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Full Length Article

Microwave assisted regeneration of a catalytic diesel soot trap

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HIGHLIGHTS

- Improvement of an innovative catalytic diesel soot trap.
- The catalytic soot trap in SiC is able to absorb microwaves and burn soot at once.
- The catalytic soot trap is totally cleaned by the MW assisted regeneration.
- The combination of microwaves and catalytic soot trap allows a higher energy saving.
- The MW assisted DPF regeneration is independent of the engine operating conditions.

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ABSTRACT

Diesel engines have low fuel consumption and enough torque compared with equivalent gasoline engines, so the registration of new diesel cars is increasing in EU year by year. On the contrary, diesel engines exhaust contains large amount of hazardous substances, such as soot and NOx. Because of increasingly stringent emission regulations, various filters are commonly used for soot abatement in diesel exhaust, among which diesel particulate filter (DPF) is one of the most important. It consists of a bundle of small axial parallel channels, which are of small and, typically, square cross-section. Adjacent channels are alternatively plugged at each end, so that the gas enters into the monolith through the open channels in the inlet monolith cross-section (inlet channels) and is forced to flow through the porous inner walls: in this way the particles are collected on the surface and in the porosity of the channel walls, progressively blocking the pores and increasing the pressure loss. So a periodic regeneration is necessary, by burning off the accumulated soot. In our previous works we showed that the simultaneous use of a microwave (MW) applicator and a specifically $CuFe_2O_4$ catalysed DPF, allows to reduce the ignition temperature, the energy and the time required for the filter regeneration. Starting by these very promising results, the objective of this work is to modify the active species formulation in order to simultaneously further reduce the ignition temperature and keep low the pressure drop.

1. Introduction

The diesel internal combustion engine is one of the most environmentally-friendly vehicle devices, because it emits less carbon dioxide and is more fuel efficient than the universal gasoline stoichiometric engine. However, considerable challenges still exist regarding the emission control of particulate matter (PM), commonly known as soot, and NOx emitted by the diesel combustion process [1]. Since the reduction of both NOx and PM to the admitted level cannot be accomplished by engine modifications alone, after treatment processes for the reduction of diesel emissions should be developed [1]. Currently, PM emissions are generally controlled through the use of a diesel particulate filter (DPF), that consists of a silicon carbide (SiC) structure in the Wall Flow configuration, characterized by alternately plugged parallel square channels [2]. As a consequence, the exhaust gases entering in the monolith are forced to flow through the porous channel walls that act as filters; in this way high particulate trapping efficiency (>95%) can be achieved, as reported in our previous work [3]. As the particulates are accumulated in the pores of the filter, the pressure drop increases, and, after some time, burning of soot particles inside the filter is necessary. Soot burns at 550–600 °C with oxygen, while the diesel exhaust temperature is only 200–400 °C, therefore to force the soot burning two different systems are used: the first ones are continuous systems lowering the temperature of the soot oxidation using a catalyst [4], the second ones are active systems boosting the soot temperature up to the ignition

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temperature [5]. The microwave assisted regeneration of a loaded DPF is an active system of regeneration, because, depending on the filter load, the soot is heated up to its oxidation temperature; furthermore it differs from conventional systems in the point of heat introduction, since differently from conventional systems in which the heat is transferred to the filter indirectly by heating the exhaust gas [6], in the microwave regeneration system the heat is coupled directly into the soot. In this way combining the good dielectric properties of SiC, catalyst and soot, with MW heating and catalytic combustion, the effective oxidation of diesel soot at lower temperature and higher reaction rate may be reached [7]. In this way the independence of the MW assisted regeneration technology by the operating conditions of the engine, and the consequent energy saving in the regeneration step due to the combination of this technology with the catalytic DPF, could confirm the ability of our proposed technology as a potential alternative to actually employed technologies (such as fuel post-injection) in the DPF regeneration phase. The comparison of the performances of not catalysed and Copper-Ferrite loaded DPFs during the regeneration step, reported in our previous works, showed that the increase of catalyst load up to 30 wt.% and the simultaneous use of microwaves at lower gas flow rate, allows to reduce the energy supplied and the regeneration time than that required for the not catalysed filter [8]. Starting by these very promising results, the objectives of this work are to modify the active species formulation in order to simultaneously further reduce the PM oxidation temperature and keep low the pressure drop: in particular the aim of this work is to verify the effect of K addition to our catalyst formulation, since Liu et al. [8] observed that in the case of a DPF loaded with a K-doped copper ferrite ($Cu_{0.95}K_{0.05}Fe_2O_4$), the NOx presence in the exhaust stream had a positive effect on the catalytic activity.

2. Materials and methods

In this work $Cu_{0.95}K_{0.05}Fe_2O_4$ catalysed DPFs with different loads were prepared and characterized by several techniques, as described below.

2.1. Catalyst and Wall Flow Filters preparation

The catalyst ($Cu_{0.95}K_{0.05}Fe_2O_4$) was prepared starting from the precursors salts properly mixed with the right molar ratio, and distilled water, continuously stirred at 60 °C. Silicon Carbide (SiC) Wall Flow monoliths (Pirelli Ecotechnology, 150 cpsi) were selected as supports for the preparation of the catalytic filters. The catalytic monoliths were prepared by repeated impregnation phases in the precursors solution, drying at 60 °C and calcination at 1000 °C after each impregnation, following the previously optimized preparation procedure [9], so obtaining various loads of active species up to 30 wt.%.

The prepared filters, previously opportunely conformed in rectangular shape, showed in Fig. 1, and calcined at 1000 °C for 48 h, were then wrapped in an expanding intumescent ceramic-mat (Interam by 3M) which expands with heat and enclosed in a stainless steel wave guide.

Further geometrical characteristics of the filters are reported in Table 1.

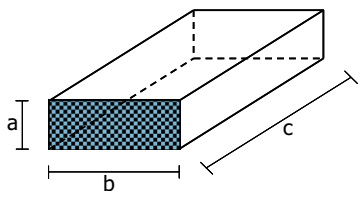

Fig. 1. Geometrical features of the 150 cpsi Pirelli Ecotechnology SiC Wall Flow Filter.

2.2. Textural and morphological characterization

The prepared powders of $Cu_{0.95}K_{0.05}Fe_2O_4$ were characterized by X-ray Diffraction (XRD) and TG-DTA analysis, while the catalysed DPFs were characterized by Scanning Electron Microscopy (SEM), Energy dispersive spectroscopy (EDAX), Hg porosimetry tests, H_2-TPR measurements, N_2 adsorption at −196 °C, applying BET method for the calculation of sample's specific surface area (SSA). In addition the adherence of the catalyst to the filter was evaluated measuring the weight loss caused by exposing the monoliths to ultrasound, according to the following experimental procedure [10]: a beaker containing the samples immersed in petroleum ether (Carlo Erba), 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 regular intervals of 5 min. Before the test, compressed air was blown through the monoliths in order to remove any possible residue. The weight changes were recorded after monoliths drying at 120 °C and cooling up to room temperature at the end of any interval. Furthermore catalytic activity tests in oxidizing atmosphere and some preliminary tests of soot deposition and on line MW assisted regeneration of catalytic DPFs were performed by means of our diesel emission control laboratory plant [9].

3. Results and discussion

The thermal surface pre-treatment of the bare monoliths allowed the coating of the SiC particles with SiO_2 streaks, that can greatly help the adherence of the active species to the filter, even in the absence of a washcoat [9]. Furthermore the optimized preparation procedure allowed to avoid the occurrence of the filter fractures shown in literature for the thermal shock of SiC monoliths [12].

3.1. X-ray diffraction analysis

In order to verify the formation of the desired active species, the catalyst powder was analysed by X-ray Diffraction (XRD), performed with a micro diffractometer Rigaku D-max-RAPID, using Cu Kα radiation. XRD analysis showed the presence in our prepared catalyst of the typical peaks of $CuFe_2O_4$ in its tetragonal and cubic form [2], and the absence of mixed oxides peaks.

3.2. TG-DTA analysis

The catalytic activity of the prepared catalysts was evaluated starting from powders, by simultaneous TG-DTA analysis (SDT Q600 TA Instruments) of soot mixed in a mortar with milled catalysed monolith samples at different active species load. The results were compared with the same analysis performed on soot alone.

Table 1
Geometrical characteristics of the filters.

Total channels	Open channels	Channel length (L) [mm]	Filter wall thickness [mm]	a [mm]	b [mm]	c [mm]
585	277	1.5	0.6	36	80	124