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Particle Number Emissions During Regeneration of DPF-equipped Light Duty Diesel Vehicles

A Literature Survey

Athanasios Mamakos, Giorgio Martini

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Contact information
Giorgio Martini
Address: Via Fermi, 2749 – 21027 – Ispra (Va) Italy
E-mail: giorgio.martini@jrc.ec.europa.eu
Tel.: +39 0332 789293
Fax: +39 0332 785236

http://ie.jrc.ec.europa.eu/
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1 INTRODUCTION

Diesel Particulate Filters (DPF) have become an indispensable component of diesel light duty exhaust aftertreatment systems, following the introduction of a particle number standard at Euro 5 stage. The operation principle of DPF systems is based on the separation of the airborne particles from the gas stream by deposition on a collecting surface. As soot accumulates onto the DPF there is a need for a periodic regeneration (i.e. consumption of the accumulated soot) in order to avoid clogging of the DPF or uncontrolled oxidation of the soot that can potentially damage the DPF. Combustion of soot with oxygen occurs at a temperature of about 650°C which is rarely achieved in the exhaust of the diesel vehicles. The combustion temperature can be reduced however through the use of a catalyst, by means of either coating the DPF walls or directly adding it in the fuel. Alternatively or in addition, the oxidation rate of soot can be increased, and therefore the regeneration temperature can be decreased, by production of nitrogen dioxide (NO_2) which is a more active oxidant than oxygen.

Under certain operating conditions, the exhaust gas temperature can exceed the temperature required for the oxidation of the accumulated soot, that depends on the aftertreatment configuration employed and the soot loading of the DPF, at which point regeneration of the DPF occurs. This type of regeneration is referred to as passive regeneration since it occurs naturally without requiring any kind of interference from the Emissions Control System (ECS). In practise however, the exhaust gas temperature is too low to sustain regeneration of the DPF under all operating conditions.

In order to ensure that the DPF will regenerate regularly and, most importantly, avoid excessive soot accumulation and stochastic regeneration, the ECS continuously monitors the pressure drop across the DPF which is indicative of soot loading. If deemed necessary, the ECS can also trigger regeneration (active regeneration) in order to increase the exhaust gas temperature to levels that would initiate and sustain regeneration. This is achieved by means of generating an exotherm through delayed fuel injection in the cylinder or even a post-injection of fuel in the exhaust manifold in order to increase the exhaust gas temperature to levels that would initiate and sustain regeneration. Additional or complementary engine measures to trigger regeneration include Exhaust Gas Recirculation (EGR) shut-off and throttling of the exhaust.

A side effect of DPF regeneration is the increased emission of gaseous pollutants and particulate matter. The contribution of such elevated emissions occurring during regeneration events to the total emitted quantity of pollutants during the lifetime of the vehicle will depend on the frequency of such regeneration events. Accordingly, the European regulations classify the regenerating aftertreatment devices (including DPF systems) to continuous or periodic depending on whether the regeneration occurs in each repetition of the legislated test cycle or not. Vehicles equipped with continuously regenerating DPF systems do not require a special test procedure. On the other hand, vehicles equipped with DPF systems that regenerate on a periodic basis need to be tested under both conditions (i.e. with and without regeneration events occurring over the test cycle) and the emission levels are determined by means of weighting the two different emission levels according to the frequency of regeneration events. Currently the procedure applies only to gaseous pollutants and PM but not for the recently introduced particle number emissions.

There is limited information available in the literature on the particle number emissions during regeneration of DPF systems employed in light duty vehicles [1, 2, 3, 4, 5, 6, 7, 8, 9, 10]. The situation is further complicated by the fact that different experimental setups were employed

in the different studies, thus hindering a direct comparison of the results. In the present study an attempt will be made to consolidate the data available from the aforementioned publications in a consistent way in order to get some better insight on the nature of particulate emissions during DPF regeneration.

2 LITERATURE REVIEW

2.1 Size distribution of the total emitted particle population

All studies indicate that the regeneration of the DPF results in a significant increase of the total number of particles emitted. Bergmann et al. [8] measured a 2 litre turbo-charged common-rail direct injection diesel vehicle equipped with a catalyzed Silicon Carbide (SiC) wall-flow DPF. The vehicle was running on a Euro 4 certified diesel fuel (<10 ppm sulphur). Their experiments included both real-world chasing of the vehicle on a high-speed test track but also measurements on a chassis dynamometer. Regeneration was investigated at constant speed of 100 km/h (with the 6^{th} gear engaged). The size distribution of the emitted particles was measured in real time with a TSI's Engine Exhaust Particle Sizer (EEPS), which during the lab experiments was sampling directly from the tailpipe using a dilution system designed to mimic ambient dilution (dilution ratio ~ 1600). Both tests revealed a formation of a distinct nucleation mode peaking at approximately 10 nm. This nucleation mode was almost 3 orders of magnitude larger than the background distribution. The latter was similar to that measured during non-regenerating conditions, being lognormal in shape with a peak at a much larger size (60-100 nm). The good agreement between the laboratory and the real-world chasing experiments suggest that these particles are produced by the DPF system and are not an artefact associated with desorption of volatile and semivolatile material deposited on the sampling/dilution system at the elevated exhaust temperatures occurring during regeneration.

Campbell et al. [4] tested a 2.2 litre Peugeot 406 HDi diesel vehicle on a chassis dynamometer under hot start NEDC test cycles. The aftertreatment system of the vehicle consisted of an oxidation catalyst directly upstream of a DPF within the same enclosure that would generate the necessary exotherm for regeneration when post injecting fuel in the engine cylinder. The vehicle also utilized a fuel additive system to reduce the temperatures necessary for the oxidation of soot. A Cambustion fast particulate spectrometer (DMS500), sampling directly form the tailpipe, was employed to measure the size distributions of the total emitted particles. The data inversion software of the DMS500 performs a bimodal lognormal distribution fit to the measured instrument responses, thus allowing for a discrimination between nucleation mode and accumulation mode particles. During the part of the EUDC where regeneration took place, a substantial production of nucleation mode particles was observed peaking at 8 nm reaching concentrations as high as 4×10^{9} #/cm^{3}, when little nucleation mode could be observed over the remaining part of the cycle. The background accumulation mode was peaking at approximately 100 nm and at a much lower concentration (peaking at around 5×10^{6} #/cm^{3} over the ECE part of the cycle).

Giechaskiel et al. [1] measured a Peugeot 407 HDi FAP (Filtre à Particulates). The vehicle was a turbocharged common rail direct injection diesel equipped with a DPF system consisting of an oxidation catalyst upstream of an uncoated SiC wall-flow particulate filter. The vehicle also employed a cerium based fuel borne catalyst to reduce the regeneration temperature. Regeneration was investigated over the NEDC test cycle. Size distributions were measured in real time with an EEPS connected to the CVS tunnel. The size distributions recorded during regeneration revealed a nucleation mode peaking in general below 12.5 nm. One notable exception was the start of the first regeneration event during

which a nucleation mode peaking at 35 nm was observed. This event lasted for a few seconds after what a second nucleation mode peaking at a lower size (at 10 nm) was observed. Accordingly the total particle number emission rates over the EUDC part of the cycle were more than 3 orders of magnitude higher compared to those when no regeneration occurred.

The same vehicle was tested by Dwyer et al. [7], who also used an EEPS sampling from the CVS tunnel to monitor the size distribution of the total emitted particles in real time. Regeneration at 120 kph revealed a distinct nucleation mode peaking at ~10 nm increasing the number concentrations by more than 3 orders of magnitude, resulting even in a saturation of the EEPS.

Bikas et al. [9] measured particle emissions during regeneration of an uncoated SiC DPF (2.5 litre) retrofitted on a 1.9 litre Euro 4 diesel engine. The exhaust aftertreatment system also included a commercial oxidation catalyst of 1.6 litre coated with 50 g/ft^3 of Platinum (Pt), installed upstream of the DPF. The engine operated on diesel fuel containing 50 ppm of sulphur (S) but also 30 ppm of Cerium Oxide (CeO_2) to assist regeneration. The regeneration was externally triggered by means of initiating delayed fuel injection in the cylinder. Size distribution measurements were performed with an SMPS but it was not specified where sampling took place. Regeneration of the DPF system at 100 kph steady speed cruising revealed the formation of a nucleation mode superimposed on the background lognormal distributions (peaking at 60 nm). The peak size of this nucleation mode progressively increased during the regeneration event from <10 nm to 40 nm. The concentration of this nucleation mode was 3 orders of magnitude higher compared to the background accumulation mode. After completion of the regeneration event, the nucleation mode gradually decreased in both number and size and was eventually suppressed.

2.2 Non-Volatile particle emissions

Giechaskiel et al. [1] measured non-volatile particle emissions over NEDC test cycles before, during and after regeneration, following the legislated procedure. The particle number concentrations measured (hereinafter PMP-PN) correspond to particles larger than 23 nm in size that survive heated dilution at 150°C followed by heating in an evaporating tube at 300-400°C wall temperature and subsequent dilution with ambient air (that serves to reduce the vapor pressure of particle precursors and the aerosol temperature within the operating temperature of the CPC). The burst of nucleation mode particles observed during regeneration was not observed in the PMP-PN results which remained at background levels. This could be because of the very small size of these nucleation mode particles that could not be detected by the PMP CPC (having a cut-off size of 23 nm). The burst of nucleation mode particles at 35 nm that was observed at the start of the first regeneration test were not detected by the PMP system suggesting that these particles were volatile in nature. The PMP-PN concentrations were at least 3 orders of magnitude lower suggesting a removal efficiency of 99.9%. The average emissions over the NEDC test cycles where regeneration took place were found to be twice as high compared to those before regeneration, but this was due to elevated emissions over the ECE part of the cycle. In that respect, this rather reflects a reduced filtration efficiency of the DPF caused by the consumption of the soot cake. Similar higher emissions were observed in the cycle following the completion of the DPF regeneration.

In the same study [2], a PMP-compliant CPC (i.e. having a cut-off size at 23 nm) was also employed sampling upstream of the evaporating tube of the PMP particle measurement system. The particular CPC also indicated higher number concentrations compared to PMP-PN with the difference exceeding 2.5 orders of magnitude in some cases. This suggests the

presence of semi-volatile particles larger than 23 nm that survive the hot dilution at 150°C. Most importantly, this indicates that the PMP system can effectively evaporate them or at least decrease their size below 23 nm, with an efficiency of better than 99.5%.

Dwyer et al. [7] have also measured the PMP-PN emissions of the same vehicle over NEDC test cycles where regeneration took place, using the same PMP-compliant system. In addition, they have also employed a Grimm CPC having a lower cut-off size (5 nm) sampling in parallel with the PMP CPC downstream the PMP Volatile Particle Remover (VPR). At the same time an EEPS and a PMP compliant CPC (i.e. having a cut-off size at 23 nm) were sampling directly from the CVS tunnel. PMP-PN emissions remained at a very low level which was more than 3 orders of magnitude lower than the total number concentrations of particles larger than 5.6 nm measured with the EEPS. This was also generally the case for the Grimm CPC. However in a particular NEDC repetition a significant increase of the Grimm response was observed that reached almost 3 orders of magnitude (even exceeding the concentrations measured with the EEPS that got saturated at that conditions). The PMP-PN concentrations also exhibited an order of magnitude increase during this short period of time but still remained more than 2 ½ orders of magnitude below the EEPS concentrations. Unfortunately, the PMP compliant CPC that was sampling directly from the CVS got saturated under these conditions so it was not possible to draw any useful information.

Mathis et al. [6] tested a Peugeot 406 HDi/FAP diesel vehicle under the Common Artemis Driving Cycle (CADC) developed in the framework of the ARTEMIS programme [11]. Sampling was conducted using an experimental setup developed in the framework of the PARTICULATES project [12]. The core of the particular setup was a partial flow dilution system that was connected directly to the exhaust and included two branches for the concurrent characterization of the total particle population and the non-volatile fraction of them. Non-volatile particles were monitored with an Electrical Low Pressure Impactor (ELPI) and a TSI's 3022 CPC (having a cut-off size at 7 nm) connected downstream of a thermodenuder operating at 250°C. A 3022 CPC was also employed to measure in parallel the number concentration of the total particle population (i.e. in the other branch of the sampling system). Over the motorway part of the CADC, a significant increase of the total particle number emissions was observed, however no information is given on the absolute emission levels. The CPC downstream of the thermodenuder also measured elevated emissions which were at about 35% of the total particle number emissions. Interestingly, the solid number concentrations measured with the ELPI, which had a lower cut-off size at 32 nm (aerodynamic diameter) in the configuration employed, were about 200 times lower suggesting that these were sub-30 nm particles.

Anderson [10] measured a Euro 4 certified DPF-equipped diesel vehicle. The emissions control system employed a platinum coated cordierite wall-flow filter and used post-injection, exhaust throttling at light load and EGR shut-off to generate an exotherm and periodically regenerate the DPF. Non-volatile particle number emissions were measured using a PMP-like system that utilized a TSI's 3022 CPC sampling through diffusion screens to shift the cut-off size from 7 nm to 23 nm. This modified 3022 CPC however, had a relatively flat detection efficiency curve (e.g. 10% and 90% detection efficiencies occurred at 11 nm and 110 nm compared to 16 nm and 37 nm of PMP-compliant CPCs). An unmodified 3022 CPC was also employed sampling directly from the CVS tunnel, which during steady state testing was connected in an SMPS system to monitor the number weighted mobility size distributions. A comparison of the indications of the two CPCs during regeneration of the DPF over a hot start of the NEDC revealed a difference of almost 1 ½ orders of magnitude.

Mohr et al. [3] measured particle number emissions during DPF regeneration of three DPF-equipped diesel vehicles. They employed two TSI's 3022 CPCs (cut-off size at 7 nm) one of

them was sampling from the CVS tunnel through an ejector diluter and another one downstream of an evaporating tube connected to the same ejector diluter. This approach, however, is vulnerable to volatile artefacts since no precautions were taken to decrease the vapor pressure of evaporated material downstream the evaporation tube. The evaporation of volatile material from all particle sizes in conjunction to the low soot content of the particulate emissions of DPF equipped vehicles could potentially result in nucleation of such gaseous precursors downstream the evaporating tube. Another concern regarding the particular measurement pertains to the temperature of the carrier gas downstream the evaporation tube which was fed directly to one of the CPCs.

One of the vehicles tested by Mohr et al. [3] was a 2-litre VW Passat TDI that utilized an uncoated SiC DPF close coupled with an oxidation catalyst and employed fuel borne catalyst to reduce the regeneration temperature. In order to initiate regeneration of the particular DPF, the vehicle was run at 80 km/h at high engine load. During the regeneration process, the particle number concentration measured without the evaporation tube increased by more than one order of magnitude, whereas the concentrations downstream the ET remained fairly constant. One other vehicle tested was an 1.9 litre Opel Vectra CDTI that utilized a catalyzed SiC DPF close coupled with an oxidation catalyst. In order to initiate regeneration, the vehicle was run at 80 km/h at full load. Following the start of the regeneration, the total particle number emissions increase by three orders of magnitude and interestingly shortly after the concentrations downstream the ET also reached these levels. This was attributed by the authors to re-nucleation of the volatile material downstream of the evaporation tube. The last vehicle tested was a 2-litre Toyota Avensis D-Cat that employed a combined NOx adsorber and DPF system (D-cat) located downstream of an oxidation catalyst. The particular DPF system had a relatively high porosity as evident by the elevated particle number emissions (by more than one order of magnitude) compared to the other DPF-equipped vehicles tested. To initiate regeneration, the vehicle was tested at 120 km/h. The regeneration events with that particular vehicle, which were shorter in duration and more frequent, resulted in a ten-fold increase of the concentrations of both CPCs.

2.3 Nature of the nucleation mode particles emitted during regeneration

To our knowledge, there is no information available on the chemical composition of the nucleation mode particles emitted during regeneration. Thermal treatment of these particles resulted in a significant reduction of their number concentration [1, 2, 7, 10] an indication that these are volatile or semi-volatile in nature. Furthermore the number concentrations of these nanoparticles were not found to correlate with the measured total hydrocarbons following the legislated procedure [3, 4]. To a certain extent this might be due to the high volatility range analyzed by Flame Ionization Detectors (FID) that misses most of the condensable species [13].

Analysis of PM samples collected during DPF regenerations [4] revealed high concentration of soluble sulphates and sulphate-bound water. In that particular study, the regeneration of the DPF system was completed after two consecutive hot start NEDC cycles with the second one yielding significantly lower number concentrations and sulphate emissions. Sulphur can originate from the fuel and the lubricating oil and can be trapped in the DPF. At the elevated temperatures during regeneration the sulphur can be released and oxidized to SO_3 thus leading to the formation of sulphate particles. Bergmann et al [8] also found the concentrations of nucleation mode particles to correlate with the sulphate content of collected PM filters.

The importance of sulphur content of the diesel fuel on the particle emissions during regeneration was highlighted in the study of Guo et al [5]. They tested a 2.5-litre York diesel

engine that was retrofitted with an aftertreatment system consisting of two Active Lean NOx catalysts (400 cpsi cordierite, 70 g/ft^3 Pt, 6'' long × 5.66'') followed by a cordierite catalyzed DPF (200 cpsi, 50 g/ft3 Pt, 6'' long × 5.66''). In order to regenerate the DPF, fuel was injected upstream of the two active lean NOx catalysts through an air-assisted injector (at 5 psi). The engine operated on two diesel fuels of different sulphur content (<4 ppm and 340 ppm). Particle emissions were measured with an SMPS sampling downstream of a dual ejector system connected to the tailpipe. The first diluter was heated at 200°C and supplied with nitrogen at the same temperature while the second operated on ambient nitrogen. No nucleation mode was observed during the regeneration of the DPF when low-sulphur fuel was employed. On the other hand, use of 340 ppm S fuel resulted in a distinct nucleation mode forming reaching concentrations that even exceeded pre-DPF levels (more than 3 orders of magnitude above the post-DPF levels during normal engine operation).

Current fuel specifications in Europe and US have a much lower sulphur content. Still though, sulphur can be stored onto the soot trapped in the DPF and the walls of the DPF and subsequently released during regeneration. This assumption is in line with the general observation that the particle emissions decrease as the soot trapped in the DPF is consumed [4, 8].

Anderson [10] have also tested a DPF-equipped vehicle using both synthetic and mineral lubrication oils, with the former having a much less quantity of sulphur and sulphate ash (0.7% and 1.3% compared to 0.2% and 0.5%, respectively). Two diesel fuels of very low sulphur content (3 ppm and 10 ppm) were also employed. PMP-PN emissions during regeneration where similar for all fuels and lubricants tested, with individual differences lying within the experimental uncertainties of the measurements. PM emissions over hot start NEDC cycles where the DPF regenerated were 50% higher when mineral oil was employed. Unfortunately, no information is available on the effect of fuel and lubricant on the total number population of the particles emitted during regeneration.

The sulphate nature of the nanoparticles produced during regeneration raise some concerns regarding potential formation of non-volatile particles in the evaporation tube. In a recent study, Swanson et al. [14] observed some solid particles forming downstream a thermodenuder that they attributed to charring or pyrolysis reactions of hydrocarbons that are catalyzed by sulphuric acid. Such artefacts were not observed when a catalytic stripper was employed, that utilized a sulphate trap to remove sulphates before the heated oxidation catalyst. It is not clear to what extend such phenomena can also occur in PMP systems. It is interesting though that the observed artificial non-volatile particles had a very small size that would not be detected with a PMP particle number measurement system.

2.4 Emissions during passive regeneration

Most of the work available in the literature investigated particle emissions during active regeneration events. Giechaskiel et al. [1] observed passive regeneration of the DPF examined under steady speed cruising at 140 kph. Under these conditions, the total number concentration of particles larger than 23 nm (as measured upstream of the evaporation tube) increased by approximately 475 times when the PMP-PN concentrations increased moderately (about 2.5 times). Anderson [10] has also observed some passive regeneration occurring at 120 kph, following an active regeneration event. PMP PN emissions were found to be at the same or slightly higher levels to those under active regeneration. This slightly increase of PMP-PN concentrations could be associated with the reduced filtration efficiency of the DPF due to consumption of the soot cake.

3 Conclusions

This study overviewed published data on particle number emissions during regeneration in an attempt to assess the PMP methodology under these operating conditions. During regeneration of the DPF a significant increase of the emitted number of particles is observed. Size spectra reveal a nucleation mode peaking at approximately 10 nm, at concentrations that can exceed the emission levels under non-regenerating conditions by more than three orders of magnitude. These nano-sized particles are found to be mostly volatile in nature with their concentrations correlating with the sulphate content of the emitted particulate matter. Particle number measurements following the PMP methodology are found to be little affected by this burst of nucleation mode particles, partly because of the large cut-off size of the CPC (23 nm) and partly because of the semi-volatile nature of the emitted nanoparticles, exhibiting more than 97% overall removal efficiency. A high concentration of sub-23 nm particles downstream of a PMP VPR system was observed in some cases which might be semi-volatile particles that do not evaporate completely in the VPR or even an artefact resulting from pyrolysis of volatile hydrocarbons inside the evaporating tube of the VPR. Even under these conditions however, the PMP-PN results were not or moderately affected with the measured concentrations being at least two orders of magnitude lower. In fact, the highest recorded PMP-PN concentration during DPF regeneration occurring over the EUDC were more two orders of magnitude lower than that recorded over the cold start ECE part and therefore had insignificant affect on the particle number emission rate over the NEDC. The latter was mostly affected by the fill status of the DPF, and was found to be at the same levels with test cycles performed immediately after a regeneration event.

4 LIST OF SPECIAL TERMS AND ABBREVIATIONS

CADC	Common Artemis Driving Cycles
CeO_2	Cerium Oxide
CPC	Condensation Particle Counter
CVS	Constant Volume Sampling
DMS500	Cambustion's Fast Particulate Spectrometer
DPF	Diesel Particulate Filter
ECE	Urban Driving Cycle (Part 1 of the NEDC driving cycle)
ECS	Emissions Control System
EEPS	Engine Exhaust Particle Sizer
EGR	Exhaust Gas Recirculation
ELPI	Electrical Low Pressure Impactor
EUDC	Extra Urban Driving Cycle (Part 2 of the NEDC driving cycle)
Euro #	European Emission Standard
FAP	Filtre à Particulates
FID	Flame Ionization Detector
HC	Hydrocarbon
JRC	Joint Research Centre
N_2	Nitrogen
NEDC	New European Driving Cycle
NO_2	Nitrogen Dioxide
PMP	Particle Measurement Programme
PN	Particle Number
Pt	Platinum
S	Sulphur
SiC	Silicon Carbide
VPR	Volatile Particle Remover

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Abstract
The study reviews published work on the particle emissions during regeneration of diesel particulate filters in an attempt to assess the PMP methodology under these operating conditions. During regeneration of the DPF a significant increase of the emitted number of particles is observed. Size spectra reveal a nucleation mode peaking at approximately 10 nm, at concentrations that can exceed the emission levels under non-regenerating conditions by more than three orders of magnitude. These nano-sized particles are found to be mostly volatile in nature with their concentrations correlating with the sulphate content of the emitted particulate matter. Particle number measurements following the PMP methodology are found to be little affected by this burst of nucleation mode particles, partly because of the large cut-off size of the CPC (23 nm) and partly because of the semi-volatile nature of the emitted nanoparticles, exhibiting more than 97% overall removal efficiency.

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