 nm).

In a further paper describing this work, Hammerle et al[115] describes in detail size distribution data from six light duty vehicles over both Federal and European test cycles. Samples were collected that were representative of the complete cycles and distributions were found to be very similar, both with respect to cycle and vehicle.

Both references,[114,115] although dealing with emissions over a test cycle, do not attempt transient monitoring. Recent work by MIRA[84] describes work which monitors one size range of particulate over transient testing (both gasoline and diesel). Similar work by Panne[116] uses two DMPS in parallel to study transient particle sizing.

Measurement of particle size in exhaust has not been restricted to diesels. Analysis of particles from a 2-stroke engine has been conducted by Patschull[65] using the SMPS. There are also several other references to gasoline particle size distributions.[64, 69, 106-109] Recent work by MIRA[73] has used the SMPS to investigate size range and number distribution from both diesel and gasoline engines under steady state conditions. This shows the mass of gasoline particulates to be much lower than diesel overall but the numbers to be comparable at selected speed and load, where indeed the gasoline particles are much smaller. Kittelson has recently presented work[110] performed on all automotive particulate which confirms the work of MIRA.

In addition to the analysis of engine out particles, there are a number of references looking at the effect of exhaust after-treatment on particle size and distribution.[54, 74, 111] Although traps and filters can remove particles effectively from engine exhaust, there is a potential for the downstream formation of secondary particles.[54]

There is very little information available concerning fuel effects on particulate size and number. However, Baumgard and Johnson[112] have investigated the effect of low sulphur diesel fuel and have concluded that although the size of particulate is not affected the number of particles emitted are greatly reduced when the low sulphur fuel is used.

8. CONCLUSIONS

An automotive particulate consists primarily of a carbon core onto which other chemical species (both organic and inorganic) are adsorbed. The exact chemistry of the particulate will depend on fuel type, engine technology, speed and load conditions, plus lubricant quality. The initial carbon particulate is probably spherical, but the shape will change as the particulate cools and other species adsorb onto it. Finally, particulates may collide and join together, depending on their local environment, before being emitted to the atmosphere as an aerosol. The size of the particulate is greatly affected by external influences and there is also the possibility of exchange of chemicals between particulates and for other species in the air to adsorb onto them. As a result of these very complex interactions, aerosols are inherently unstable. This strongly suggests that the measurement of automotive exhaust particulate has only a tenuous relationship with air quality particulate inventories.

There are several different methods available for the measurement of particulate size, each with an optimum size range that can be measured. Great care needs to be exercised both in the interpretation of data and in the comparison of data generated by different techniques.

For measurement of automotive particulates, sampling from the dilution tunnel will reduce further agglomeration and will allow consistent sampling. However, this will undoubtedly give values very different for both size and number from those actually emitted from the engine and probably from those encountered in the atmosphere. It is generally accepted that sampling must be carried out isokinetically.

The advantages and disadvantages of various techniques can be summarized as follows:

- Disadvantages of the cascade impactor are that they cannot accurately measure ultrafine particulates and take too long to perform measurements which precludes real-time testing. The size limitations have been overcome by the use of low-pressure impactors and the MOUDI (Micro Orifice Uniform Deposit Impactor) which can collect particulates down to 0.05µm. The Quartz Crystal Microbalance (QCM) is another type of low pressure impactor that can also provide real-time measurement of the smaller particulates.

- Aerosol centrifuges give good size resolution (down to 0.08µm) and also have the advantage that subsequent analysis (chemical, microscopic) can be performed on the size separated fractions. The most sophisticated analytical techniques within this group are the Aerodynamic Particulate Sizer (APS) and the Aerosizer, which can measure particulates down to 0.05µm. These techniques enable real time analysis of particulates, but the technique can severely undersize both non-spherical and the smaller particulates. It was found that larger particulates were oversized by the APS and marginally undersized by the Aerosizer. Its use has not been recommended for automotive particulate assessment.

- Light Scattering has been used to characterise both the size and shape of airborne particulates. The amount of light scattered is a function of the particulate size, shape, composition and surface and hence interpretation is difficult. The technique is not considered suitable for very small particulates (<1µm) without sample pre-treatment.

- The use of electron microscopy (both scanning and transmission) enables a detailed analysis of the size and shape of particulates and has been successfully employed to examine diesel exhaust.

- The most referenced technique with respect to the size analysis of automotive particulate is electrical mobility. There are two widely used commercial techniques - the Electrical Aerosol Analyser (EM) and the Differential Mobility Analysers (DMA). The latter are divided into several specialised instruments, e.g. Scanning Mobility Particulate Sizer (SMPS), Differential Mobility Particulate Sizer (DMPS) and Tandem Differential Mobility Analysers. A comparison of the techniques has led one researcher to the conclusion that, although the combination of the DMPS/CNC gave the most repeatable answers, the use of the EM was better for real-time analysis.

Although the EM has been used widely for the measurement of monodisperse aerosols (where the particulates are all the same size), the application to polydisperse aerosols is less well understood. Although generally good agreement between the techniques is observed, it is considered that the accuracy of the EM is affected by particulate shape. The development of both the EM and the DMPS has led to electrical mobility becoming the standard technique for the analysis of small particulates (<0.11µm). However, measurement of changing aerosols gives problems because of the finite time needed to perform measurements.

9. RECOMMENDATIONS

There are many references in the literature relating to the measurement of automotive particle sizing. The techniques used most commonly are cascade impactors, which give gravimetric analysis of sized fractions and the electrical mobility techniques which give number distributions of sized fractions. Both techniques give valuable information and any programme of work should include both types of analysis to further assess the impact of fuels, catalysts and technology on automotive particle size and distribution.

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