

Catalytic DPF Microwave assisted active regeneration

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ABSTRACT

In the last decades, diesel engines have attracted an increasing interest from vehicles producer and public, due to their higher thermal efficiency, less CO and unburned hydrocarbons emission, and longer lifetime comparing with gasoline engines. However, the high emission of soot particulates and NOx are still their key problems. The combination of filters and oxidation catalysts is one of the most effective after-treatment techniques to eliminate soot particles from the exhaust gases: the deposited catalyst lowers the soot ignition temperature from 550°C (uncatalyzed reaction) to the typical values of diesel exhaust gases (180–400°C). In our previous works we showed that the simultaneous use of a microwave (MW) applicator and a specifically catalyzed Diesel Particulate Filter (DPF), loaded up to 20%wt of Copper Ferrite (CuFe₂O₄), allows to reduce the temperature, the energy and the time required for the regeneration. Starting by these very promising results, in this work we continued to study in order to further improve the performances of the catalyzed DPF, reducing the temperature and minimizing the MW energy required for the regeneration. Furthermore we verified the feasibility of the MW technology by assessing the energy balance of the regeneration phase, comparing it to the actually employed regeneration technologies.

1. Introduction

In the last decades, diesel-powered vehicles have attracted an increasing interest from vehicles producer and public, due to their higher thermal efficiency, less CO and unburned hydrocarbons emission, lower cost and longer lifetime comparing with gasoline-fuelled vehicles [1]. Unlike gasoline emissions that are mainly gaseous in nature, diesel emissions from passenger cars, buses, and trucks contain solid, liquid, and gaseous components [2]: the solids emitted from diesel engines are essentially dry soot (carbon-rich particles), the liquids are unburned diesel fuel and lubricating oils (commonly referred to as soluble organic fraction or SOF) and to some extent sulfates originating from the combustion of the sulfur compounds present in the diesel fuel, the gaseous pollutants are CO, HC, and NOx. The combination of solid and liquid pollutants is referred to as particulates or TPM.

Due to the deleterious effects of these emissions on human health and on the global environment, government legislations for permissible exhaust emission standards were introduced in both Europe and United States of America in 1982 for light-duty vehicles and in 1990 for heavy-duty ones [3]. In Europe a series of six steps referred as Euro Standards (Euro I to Euro VI) was adopted for the reduction of the particulate matter and the other harmful gas components in the exhaust of the passenger cars and light and heavy-duty trucks. The evolution of European emission regulations and the associated emission control systems is shown in table 1 [2].

As evident from table 1, the combination of a catalyzed soot filter (CSF) and an oxidation catalyst (DOC) is one of the most effective after-treatment techniques to control the soot

emissions from the exhaust gases of these engines: the deposited catalyst lowers the soot ignition temperature from 550°C (uncatalyzed reaction) to the typical values of diesel exhaust gases (180–400°C) [4]. Regarding the catalysts, the researches have been addressed to the formulation of a stable and effective in the soot oxidation active species. Recently, transition metals (such as cobalt, copper and iron) supported/doped ceria mixed oxides catalysts show strong soot oxidation activity via a redox-type mechanism assisted by oxygen spillover on CeO₂ or a synergistic effect between transition metal oxides and ceria [5]. Especially, MnO_x–CeO₂ mixed oxides have evidenced high soot oxidation activities in both excess O₂ and NO [5]. Furthermore, always from table 1, is clear that the more stringent legislation limitations (EURO VI) require the addition of DeNO_x catalysts to DOC and CSF. Although several investigations on the soot combustion catalysts have been conducted and catalysts for NO_x storage and simultaneous NO_x-soot removal have been developed and studied [6], some problems still remain. Among them, the too high temperature of soot combustion and the relatively low NO_x storage capacity (NSC) are the main aspects. So how to simultaneously remove soot and NO_x in the low and same temperature region, such as 200–400°C, is still a challenging task; thus exploring more active catalysts for simultaneous soot combustion and NO_x storage is highly necessary [6]. Actually, among the various type of filters usually employed, the best effective is the Wall-Flow Diesel Particulate Filter (DPF), an honeycomb monolith (Figure 1) made of ceramic materials such as cordierite or Silicon Carbide (SiC) which consists of a series of parallel channels alternatively plugged at each end to force the exhaust gas flow through the porous filter wall [7], characterized by a filtration efficiency higher than 95% [8].

During the filtration, the porosity is progressively blocked by the trapped soot particles and this may lead to unacceptable values of exhaust back-pressure, therefore a regeneration process (classified as active or passive [9]) must be induced. The exothermic PM combustion leads in some cases to excessive temperature rise, which may damage the ceramic DPF. The development of a cost effective, fast and safe regeneration procedure is the major remaining technological challenge in the use of this device [10].

Various schemes are being considered by engine manufacturers for DPF heating including the use of microwaves [11–15]. Microwave heating for soot filter regeneration offers the advantage of rapid heating and the possibility of more uniform filter heating than can be achieved by resistance heating. In addition, there are claims that microwave heating can result in lower soot combustion temperatures than realized by other heating techniques [16]. Microwave heating has been suggested to be beneficial in a range of applications: of particular interest in this regard is the report by Ma et al. [17], who reported significantly lower soot ignition temperatures with microwave heating. These features are also considered to be particularly suitable for soot combustion in a SiC DPF due to the instantaneous penetration of microwaves into the filter body, the selective absorption of microwaves by the soot layer [17], and the good SiC dielectric properties, as shown in table 2.

As indicated in table 2, SiC seems to be the most adequate filter material, and it's evident that also the soot is a very good MW absorber characterised by high values of the Dielectric constant ϵ' and of the Dielectric loss factor ϵ'' . Furthermore, formulating the soot oxidation catalyst to absorb MW, the combination of MW heating with catalytic combustion may result in the effective oxidation of diesel soot at lower temperature and higher reaction rate [8, 15, 19].

The results of our previous tests, showed that the simultaneous use of the MW and the copper ferrite (CuFe₂O₄) loaded filter at lower gas flow rate, allows to sensibly reduce the regeneration time and the energy supplied respect than that required for the uncatalysed filter

[15, 19]. So the objectives of our work are to optimize the load of the Copper Ferrite on the filter, in order to simultaneously improve the catalytic DPF performances, in agreement with a sustainable pressure drop, and to verify the feasibility of this technology by assessing the energy balance of the regeneration phase, in order to compare it to the actually employed regeneration technologies.

2. Materials and methods

2.1 *The Wall Flow Filters*

Rectangular shaped (36x80x124 mm) Silicon Carbide (SiC) Wall Flow monoliths (Pirelli Ecotechnology, 150 cpsi), showed in figure 2, were selected as conformed support for the preparation of the catalytic filters. The prepared filters were then wrapped in an expanding intumescent ceramic-mat (Interam by 3M) which expands with heat and enclosed in a stainless steel wave guide.

In table 3 are reported additional geometrical characteristics of the filters.

2.2 *Catalyst choice*

As mentioned in the previous paragraph, an active and stable soot oxidation catalyst if specifically formulated to absorb microwaves may combine microwave heating with catalytic combustion reaction for the effective abatement of diesel soot. In the literature, the activity of catalysts towards soot oxidation in the presence of microwaves was studied: Gao et al. [20] demonstrated that for microwave-assisted Diesel ceramic soot filters a regeneration efficiency close to 80% can be reached, Nixdorf et al. [21] reported similar efficiencies for filter cartridges made of silicon carbide fibres. Furthermore Ma et al. [17] performed experiments of microwave assisted soot oxidation with and without catalyst: they found that iron and copper were the catalysts most active in lowering the ignition temperature of diesel soot, and that the iron containing catalyst was very effective and energy-efficient at low microwave input.

In previous works, the regeneration of a ceramic foam filter for soot trapping at the exhaust of a gas-oil burner had been performed in a specially designed single mode microwave cavity. The presence of catalyst was shown to enhance the soot oxidation rate in all the temperature ranges investigated [11, 12, 13].

Starting from these premises, the selected catalyst is CuFe_2O_4 , due to its very well known dielectric properties and good oxidation activity [15, 19].

2.3 *The experimental plant*

A well-equipped diesel emission control laboratory plant [22] was established (Figure 3) to perform the experiments; the plant includes a 505 cm³ city car diesel engine, a diesel particulate filter system, two electrical resistances in SiC with an annular shaped geometry designed to heat the engine exhaust stream up to 900°C, an Hall effect clamp meter to monitor the electrical power supplied by the resistances, a microwave energy supply system (Fricke and Mallah Microwave Technology GmbH), an AVL 439 opacimeter, a differential pressure sensor, a temperature measurement system, an on-line gas analysis system for the continuous monitoring of CO, CO₂, NO, NO₂, SO₂ and O₂, a data acquisition system in Lab-View Ambient to log all the analogical signals.

2.4 Catalyst preparation

The Copper Ferrite (CuFe_2O_4) was prepared starting from iron nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), copper nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$), mixed in a 2:1 molar ratio, and distilled water, continuously stirred at 60 °C. The catalytic DPFs have been prepared according to the previously optimized preparation procedure [15, 19], by repeated impregnation phases in the prepared solution, drying at 60°C and calcination at 1000°C after each impregnation, in order to obtain a load of active species up to 30 %wt. In this way we have obtained an uniform and homogeneous distribution of the active species on the DPF walls and inside the porosity, avoiding the occurrence of the filter fractures shown in literature for the thermal shock of SiC monoliths [23].

2.5 Textural and morphological characterization

The prepared catalyst powders were characterized by means of X-Ray Diffraction (XRD) and Thermo Gravimetric-Differential Thermal Analysis (TG-DTA). The prepared catalytic monoliths with various loading of CuFe_2O_4 were characterized by Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDAX), Hg porosimetry tests, N_2 adsorption at -196°C , applying BET method for the calculation of sample's surface area, H_2 -TPR and bending strength tests. Furthermore the effect of the thermal pretreatment on the filter morphology has been investigated. In addition the adherence of the active species to the filter was evaluated measuring the weight loss caused by exposing the catalyst loaded monoliths to ultrasound, following the experimental procedure reported in literature [24]: the samples have been immersed in a beaker containing petroleum ether (Carlo Erba), and the whole was placed in an ultrasonic bath CP104 (EIA SpA) filled with distilled water, at a temperature of 25°C, operating at 60% of rated power, for 30 minutes. Before the test, compressed air was blown through the monoliths in order to remove any possible residue. The weight changes were recorded during the test at regular intervals of 5 minutes after monoliths drying at 120°C and cooling up to room temperature.

3. Results and discussion

3.1 CuFe_2O_4 X-Ray Diffraction analysis

In order to verify the formation of the desired CuFe_2O_4 , we analyzed the powder obtained from the precursors solution after drying at 60°C and calcination at 1000°C by X-ray Diffraction (XRD), performed with a microdiffractometer Rigaku D-max-RAPID, using $\text{Cu-K}\alpha$ radiation. The results are reported in figure 4, where the diffraction pattern of the sample obtained as previously described is compared with different samples of Cubic (database 77-0010) and Tetragonal (database 34-0425) Copper Ferrite and with a commercial Copper Ferrite (Sigma-Aldrich). XRD analysis show the presence in the prepared Copper Ferrite of the typical peaks of CuFe_2O_4 in its tetragonal and cubic form with an average crystallite dimensions of about 20 nm evaluated by the application of the Scherrer formula; in figure 4 we also observe the presence of two minor peaks correspondent to low amounts of CuO and Fe_2O_3 .

3.2 TG-DTA analysis

The catalytic activity of powder catalyst and catalyzed DPF, was evaluated by simultaneous TG-DTA analysis (SDT Q600 TA Instruments) of soot alone and of soot mixed in a mortar

with milled catalyst loaded DPF samples at different catalyst load (15, 20, 25 and 30%wt of Copper Ferrite) and with the powder catalyst. Samples were heated in air (flow rate = 100 Ncm³ min⁻¹) from 293 to 973 K with a heating rate of 10 K min⁻¹. The results are reported in Figure 5 as Derivative Weight (in %/min), referred to the total amount of soot in the sample, as function of Temperature. The TG curve of soot alone shows the typical behavior of this material (ignition temperature of about 550 °C and maximum reaction rate at about 620°C).

The results relevant to the TG of soot mixed with the CuFe₂O₄ powder show that the ignition temperature is lowered to 380 °C and the maximum combustion rate temperature is lowered to about 450 °C, confirming its very good activity toward the soot oxidation. The comparison of the results relevant to the soot mixed with CuFe₂O₄ catalyzed DPF at different catalyst load, show that increasing the load of active species on the filter the ignition temperature and the maximum combustion rate temperature are both lowered and, more important, the rate of the catalytic combustion reaction increases.

3.3 *Effect of thermal surface pretreatment*

The effect of thermal surface pretreatment on the filters morphology has been investigated, in particular to verify the formation of a layer of SiO₂ on the SiC particle surface by oxidizing Si atoms on the surface at high temperature. The SiC filters were placed in a Muffle furnace and the temperature was raised to 1000°C and maintained in air atmosphere up to 48 hours. In this process, SiC can be oxidized by oxygen in air, according to the following equation:



In order to detect the morphology changes of the silica layer on the SiC particle, SEM was used to study the samples during the thermal treatment (Figure 6).

In the SEM images of the thermal treated SiC particles, it can be seen that the increase of the calcination duration results in a growing coating of the SiC particles with SiO₂ streaks, that, as reported in literature [25], can greatly help the adherence of the active species to the filter, even in the absence of a washcoat.

3.4 *Active species adherence testing*

The samples weight losses were evaluated according to the following formula:

$$\text{Weight loss} = \frac{\text{initial weight} - \text{final weight}}{\text{Initial weight}} \times 100 \quad (2)$$

The results are reported in the figure 7.

The above reported results, characterized by weight losses lower than that reported in literature for washcoated supports [24], show the high mechanical stability of the catalyst loaded monolith, so demonstrating the good adhesion of the catalyst on the filter surface even in absence of a washcoat.

3.5 *SEM-EDAX results*

Catalyzed and uncatalyzed samples have been investigated by Scanning Electron Microscopy (SEM), using a Scanning Electron Microscope (SEM mod. LEO 420 V2.04, ASSING), and Energy Dispersive X-ray Spectroscopy (EDX), performed in an Energy Dispersive X-Ray analyzer (EDX mod. INCA Energy 350, Oxford Instruments, Witney, UK): the results are shown in figures 8 and 9.

The comparison between the frontal channels showed in figure 8, highlights the coating of the filter surface with the active species, that are very homogeneously distributed. In figure 9 is shown the comparison among the channel walls of the uncatalyzed filter and the catalyst loaded filter with 15%, 20% and 30%wt of Copper Ferrite: besides the homogenous distribution of the active species on the SiC granules, is clear that their increasing load results in the decrease of the pore diameter, but not in their occlusion, so allowing its use as catalytic filter.

In Figures 10 and 11 are reported the SEM images and the elements distribution as obtained by EDX element mapping, for the uncatalytic and for the 15%wt of active species catalyzed filter, respectively.

The figures 10 and 11 reveal that while on the uncatalytic filter the encountered elements are only C, O and Si (the structural elements of the filter), on the catalytic filter the encountered elements are also Cu and Fe, the catalyst active species: these results confirm that with our catalytic filter preparation procedure, we can obtain the deposition of the active species on the support without any washcoat.

The figure 12, furthermore, highlights respectively the homogenous distribution and the good adhesion of the active species on the SiC granules.

The figure 12b, in particular, evidences the tight contact between the active species and the SiC granules; furthermore we can see that the CuFe_2O_4 granules have an average diameter of about 1 μm and that the average thickness of the catalyst layer is of about 3 μm . From figure 12 we can see more clearly that the active species cover all the SiC granules surface and that they don't deposit inside the inner wall pores but only on the external surface, and so on another layer of Copper Ferrite.

Some SEM images at various magnifications of a 15%wt catalyzed filter after the filtration step are reported in figure 13.

From figure 13 we can observe that all the filter surface is covered by finely dispersed soot particles; in particular figures 13b and 13c evidence the very tight contact between soot and active species. This tight contact is very important for the active species to fulfill their catalytic activity. Furthermore from figure 13c we can see the typical chainlike soot aggregate morphology, characterized by an average diameter in the range 50-200 nm.

3.6 Specific Surface Area

The Specific Surface Area (SSA) of the filters has been obtained by the mean of the SORPTOMETER Kelvin 1040 Costech instrument, applying BET method for its calculation. The values are summarized in table 4.

The values in table 4 show that the growing catalyst load over the bare monoliths induces an increase of the BET specific surface areas, as expected, since that with the deposition of active species on a support we increase surface roughness to the composite, without plugging pores.

3.7 Hg porosimetry tests

In order to evaluate the decrease in the median pore diameter, revealed by the SEM images in figure 9, the porosimetric characteristics of the filters have been measured by the Hg penetration technique using the “PASCAL 140” and “PASCAL 240” Thermo Finnigan instruments: the results are reported in figure 14 and summarized in table 5.

From the data above showed, it is evident that the increase in the CuFe₂O₄ load results in the median pores diameter and total pore volume decrease. In particular, the main changes of these values are observed up to 20% wt of active species, while the further increase from 20% to 30% wt gives only little decreases. These results indicates that the highest pore diameter reduction occurs at the lower catalyst load, while for the two highest catalyst loads the reduction in the pores diameter seems to be very small.

3.8 H₂ TPR Analysis

The H₂-TPR measurements were carried out using a SiC monolith loaded with 15% wt and 30% wt of CuFe₂O₄ from room temperature to 900°C at a heating rate of 5 °C/min in 5% H₂/N₂ flow. The reaction parameters (temperature and concentrations) have been monitored by the mean of an HIDEN Analytical system, able to discontinuously sample up to 20 gas streams, subsequently sent to a mass spectrometer. The results are shown in figure 15.

Figure 15 shows that the H₂-TPR profiles have two pronounced reduction peaks at about 300 °C and 610 °C; these peaks are attributed to the reduction of CuFe₂O₄ to Cu and Fe₃O₄, and subsequently of Fe₃O₄ to Fe [26]. The two reactions are:



The total amount of H₂ consumed for Cu mole (H₂/Cu ratio) was about 4 in the two cases, and this value is consistent with that for the complete reduction of CuFe₂O₄ to Cu and Fe according to the following reaction:



The total amount of H₂ consumed in the two cases corresponds to about 17% wt and 30,5% wt of CuFe₂O₄, that is in a quite good agreement with the estimated values of 15% wt and 30% wt of CuFe₂O₄ on the monolith.

From figure 15 is evident that the increase of the active species load results in the shift of the reduction peaks temperatures: in particular we observed that the first reduction peak temperature increases of about 10°C, while the second one decreases of about 20°C.

Furthermore, as shown in literature [27], after the reduction, mixture of Cu and Fe is favorable for the formation of CuFe₂O₄ at high temperature (about 800 °C in air). The TPR profiles showed in conclusion that the catalyst loaded monolith (up to 30% wt of active species) is able to act as a redox oxidation catalyst active in the temperature range 300 – 800°C due to the presence of a very good and homogeneous Copper Ferrite dispersion on the SiC carrier.

3.9 Bending strenght tests

In table 6 are shown the results of the bending strength tests performed at the Pirelli Ecotechnology laboratory on the catalyzed and uncatalyzed SiC samples.

The results clearly showed that the increase in the catalyst load results in a higher monolith bending strength, even if the main gain is observed up to 15%wt of CuFe_2O_4 , while the further increase to 30% gives an almost unappreciable rise. These results are in line with the Hg porosimetry results, indicating that at the lower catalyst loads the active species deposition occurs simultaneously inside the pores and on the walls of the DPF, while at the higher catalyst loads the Copper Ferrite seems to mainly deposit on the external surface.

3.10 Catalytic activity tests

3.10.1 Diesel soot filtration efficiency

As described in the introduction paragraph, one of the main characteristics of a DPF system is its soot filtration efficiency. So preliminary tests have been performed to evaluate the filtration efficiency of the Pirelli Wall Flow Filters, using the following equation:

$$\eta_f = \frac{C_{\text{before}} - C_{\text{after}}}{C_{\text{before}}} \times 100 \quad (6)$$

with:

C_{before} is the soot concentration in the exhaust stream before the filter

C_{after} is the soot concentration in the exhaust stream after the filter.

All the soot concentrations are obtained correlating the opacity and the soot load in the exhaust stream at different operating conditions of the diesel engine in terms of rpm: the exhaust gas opacity is measured by the mean of the opacimeter, while the soot concentration in the exhaust stream has been evaluated by weight difference between a clean filter and a filter on which is sent to a known and constant flow rate regulated by a MFC.

The concentration is then calculated as

$$C = \frac{M}{Q \cdot \Delta T} \quad (7)$$

With:

-M is the weight of soot collected on the filter

-Q is the gas volumetric flow rate through the filter

-\Delta t is the collection time

The relation that best fits the variation of opacity in the exhaust gas as a function of the soot concentration was evaluated by graphic interpolation: thanks to this correlation was subsequently possible to determine, from the opacity values continuously measured during the experimental tests in the various engine operating conditions, the average concentration of soot present in the gaseous stream, and so calculate the filtration efficiency according to eq. 6.

As reported in our previous works [19] the filtration efficiency is always higher than 95% in all the different engine operating conditions, so confirming the efficiency and effectiveness of these systems as soot filters.

3.10.2 Deposition and on-line MW assisted regeneration tests

Some deposition and MW assisted regeneration tests have been performed on catalytic DPFs with different CuFe_2O_4 load, applied directly at the exhaust of the diesel engine available in our laboratory plant. The typical behavior of the pressure drop through the filter (DP) during the deposition phase at the operating engine conditions of 1500 rpm and $P_{\text{oil}} = 30$ bar, with a fixed flow rate into the filter of about 100 l/min, is reported in Figure 16.

The data show the stages involved in the soot loading in the DPF: depth filtration, in which soot deposition is inside the filter wall, which results in changing of the wall microstructure, and is characterized by a slight increase in pressure drop with soot loading, and cake filtration, in which no more soot collection occurs inside the filter wall, but it takes place on the channel walls resulting in the formation of the “soot cake”, that starts to act itself as a filter. When this second filtration mechanism starts, we observe a sharp increase of the slope of pressure drop curve.

The effect of the active species load on the Pressure Drop (DP) through the filter is reported in Figure 17, in which the DP profile for the 15%, 20% and 30%wt loaded WFF is shown.

Figure 17 shows that the increase of the catalyst load results in the increase of the DP through the filter, as expected: in particular the starting DP increases from about 80 mbar for the 15%wt catalyst loaded filter up to about 110 mbar for the 30%wt catalyst loaded filter. It is important to note that the filtration efficiency is always higher than 95% in all the deposition tests.

When the valued soot load is about 5 g/l, we started the on line MW assisted regeneration phase. In Figure 18 are reported the DP and the outlet gas temperature as function of time during the regeneration phase of two catalytic DPFs, with 20% and 30% wt load of active species. In all the tests, as showed in our previous works [8, 15, 19], we heat the DPF with a lower gas flow rate (30 l/min), with the MW generator set at 50% of its maximum power.

As evident from figure 18, simultaneously to the MW application, the outlet gas temperature and the slope of DP-time curve both increase; by looking more deeply, it is important to note that the increase in the catalyst load from 20%wt to 30%wt results in a similar catalyst threshold temperature value (about 350°C) and, more important, in a faster regeneration phase (about 15 minutes instead of 25 minutes).

The comparison of the MW energy supplied during the regeneration of the two different DPFs (about 1185 kJ and 816 kJ for the 20% and 30%wt CuFe_2O_4 loaded filters, respectively), indicates that the increase of the active species load results in a higher catalytic activity and a further lower energy consumption than our previous results [19]. The comparison of the energy required in MW assisted catalytic DPF regeneration and in the actually employed regeneration technologies, summarized in table 7 as Regeneration Energy required (kJ) for DPF litres, performed with a soot load on the filter of about 5 g/l, shows that the simultaneous use of the MW technology and the 30% CuFe_2O_4 loaded DPF allows an energy saving of about 70% with respect to the traditional fuel post-injection regeneration technology [28], performed adding the fuel via in-cylinder post-injection event to heat the exhaust gas in the DOC and held until the soot oxidation is complete (typically about 430 g of extra fuel are required in a 1.9 liter 4 cylinder CIDI diesel engine).

The main difference between our proposed MW assisted regeneration technology and the traditional fuel post-injection is that it is independent from the engine operating conditions: both the two regeneration technologies may be started by the ECU (electronic control unit)

when the soot loading in the filter reaches a set limit, but while the former can be performed also at low engine rpms, the latter must be performed at speeds higher than 120 Km/h (at higher engine rpms). In this last case it is very important to keep constant the rpms engine operating conditions while the regeneration is in progress, otherwise it may not complete, leaving the filter partially blocked. If the regeneration is unsuccessful the extra fuel injected will not burn and will drain into the sump, oil quality will deteriorate as a result of this and its level will rise, leading to engine rupture.

Anyway, it is important to note that the optimal catalyst load is represented by the maximum load at which the activity is the highest, with a simultaneous still acceptable pressure drop value, in order to avoid too high pressure drops across the DPF and a consequently abnormal engine's operation.

4. Conclusions

The objectives of our work were to optimize the load of the active species on the filter, and to verify the feasibility of this technology by assessing the energy balance of the regeneration phase, in order to compare it to the actually employed regeneration technologies. In this work we have preliminarily further improved the previously optimized preparation procedure, by the increase of the bare SiC monolith calcination duration: in this way we have obtained the formation of a growing coating of the SiC particles with SiO₂ streaks, that help the adherence of the active species to the filter, even in the absence of a washcoat, as confirmed by the active species adherence tests that showed the high mechanical stability of the catalyst loaded monolith, so demonstrating the good adhesion of the catalyst on the filter surface. The XRD analysis of the prepared CuFe₂O₄ confirmed the presence of the characteristic peaks of the Copper Ferrite in its tetragonal and cubic form, so indicating that with our preparation procedure we can obtain the desired catalyst. The analysis performed on the DPFs showed that the increase in the load of active species up to 30%wt resulted in:

- lower soot oxidation temperature and increased reaction rate;
- decrease of the median pore diameter and total pore volume;
- increase of the Specific Surface Area;
- higher bending strength.

The deposition and on-line regeneration tests performed using a catalytic DPF with 15%, 20% and 30%wt catalyst load showed that the increase in the active species load resulted in higher initial pressure drops across the filter, in a lower catalyst threshold temperature value and in a faster regeneration phase, so confirming the TG-DTA results, and, more important, in a further lower energy consumption. The comparison of the energy required in MW assisted catalytic DPF regeneration and in the actually employed regeneration technologies, shows that the simultaneous use of the MW technology and the CuFe₂O₄ loaded DPF allows an energy saving of about 70% with respect to the traditional fuel post-injections regeneration technology. These data, together with the independence of the MW assisted regeneration technology by the operating conditions of the engine, confirm the palatability of our proposed technology as a potential substitute of the actually employed technologies in the DPF regeneration phase.

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