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National Clean Air Conference: THE IMPACT OF SULPHUR CONTENT OF DIESEL FUEL ON ULTRAFINE PARTICLE FORMATION

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Summary

Early this year the Australian Department of Environment and Heritage commissioned a desktop literature review with a focus on ultrafine particles including analysis of health impacts of the particles as well as the impact of sulphur content of diesel fuel on ultrafine particle emission. This paper summarizes the findings of the report on the link between the sulphur content of diesel fuels and the number of ultrafine particles in diesel emissions. The literature search on this topic resulted in over 150 publications. The majority of these publications, although investigating different aspects of the influence of fuel sulphur level on diesel vehicle emissions, were not directly concerned with ultrafine particle emissions. A specific focus of the paper is on:

- summary of state of knowledge established by the review, and
- summary of recommendations on the research priorities for Australia to address the information gaps for this issue, and on the appropriate management responses.

Keywords: Diesel, ultrafine particles, emissions, diesel fuel, sulphur.

1. Introduction

This paper presents a summary of the literature review on the relationship between diesel fuel sulphur content and the number of ultrafine particles in diesel emissions. The full review was presented as a report to the Australian Department of Environment and Heritage earlier this year. The initial search has led to more than 150 references (journal articles, conference presentations, reports, etc.) on the fuel sulphur level and diesel emissions but only a small number of them covered the topic of nanoparticle formation and emissions. A summary of the results and conclusions from these relevant references is presented in the report.

Before, however, tackling with the question of the influence of the fuel sulphur level on nanoparticle emission it was necessary to first review the current state of knowledge as to what constitutes diesel exhaust, on the complex issues of nanoparticles in diesel exhaust, on their physical properties, chemical composition and formation and on the mechanisms governing nanoparticle formation and their physical and chemical properties. It is important to stress that the mechanisms governing nanoparticle formation are still not fully understood and the theories presented here still have to be confirmed by more hard scientific data.

Finally the report presents conclusions regarding the current state of knowledge and the scale of the problem as well as recommendations as to directions for addressing the gaps in knowledge and well as for adequate management responses. The structure of this paper is similar to the structure of the report.

2. Diesel Particulate Matter

Particulate matter, perhaps the most characteristic of diesel emissions, is responsible for the black smoke traditionally associated with diesel-powered vehicles. The diesel particulate matter emission is usually abbreviated as PM or DPM, the latter acronym being more common in occupational health applications. Diesel particles form a very complex aerosol system. Despite a considerable amount of basic research, neither the formation of DPM in the engine cylinder, nor its physical and chemical properties or human health effects are fully understood. Nevertheless, on the basis of what is already known, DPM is perceived as one of the major harmful emissions produced by diesel engines. Diesel particles are subject to diesel emission regulations worldwide and, along with NOx, have become the focus in diesel emission control technology.

Contrary to gaseous diesel emissions, DPM is not a well-defined chemical species. The definition of particulate matter is in fact determined by its sampling method, the detailed specification of which is an important part of all diesel emission regulations. DPM sampling involves drawing an exhaust gas sample from the vehicle's exhaust system, diluting it with air, and filtering through sampling filters. The mass of particle emissions is determined based on the weight of DPM collected on the sampling filter. It is quite obvious that any changes in the procedure, for example using a different type of sampling filter or different dilution parameters, may produce different results. Standardization of sampling methods is of the utmost importance if results from different laboratories are to be comparable. Such standards have been developed for the measurement of PM mass in the area of public health regulations (i.e., emission standards for diesel engines and vehicles) worldwide. Ongoing research in Europe is aimed at developing standardized measuring methods based on particle number emissions, rather than mass, for the inclusion in future emission standards in addition to the PM mass metric (Andersson, 2002). So far no common standard has been reached in the area of diesel occupational health regulations, where a number of different measuring methods and corresponding DPM definitions exist in parallel.

Interest in particulate size distributions and nanoparticle emissions from diesel engines was sparked by reports that newer technology engines—designed for low PM mass emissions—may still generate high particle numbers. The most significant study indicating such possibility (involving measurement of particle size distributions from an older and a newer generation heavy-duty diesel engine) was published by the Health Effects Institute (HEI) (Bagley, 1996). As indicated by later research which was based on more comprehensive data, from several engine models, old and new technology engines produce, in general, nuclei modes of similar magnitude; the PM mass reductions in new engines are due to a smaller number of particles in the accumulation mode (Kittelson, 2002; Ristovski, 2002; Andersson, 2001). It is believed that emissions from the new engine in the HEI study—an experimental 1991 design—have not reflected general emission trends in new technology engines. That study, however, must be credited with prompting quality research by the diesel industry, academia, and governments, leading to a greatly increased understanding of particulate emissions from internal combustion engines.

2.1 Diesel Particle Size Distribution

A typical size distribution of diesel exhaust particles is shown in Figure 1 (note that a logarithmic scale is used for particle aerodynamic diameter). Nearly all diesel particles have sizes of significantly less than 1 µm. As such, they represent a mixture of fine, ultra-fine, and nanoparticles. Due to the current PM sampling techniques (diluted exhaust, temperature <52°C), diesel particulate matter includes both solids (such as elemental carbon and ash) and liquids (such as condensed hydrocarbons, water, and sulphuric acid). Formation of particles starts with nucleation, which is followed by subsequent agglomeration of the nuclei particles. The nucleation occurs both in the engine cylinder (carbon, ash) and in the dilution tunnel (hydrocarbons, sulphuric acid, water), through homogeneous and heterogeneous nucleation mechanisms.

Size distributions of diesel particles have a well-established bimodal character, which corresponds to the particle nucleation and agglomeration mechanisms, with the corresponding particle types referred to as the nuclei mode and the accumulation mode. Size distributions are usually presented using either particle mass or particle number weighting. In each representation normal-logarithmic distribution curves are produced, as shown in Figure 1. Both the maximum particle concentration and the position of the nuclei and accumulation mode peaks, however, depend on which representation is chosen. In mass distributions, the majority of the particles (i.e., the particulate mass) are found in the accumulation mode. In number distributions, on the other hand, most particles are found in the nuclei mode. In other words, diesel particulate matter is composed of numerous small particles holding very little mass, mixed with relatively few larger particles, which contain most of the total mass. A small fraction of diesel particles reside in a third, coarse mode (Figure 1). These three particle modes can be characterized as follows:

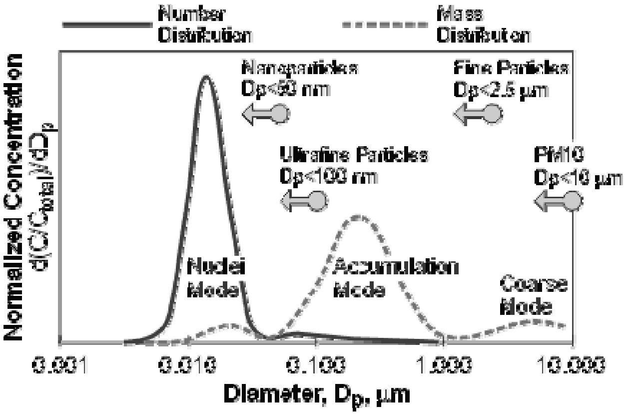

Figure 1 Diesel Particulate Size Distribution (Kittelson, 2002)

Nuclei mode: The diameter of the original nucleus, such as formed during sulphuric acid nucleation, is about 1 nm (Abdul-Khalek, 1999). Today's measuring techniques are capable of detecting a minimum particle size of approximately 3 nm. According to various definitions, the diameters of nuclei mode particles were generally less than 40-50 nm (0.04-0.05 µm). Based on particle size research in the 1990's technology heavy-duty diesel engines, it has been postulated that the nuclei mode extends through sizes from 3 to 30 nm (0.003-0.03 µm) (Kittelson, 2002). All of the above size ranges place nuclei mode particles entirely within the nanoparticle range. The maximum concentration of nuclei mode particles occurs at 10-20 nm. The nuclei mode, depending on the engine technology and particle sampling technique, typically contains only 0.1-10% of the total PM mass; however it often includes more than 90% of the total particle count. Sometimes the nuclei mode particles present as much as 99% of the total particulate number. Nuclei mode particles are composed mostly of volatile condensates (hydrocarbons, sulphuric acid) and contain little solid material.

Accumulation mode: The accumulation mode is made of sub-micron particles of diameters ranging most often from 30 to 500 nm (0.03-0.5 µm nm), with a maximum concentration between some 100-200 nm (0.1-0.2 µm). As shown in Figure 1, the accumulation mode extends from the upper end of the nanoparticle range through to the ultrafine and fine particle range. Accumulation mode particles are made of solids (carbon, metallic ash) intermixed with condensates and adsorbed material (heavy hydrocarbons, sulphur species).

2.2 Current Theories on Nanoparticles: Composition and Formation

Generally, diesel nanoparticles include the same constituents that are found in the total particulate matter emissions including elemental carbon, ash, hydrocarbons, sulphuric acid, and water. Contrary to the total PM emission, diesel nanoparticles cannot be chemically analysed to precisely determine their composition. There is no sampling/analytical procedure that would allow for separation of a sufficient mass of the nanoparticle material for such an analysis. As a result, the exact formulation of diesel nanoparticles has to be studied through indirect experiments. A consensus has been forming that diesel nanoparticles are mostly of volatile nature. In most cases, they are composed primarily of hydrocarbon and sulphuric acid condensates with small contribution of solid material, such as ash and carbon.

The nanoparticle composition can be dramatically changed by exhaust emission control measures, such as diesel particulate filters or fuel additives. Very high numbers of nanoparticles were measured downstream of particulate filters in the VERT study (Mayer, 1997). The high nanoparticle emissions observed downstream of particulate filters were composed primarily of condensates (strictly speaking, this conclusion was valid only within the measuring system detection limit of 15 nm, below which solid particles still could be found).

Nanoparticle volatility experiments were also conducted in the CRC E-34 study (Kittelson, 2002). These results suggest that hydrocarbons that make up diesel nanoparticles are derived mostly of the engine lube oil. It should be mentioned, however, that some fuel effect studies found increased nanoparticle numbers with more volatile fuels, indicating that fuel derived HCs may also present a noticeable contribution (Wedekind, 2000).

Various hypotheses have been formulated to explain the increased particle numbers that were seen in certain studies with new diesel engines. It was once suspected that the high particle numbers were related to high injection pressures which are used by engine manufacturers as a strategy for meeting emission targets. That theory, however, has not been confirmed. On the contrary, a continuous decrease in mass and number emissions was shown when injection pressure increased from 400 to 1000 bar. In another study, the injection pressure had only a small influence, and only under some engine operating conditions, on the particle size distribution (Pagan, 1999).

It is believed that increased number emissions are a simple consequence of:

a. high concentration of nanoparticle precursors (HC, SO4), combined with
b. decreased mass of accumulation mode particles.

Normally, the accumulation particles act as a “sponge” for the condensation and/or adsorption of volatile materials. In the absence of that sponge, gas species that are to become liquid (or solid) will nucleate to form large numbers of small particles. The driving force for the gas to particle conversion is the saturation ratio, defined as the ratio of the partial pressure of a species to its saturated vapour pressure. For the constituents of the SOF or sulphuric acid, the maximum saturation ratios occur during dilution and cooling of the exhaust and are typically achieved at dilution ratios between 5 and 30 (Abdul-Khalek, 1998).

The above nucleation theory explains the high number emissions seen downstream of particulate traps (which remove solid accumulation mode particles, but not necessarily nanoparticle precursors), as well as high number emissions from engines of high SOF fraction, such as the ’91 Cummins engine in the HEI study (Bagley, 1996). It is also consistent with the observation of high particle numbers from gasoline engines, which are believed to be composed primarily of liquid condensates (Graskow, 1998). In all of these cases, the low quantities of agglomerated particles present in the gas cannot provide enough surface area for the condensation/adsorption of volatile material. As the species approach saturation, high numbers of small particles are produced through nucleation.

A similar nucleation mechanism may apply to the formation of ash particles. In that case, the ash nucleation takes place inside the engine during the expansion stroke rather than in the dilution tunnel. Once these ash nuclei are formed, they may serve as heterogeneous nucleation sites for SOF and other species during dilution and cooling in the exhaust. It was suggested that some particles in the high SOF nuclei mode might include ash cores (Abdul-Khalek, 1998).

3. Influence of The Fuel Sulphur Level on Nanoparticle Emissions

Out of more than 150 publications, which mention the topic of the influence of fuel sulphur level on nanoparticle emissions, only a few are directly focused on this topic, while most of the studies concentrated on particle mass and not number emissions. However, even these few are not without limitations. Firstly, only one study was conducted on a larger number of vehicles while all the other studies either investigated a single type of engine/vehicle or just a few engines, where each engine represented a certain type (EURO I, EURO II, etc.). Secondly, some of the studies were conducted on engine dynamometers while others investigated vehicles on chassis dynamometers. This added additional uncertainties when comparing different studies. And finally there is still not a clear consensus in the scientific community on the procedures for particle number size distribution measurement during vehicle emission testing. Different studies have used different sampling methods, which were very often not described in full detail. Since sampling method itself affects particle formation processes, a meaningful comparison of the results of different studies is not always possible.

Historically the first program was conducted by the Health Effects Institute (HEI) (Bagley, 1996). They analysed the influence of fuel sulphur content on the emissions from 2 heavy-duty diesel engines (1988 and 1991 model Cummins engines) with 2 types of fuel with sulphur level of 0.32% High Sulphur (CS), and 0.01% (100ppm) or Low Sulphur (LS). Although the investigators observed a reduction in SO_4 below the detection limits, a statistically significant difference in TPM levels between the CS and LS fuels was found only at 25% load. They have conducted a limited number of measurements of particle size distributions. Their main finding was a significant reduction of the number of smaller (nuclei-mode) particles when the sulphur levels were reduced from 3200ppm to 100ppm. The study recommended further investigation of the influence of the fuel sulphur level and after treatment devices on particle number and size distribution.

Only recently several reports have been published on the influence of fuel specification on particle number, mass and size of emitted particles (Andersson, 2001; Kittelson, 2002; Wedekind, 2000; Ristovski, 2002).

A recent European study conducted within the DETR/CONCAWE/SMMT Particle Research Programme has concentrated on the influence of the sulphur level on nanoparticles emissions (Andersson, 2001; Wedekind, 2001). The Anderson (2001) and Wedekind (2001) study investigated only 3 heavy duty diesel vehicles (EURO I, EURO II and EURO III) for 3 different sulphur fuel levels: 340-ppm, 53-ppm, and less than 10-ppm. Measurements were conducted for both steady state (R49) and transient cycles (ETC).

For the steady state R49 cycle they found that the changes in accumulation mode particles could be attributed to changes in engine technology. However, the variation in nanoparticles might be influenced by fuel properties. The effects of engine technology proved larger on regulated particle mass emissions than those of fuel specification. With both engines, fuel with highest sulphur content (340 ppm) emitted highest and fuel with lowest sulphur content (<10ppm) lowest weighted cycle nanoparticle emissions. In chemical terms although hydrocarbon and sulphate masses were small, the influences on nanoparticle formation can be significant.

The light duty part of their program (Anderson, 2001) studied three different vehicles (EURO II and EURO III class equipped with a DPF) over four different sulphur fuel levels (500, 300, 50 and <10 ppm sulphur content) and transient and steady state conditions. They found that the lowest numbers of nucleation mode particles were emitted by the fuel with less than 10ppm sulphur, which resulted in the lowest total number of particles. They concluded that the engine technology effects dominated the accumulation mode (particles larger than 50nm), while the fuel dominated the nucleation mode particles.

Another study concentrating only on a single engine and on an engine dynamometer was conducted by Wei et al (2001). Wei et al (2001), who studied the emissions from a 1995 model medium-range diesel engine operating at 50% load. Two fuel sulphur contents were used: 440 ppm (low sulphur) and 10 ppm (ultra low sulphur). They found that increasing the fuel sulphur content increased the formation of nucleation mode particles, but did not significantly influence the accumulation mode. The 10 ppm sulphur fuel gave smaller concentrations of nucleation mode particles than the 440 ppm sulphur fuel. The peak particle number concentration in the nucleation mode was much higher with the low sulphur fuel than with the ultralow sulphur fuel. The number of particles in the nucleation mode was also strongly influenced by temperature. The total number concentration produced by the two fuels was quite similar at temperatures above 30°C, but, as the temperature was reduced further, the total number produced by the higher sulphur fuel increased much more rapidly. At 15°C, the total number concentration produced by the low sulphur fuel was nearly 7 times higher than that produced by the ultra-low sulphur fuel.

Recent Australian study conducted by Ristovski et al. (Ristovski, 2002) examined particulate emissions from a fleet of twelve in-service buses fuelled by low (500 ppm - LS) and ultralow (50 ppm -ULS) sulphur diesel at four driving modes on a chassis dynamometer. The examined busses were between 1 and 12 years old (pre EURO I, EURO I and EURO II). Both size and number as well as particle mass were measured. The study found that the particle mass emission rates were not statistically significantly different for the two fuel types. However, the particle number emission rates were 30-60% higher with the LS fuel over the ULS fuel. Most of the excess particles were smaller than 50 nm (nanoparticles) and resided in the nucleation mode.

The study further investigated whether the age or mileage of a bus had an influence on the particle emissions and how, if at all, they are affected by the sulphur content of the fuel. The reduction of particle number emissions with reduced fuel sulphur content was highest for the modern buses. It decreased with age and mileage but did not show any difference between the emissions with the two types of fuel after they passed a certain age and mileage (500,000 km or 8 years).

As in newer types of engines the majority of particles are in the nucleation mode: preventing the formation of this mode will result in a significant decrease of the total number of particles, in some cases up to 2 orders of magnitude. Therefore the reduction of the particle number would be much more prominent in the engines that have lower particle mass emissions, such as Euro 2 type engines. In the instances where the formation of the nucleation mode was already suppressed with 500ppm (LS) fuel (i.e. possibly due to a greater particle surface area being available in the accumulation mode) there was only a small reduction, if any, in the total particle number emission with 50ppm (ULS) fuel.

It is interesting to note that the nucleation mode in this study was also not totally suppressed with the ULS fuel and in a small number of cases (only in 3 cases out of around 50) the nucleation mode was observed with ULS fuel but not with LS fuel. This indicates that the sulphuric acid inhibits the formation of the nucleation mode, but is not the only component responsible for the formation of this mode. A similar finding that lubricating oil, unburned hydrocarbons from the fuel as well as PAH could also play a critical role in the formation of the nucleation mode was confirmed by Kittelson et al 2002.

4. Summaries and Recommendations for Future Work

4.1 Summary: Nanoparticle Formation and Emissions

Size and concentration of nuclei mode particles

a. The nuclei mode extends through sizes from 3 to 30 nm (0.003-0.03 µm). All of the above size ranges place nuclei mode particles entirely within the nanoparticle range.
b. The maximum concentration of nuclei mode particles occurs at 10-20 nm.
c. The nuclei mode, depending on the engine technology and particle sampling technique, typically contains only 0.1-10% of the total PM mass, but it often includes more than 90% of the total particle count. Sometimes the nuclei mode particles present as much as 99% of the total number of particles.

Chemical Properties:

a. The nature of nuclei mode particles is still being studied in laboratories.
b. Nuclei-mode particles and accumulation mode particles are externally mixed across a wide size range, with the chemical components being distributed between two particle types: “less volatile” particles, probably comprised of an elemental carbon core with a small organic component; and “more volatile” particles.
c. The volatility of the Diesel nanoparticles was found to resemble that of C24-C32 normal alkanes, which implies a significant contribution of lubricating oil to these particles.
d. The organic component of total Diesel particles and nuclei mode particles appears to be comprised predominantly of unburned lubricating oil, whereas the contribution of fuel to the total organic component appears to be relatively small, no more than 20 % and probably much less.

What influences the nuclei mode particles:

a. The nuclei mode is much more sensitive to engine operation, dilution and sampling conditions than is the accumulation mode.
b. Cold temperatures favoured nuclei mode formation.
c. The formation of nanoparticles from particle precursors is influenced by the residence time in the dilution tunnel or exhaust system. Short residence time in the exhaust and sampling system prior to dilution favour nanoparticle formation, while short residence time in the dilution system suppresses nanoparticle growth.
d. Storage and release of volatile material in the exhaust system, and prior engine operating history influence the formation of nuclei mode particles.

Control and mitigation:

a. Engine technology effects were observed to be larger than fuel effects for accumulation mode particles, which reflected the observations for particulate mass. Fuel effects were observed to be larger than engine technology effects for nucleation mode particles, which was reflected in particulate number.
b. Diesel particulate filters can effectively remove accumulation mode (solid) particles from the exhaust, but can emit volatile precursors that lead to nanoparticle formation and a large nuclei mode under high load conditions.

Summary: Influence of the fuel sulphur level on nanoparticle formation

a. Sulphuric acid nanoparticles form as a result of condensation of hydrated sulphuric acid. They are formed from gaseous precursors as temperature decreases in the exhaust system, and after mixing with cold air, be it in the laboratory dilution tunnel or in the ambient air. The diameter of the original nucleus is believed to be about 1 nm.
b. Fuel sulphur enhances nucleation but is not the major component of the nuclei mode. The C24-C32 normal alkanes, from the lubricating oil, have a more significant contribution to these particles Nanoparticles are more easily formed when fuels with high sulphur content (500ppm and above) are used
c. It has been observed that in some engines particle number emissions with low sulphur fuels (below 50ppm) can be up to 100 times lower than with higher sulphur fuels (500ppm). For these engines the reduction in particle mass emission was negligible.

d. The reduction of particle number emissions with reduced fuel sulphur content is greater in engines that emit a smaller concentration of accumulation mode particles, smaller mass emissions (new technology vehicles or vehicles with DPFs).
e. The reduction in particle number emission with the reduction of sulphur level will not show any statistically significant change as the vehicles reach an age of 8 years.

4.2 Recommendations for Future Investigations

All of the studies except one (Ristovski, 2002) examined only a few vehicles/engines from a limited fleet with most of the engines of a newer design. New engine designs and after treatment technologies may present new particle production challenges and solutions. In order to assess the magnitude of the problem, a more extensive investigation should be designed to better represent the current and future fleets.

The reduction of fuel sulphur level is very often accompanied by a significant change in other fuel properties such as aromatic content and volatility. In many of the studies so far these parameters were not decoupled. The specific influence of fuel and lubricants should be studied by testing matrices where key parameters of interest, such as sulphur, volatility and aromatic content are decoupled. The effect of not only fuel sulphur content but also lubricant sulphur content should be studied to determine the influence of this parameter on the formation and emissions of nanoparticles.

Further work is required to develop sampling and measurement standards for particle size and number so that comparable data sets can be produced. For this purpose assessment and adoption of the existing instruments and techniques should be conducted.

4.3 Recommendations on Management Response

Since sulphates are just one of several components of the particulate mass (PM) emissions, lowering fuel sulphur levels has only limited potential as a means of PM control. The reduction of diesel fuel sulphur levels from 3000 ppm to 500 ppm, as legislated in the U.S. in 1994, yielded relatively large benefits of about 0.04-0.08 g/bhp-hr PM reduction. However, a further reduction of fuel sulphur from the 500 ppm to lower levels has only small incremental PM reduction benefit of about 0.008-0.016 g/bhp-hr. The main benefit in reducing sulphur levels further below 500 ppm towards 50 ppm and lower will be in the reduction in particle number emissions. This reduction will be in the number of particles emitted in the nanoparticle range. Further to achieve EUROIV and even EUROIII standards of emissions new diesel emission control technologies have to be implemented. The influence of the sulphur level on the emission of nanoparticles with after treatment devices is still unknown.

Previous studies have shown that the reduction of nanoparticle emission with the reduction of fuel sulphur level below 500 ppm depends on the age/mileage of the vehicle. In order to assess the scale of the problem on the whole Australian diesel fleet, more data is needed on the dependence of the reduction of nanoparticle emission as a function of age/mileage of the vehicles. The available scientific data, which is from a single study, cannot give us this information as that study has been conducted on only one type of vehicle present in the diesel fleet (buses).

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