GAS AND PARTICLE SIZE CHARACTERIZATIONS OF A SOOT GENERATOR FOR FOULING STUDIES IN EGR SYSTEMS

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ABSTRACT

More new stringent regulations have made fouling one of the major problems affecting the design of EGR systems since their performance must be warranted over the life time of the vehicle. Soot deposition and hydrocarbon condensation are mainly responsible for fouling of EGR cooler in diesel engines, highly dependent on the engines operating conditions.

In this work, a study was carried out in an experimental setup designed and assembled for analysing the fouling process in the technologies commonly employed in EGR coolers. A soot generator, mini-CAST burner, has been used to reproduce the conditions particles emitted from diesel engines. This equipment allows to control the size and concentration of soot particles in a controlled environment, avoiding the influence of other variables such as lubricants, fuel composition, etc. Six calibrated points were obtained, changing the combustion conditions, and they were characterized. The fouling layer is deposited on a sample collection surface located in the test section, that is provided with the precise instrumentation (thermocouples and pressure sensors) required. The soot particles diameters found a range from 10nm to 650nm while soot particle concentration varies between 0 to 5E+08 part/cm³, both according to typical values of current diesel engines under EGR conditions.

For each point, the exhaust gas composition was determined with an AVL DITEST 1000 gas analyser while a TEXA OPABOX opacimeter was used for the determination of the opacity values. A scanning mobility particle sizer (SMPS) was employed for the measurement of the different particle size distribution generated, the exhaust gas being previously diluted in two stages at different temperatures. In addition, Scanning Electron Microscopy (SEM) and TGA-DSC techniques were carried out to obtain information about morphology and composition of the fouling layer in each experimental point.

1. INTRODUCTION

Hundreds of industrial applications require a heat exchange process, where the fouling process appears in different intensity. Definitively, this process of fouling involves higher costs of operation, and can even be catastrophic for the operation of the system [6]. The fouling in exhaust gas systems in absence of hydrocarbons condensation is usually associated with particle deposition. It has been proved by many authors [9; 10; 21] that the main consequences of fouling are the reducing of thermal efficiency and increasing of pressure losses in all technologies employed.

For the study of this phenomenon there are currently three main ways: The theoretical model in which many authors such as Grillot et al. [8] and Talbot et al. [20], which explains the particle deposition and the modification of the thermal conductivity due to the fouling process. Other way is the CFD (Computational fluid dynamics) tools. The analyses of the phenomenon of fouling that are carried out today focus their bases in creating tools for its estimation and in the study of the formation mechanisms that predict how it originates and evolves, the layer of deposited particles. Over the years, researchers have presented different models of fouling in order to reproduce and explain this phenomenon. Although most of them have made great progress in this area and exposed the factors that produce it, none of the models developed has achieved accurately predictions and has explained what actually happens inside the heat exchangers. Authors such as Abarham et al. [1; 2] developed a model to predict soot deposition in a concentric EGR and with a hydrocarbon condensation, Paz et al. [18] proposed a model in order to study particle deposition in diluted turbulent gas flows in ducts. Both ways commented need the experimental part, which has been the most studied due to technological limitations made it the only possible way possible of acquiring knowledge about the fouling. Many researchers have developed test bench for studying this phenomenon [3; 11; 16; 19]. They focused their research in two possible modes of particle generation, using diesel engines and aerosol capable to reproduce the exhaust gas emitted by the first. Although use of particle generator allows more control avoiding the influence of variables such as oils, fuel compositions, etc. There is a general agreement on the method of measuring the particle, size and concentration, in that the scanning mobility particles sizer seems to be the more accurate method.

In this research, a test bench was developed to reproduce the exhaust gas of the current diesel engines. Six points have been studied and characterized using a SMPS to get the size and concentration of particles, as well as different techniques such as TGA-DSC and SEM.

Nomenclature

EGR	Exhaust Gas Recirculation
SMPS	Scanning Mobility Particle Sizer
EC	Elemental Carbon
OC	Organic Carbon
FSN	Filter Smoke Number
HC	Hydrocarbons
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis

2. EXPERIMENTAL SETUP

2.1. Test Bench and measure equipment

For the realization of this study, an experimental test bench was designed which is able to reproduce the exhaust gases of current diesel engines. The test bench is represented in Figure 1. The most significant parts: (1) Soot Generator, (2,3) Heating Zone, (4) Test section, (5) Gas analyzer, (6) Opacimeter, (7) Diluter and (8) Scanning Mobility Particle Sizer

For the construction of this test bench, a soot generator has been used (1) (miniCAST 5203 Type C) in order to work under controlled conditions, avoiding the influence of other variables such as lubricants, fuel composition, etc. ensuring repeatability and accuracy. The operating principle of the miniCAST 5203 Type C [12; 13] is shown in Figure 2. In order to generate the intended soot particle load, the coating air supply is kept insufficient. As a consequence, a particulate flow that is surrounded by the exhaust gases of the flame arises out of the flame and leaves the combustion chamber. In the next step the particle stream is mixed with the quenching gas in order to prevent further combustion processes in it and to stabilize the soot particles. The quenching gas inhibits condensation in the particle stream when it escapes from the flame unit. Subsequently, air is supplied to dilute the particle stream. The soot generator has a total exhaust gas flow rate of 200 l/min and a maximum mass particle output of about 1.5 g/h. The mean particle size can be adjusted in the range of 20-200nm (mean mobility diameter) with a particle concentration up to 10^8 part/cm³. The control of the particle diameter and concentration is carried out by modifying the combustion conditions. The maximum flow of the working gases are: propane (up to 0.2Nlpm), air (up to 5Nlpm), nitrogen -as quenching-gas with a constant flow rate of 10Nlpm and as mixing gas (up to 0.5Nlpm) and finally, air (up to 200Nlpm) can also be used as dilution gas.

The gas temperature at the outlet of the soot generator varies between 80 and 140°C. Therefore, it was necessary to heat the gas to reach normal temperatures in EGR systems, between 250°C and 800°C. A ceramic furnace (3) and a heating cable (2) with a total power of 4.5kW have been chosen to heat the pipe by conduction and radiation. This method of heating was chosen so as not to interfere with the flow and thus avoids changes by contact with any heating system in the emitted particles.

For measurement of particles upstream of the sampling area a Scanning Mobility Particle Sizer (SMPS, model 3936L75-N, TSI) was employed. This device allows the measurement of wide ranges of particle sizes, 10-1000nm, and concentration, up to 10^7 particles/cm³ respectively. The gaseous stream employed in this system has two features that make difficult to measure by the SMPS. On the one hand, its high temperature, much higher than the maximum temperature supported by the measuring equipment (35°C), and secondly, an elevated concentration of particles also exceeding the maximum range of the particle analyzer. Therefore, a dilution of the gas stream generated by the test bench was employed. The dilution system (7) (PALAS PMPD 100) dilutes a portion of the exhaust gas stream with a known flow of clean air. It is essential to perform this dilution carefully, since it is not only necessary to achieve a reduction of the temperature and the concentration of particles, it is also necessary to do it with a controlled dilution ratio, and temperature evolution. The process dilution was carried out in two stages, each one with a dilution ratio of around 1:10, the first dilution occurs at a relatively high temperature, with an outlet temperature of around 150°C to avoid hydrocarbon condensation and prevent nucleation. Once the HC concentration is suitably lowered by the effect of the first dilution, a second dilution using ambient temperature gas is used to achieve a non-condensing, cool, and suitably dilute condition to allow for accurate particle measurements.

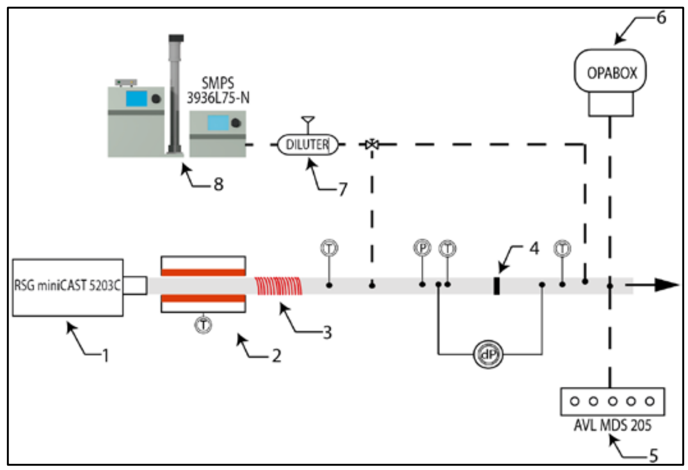

Figure 1 Fouling Test Bench

To characterize the emited stream gas generated by the miniCAST, a gas analyzer (DITEST GAS 1000 MDS 205, AVL) has been used for the gas composition and an opacimeter (OPABOX autopower, TEXA) to obtain the FSN.

In addition, the test bench was provided with various thermocouples (k-type, class-2, 1.5mm diameter), which allow the precise control of the temperature throughout the entire path of the gas stream. These thermocouples are placed forming an L, with the tip of the thermocouple towards the flow.

A cooling circuit of deionized water regulates the coolant temperature up to 95°C, allowing regulation of pressures between 1-3bar and flow rates in a range of 100-2500l/h. Although this circuit was not employed in the present research, it will be essential for further tests with EGR- coolers.

Data acquisition is comprised of an acquisition card (9) (NI USB-6363, National Instruments) acquiring a frequency of 1 kHz, and filtering the signal with a frequency of 2 Hz low pass.

2.2. Test section

The geometry shown in Figure 3 was designed for this research in order to be fouled for subsequent studies of the deposited layer, allowing gas mixture flow at the same time. This geometry consists of a circular section, made of stainless steel AISI 304, with a diameter of 50mm and a thickness of 10mm. In this section, six holes were made with a diameter of 2mm representing a 3x3 square matrix.

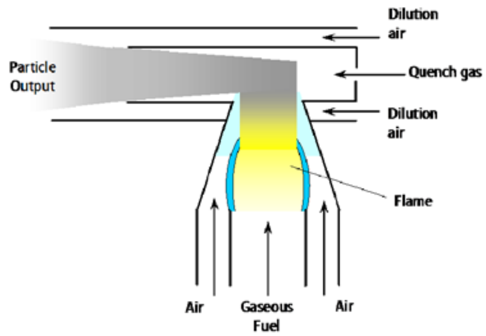

Figure 2 Operating Principle described *Instruction Manual of Real Soot Generator Model 5203 Type C miniCAST* [13]

Figure 3 Fouled Geometry

The test section in which sample geometry collected was mounted, is schematically shown in Figure 4. K-type thermocouples have been located at different positions upstream (1, 3) and downstream (5) of the fouled surface (8). Moreover, a turbulator (2) was placed upstream of the sample collection zone to homogenize the gas temperature profile preventing the getting gas is hotter at the pipe wall, externally heated, than in the center of it.

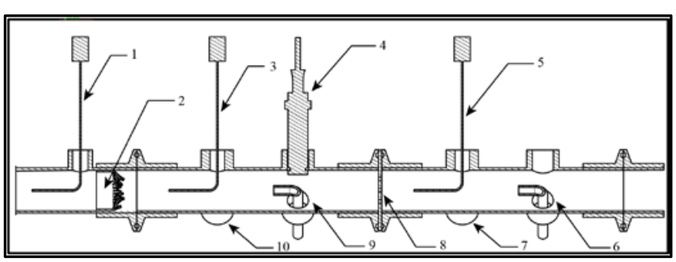

Figure 4 Test section

Furthermore, differential pressure (SITRANS P500, Siemens) was measured between points 10 and 7, upstream and downstream of the sample collection surface and absolute pressure sensor (OPTIBAR 1010C, Khrone) was placed in (4). Finally, (6) and (9) correspond to the measuring probes employed for the sampling of SMPS, which were designed to behave isokinetically in order to prevent changes in the flow.

3. EXPERIMENTAL PROCEDURE

A total of six points of miniCAST combining different flow rates of different gases have been used in this work (Table 1). These points are close to the calibration points provided by the manufacturer, and were chosen for generating particles of similar size and concentration to those found in an EGR loop of a current diesel engine under typical operating conditions.

Point	C_3H_8 (Nlpm)	Mixing N_2 (Nlpm)	Oxidation Air (Nlpm)	Dilution Air (Nlpm)	Quench gas N2 (Nlpm)
#1	0.2	0	4.2	100	10
#2	0.2	0.1	4.1	100	10
#3	0.2	0.3	3.9	100	10
#4	0.2	0.5	3.7	100	10
#5	0.2	0.7	3.5	100	10
#6	0.2	0.8	3.4	100	10

Table 1 Operating Conditions

All points were analyzed under the same conditions, a gas inlet temperature of 250 °C and a mass flow rate of 8.87kg/h, varying only N_2 gas mixing and oxidation Air.

The test begins with a differential pressure of 80mbar, and is stopped collecting soot when it reached a differential pressure of 100mbar, with an average test duration of about five hours. In order to ensure the experiments reproducibility, each test was carried out five times.

As discussed in section 2.2, sampling probes in the test bench have been provided, upstream and downstream of the tested geometry, to allow the analysis of SMPS. In this article we have only studied the particles that were collected, so that the sampling probe downstream in the test area was not employed. The interpretation of measurements by the SMPS is made by the software Aerosol Instrument Manager (A.I.M.v9.0).

Mathematical corrections have been applied as recommended by the manufacturer, the following corrections are selectable in software: Multiple Charge Correction, Diffusion Correction and Nanoparticle Aggregate Mobility Analysis.

1. The particles increase their mobility due to multiple charges. By default, the program assumes that the particle has an only one charge, the Multiple Charge Correction allows properly grouping particle into a smaller-sized particle channel. The effects of multiple charged particles are most pronounced for particles above approximately 100nm, so it was necessary to apply this correction in this case [22].
2. It was necessary to correct the measurement due to diffusion losses in the internal flow path through SMPS, these losses are significant for particles whose diameter is less than 100nm. Due to the fact, that the studied points have a modal diameter smaller than 100nm, the Diffusion Correction was applied [23].

3. By default, the software assumes that all analyzed particles are spherical. Nevertheless, in the process through particles are generated from combustion sources, such as in the case of miniCAST, this assumption is not correct. The particles are composed of aggregated primary particles, which always have the same diameter, usually with diameters from 5 to 50nm. The Nanoparticle Aggregate Mobility Analysis correction allows to have a more accurate estimation of the number, surface area and volume distributions.

In this study the first two corrections have been applied due the lack of accurate information to apply the third correction, for which, it would be necessary to obtain primary diameters by Transmission Electron Microscopy (TEM).

Measurements made with the SMPS as well as those made with the opacimeter and gas analyzer were performed in all cases at a differential pressure of 90mbar.

Afterwards, the sample bags were filled with the collected sample using a small spoon, by scraping the tested geometry.

Then, they were employed in the realization of the thermogravimetric analysis (TGA). For this purpose, the equipment SETARAM SETSYS Evolution TGA-DTA/DSC was employed. The TGA method enables analysis of the continuous weight loss of a sample at a controlled heating rate in an inert and/or oxidant atmosphere. It was decided as a method to perform the TGA after an extensive bibliographic review: Lapuerta et al. [14], Paredes et al. [17] and Forcada [7]. A heating rate of 10°C/min from 20°C to 800°C and then keeping at 800°C for 15 minutes under an inert atmosphere of N2, this first phase of TGA eliminates the amount of volatile organic fraction (water between 100°C-150°C, light hydrocarbons between 150°C-350°C and heavy hydrocarbons up to 800 ° C). Later, it was changed to an oxidant atmosphere, also at 800°C and it was kept for 30min, this allows the amount of elemental carbon.

With the remaining sample not used in carrying out the TGA a Scanning electron microscope (SEM) was performed in the microscope JEOL JSM6700F. This technique through an electron beam forms an image of high resolution enabling high magnification. This analysis provides information on the morphology and composition of the different samples.

Moreover, it was performed a 3D analysis with a S neox 3D Optical Profiler (SENSOFAR) (Figure 10).

4. RESULTS AND DISCUSSION

As it was explained in section 3, repeatibility experiments were carried out in all cases. One of this experiments is shown in Figure 5, where the five distributions of particle number concentration for point 1 are depicted.

In Figure 6 the distributions obtained by SMSP measures for the different evaluated points are represented. The results of the generating distributions with different gas mixture are shown in Table 2. Whenever the mixing gas flow was increased, smaller mean diameters were obtained, which resulted in an increase of the concentration.

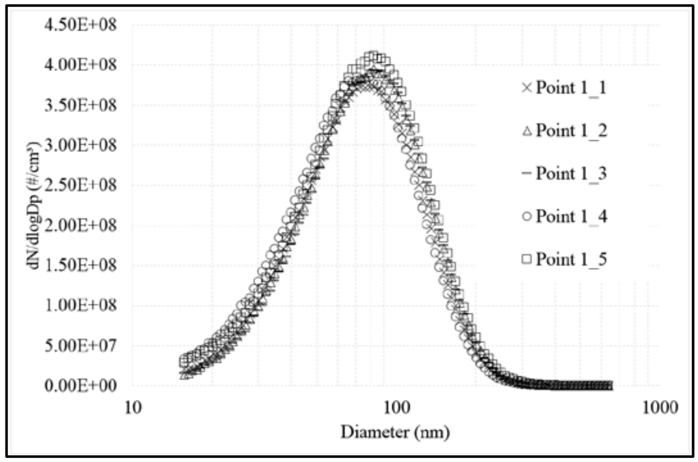

Figure 5 Repeteability of particle analysis about point 1

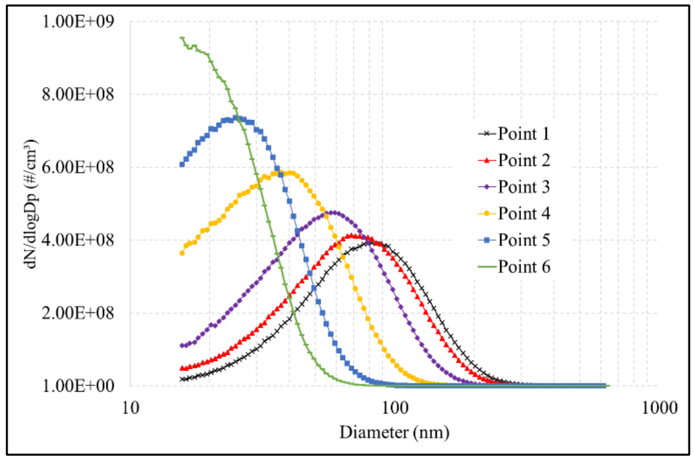

Figure 6 Size distributions of soot particles obtained from miniCAST

At the same time, a reduction of the richness gas mixture, due to a decrease of oxidation air, involves a worse combustion, which in turn results in an increase of the emission of hydrocarbons (HC). It was detected an increase in number of HC (ppm) measured by the gas analyzer. Similarly, as the mean diameter of the particles was reduced, a reduction in opacity was observed as shown in table 3.

Point	Mode (nm)	Total Number Concentration (#/cm³)	Total Mass Concentration (mg/m³)	Standard Deviation
#1	82	2.41E+06	1.7700	1.71
#2	68.5	2.64E+06	1.3500	1.72
#3	59.4	3.02E+06	0.7418	1.70
#4	37.2	3.65E+06	0.2776	1.60
#5	25	3.55E+06	0.0927	1.43
#6	15.7	3.24E+06	0.0450	1.35

Table 2 Evaluated points and properties, dilution ratio 1:100

The filter smoke number (FSN) number has been calculated according to the modified correlation (Eq. 1) by Lapuerta et al.

$$N = 0.0068\,FSN^3 + 1.007\,FSN^2 + 2.5001\,FSN \quad \text{(Eq. 1)}$$

[15] based on a AVL previous proposal [4]. The N opacity data was measured directly by the opacimeter. Note than point 5 and point 6 values are equal because of the accuracy of the opacimeter is less than the difference of light attenuation between them.

Changes were observed in the composition of the sample, increasing the amount of organic carbon when the oxidation air was reduced (Figure 7). The values obtained confirm the influence of the oxidation air demonstrated by Barthazy et al [5].

Finally the SEM of the samples was also performed, as is shown in figures 8 and 9 which correspond to points 1 and 4 respectively. The SEM performed for each point, shown accordance with the diameters of particles measured by SMPS. These figures show the change in the structure of the deposit. Figure 8 shows a dry deposit with small presence of HC, while Figure 9 is a wet sample with a higher presence of HC. The wet deposit is the most similar to the structure that often have the deposits of EGR systems in the current diesel engines.

Point	CO (%vol)	CO_2 (%vol)	O_2 (%vol)	HC (ppm)	N (%)	FSN
#1	0.08	0.33	18.58	0	11.8	2.47
#2	0.09	0.31	18.46	0	9.7	2.13
#3	0.1	0.35	18.45	0	4.7	1.21
#4	0.1	0.28	18.60	15	1.2	0.41
#5	0.1	0.29	18.57	31	0.2	0.15
#6	0.09	0.26	19.61	33	0.2	0.15

Table 3 Gas analyzer and opacimeter results

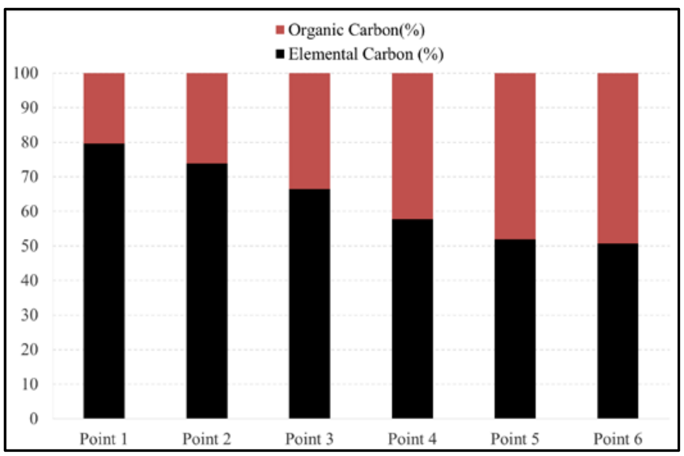

Figure 7 Thermogravimetric analysis results for each evaluated point.

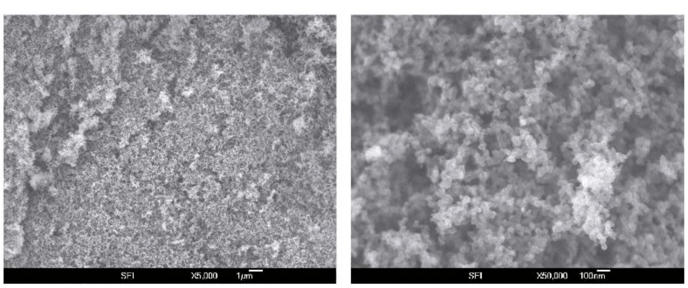

Figure 8 Scanning Electron Microscope of sample point 1

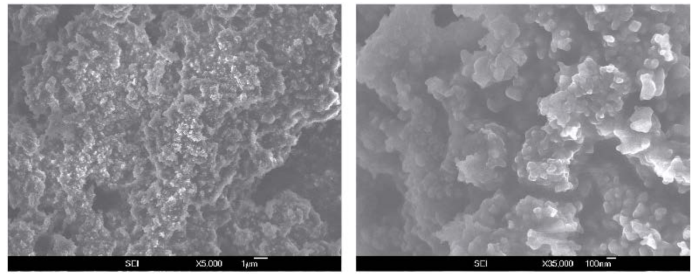

Figure 9 Scanning Electron Microscope of sample point 4

The extracted profile for Point 1 is shown in Figure 10. For this point, an average deposition height obtained was 0.2mm and a maximum height of 0.7mm. A homogeneous average profile is observed due to the influence of the effect of turbulators used for homogenizing the temperature at the inlet of the perforated plate. It is true that in some areas peaks are observed, which could be due to the difficulty in placing the perforated plate perfectly perpendicular to the gas flow.

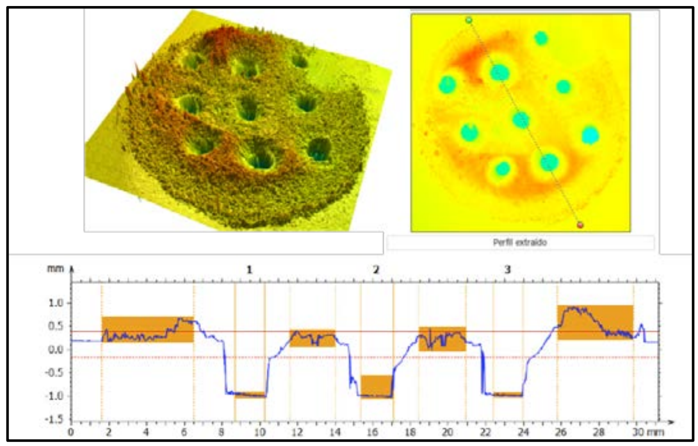

Figure 10 3D Image fouled plate for point 1

5. CONCLUSIONS

The purpose of this study was to characterize the gases and particle size emitted by the soot generator miniCAST in a experimental test bench in order to evaluate the operating conditions for further studies.

Six points were evaluated and the performance of the equipment was excellent with respect to the indicated in the manufacturer's manual. Obtaining the expected results by modifying the flow rates of both the mixing gas and the oxidation air. It was shown that an increase in the gas mixing flow results in a reduction of particle diameter, and equally a reduction in the oxidation air flow involves an increase in the percentage of hydrocarbons emitted.

Also, the expected results were obtained, both particle diameters as their concentration are between the ranges of current diesel engines.

It is necessary to take into consideration the third correction referred to in point 3 for the interpretation of the measurements made by the SMPS. There is a high influence of the primary diameter in this correction. Transmission Electron Microscopy (TEM) is recommended of the generated particles to obtain deep knowledge of the primary particles and the aggregates. This is not the case most of the time and the assumption of the primary diameter can lead to wrong corrections.

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