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Towards a single brick solution for the abatement of NO_x and soot from diesel engine exhausts

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Abstract

Nano-structured perovskite-type lanthanum ferrites $La_{1-x}A_xFe_{1-y}B_yO_3$ (where A = Na, K, Rb and B = Cu), prepared by the solution combustion synthesis (SCS) method and characterized by BET, XRD, FESEM, AAS and catalytic activity tests in microreactors as well as on an engine bench, proved to be effective in the simultaneous removal of soot and NO, the two prevalent pollutants in diesel exhaust gases in the temperature range 350–450 °C. The best compromise between soot and nitrogen oxide abatement was shown by the La-K-Cu-FeO_3 catalyst which displayed the highest catalytic activity towards carbon combustion and the highest NO conversion activity.

Keywords: Diesel particulate; Perovskite catalyst; Catalytic combustion; Nitrogen oxides reduction

1. Introduction

Diesel engines show both carbon monoxide and unburned hydrocarbons outlet concentrations much lower than those produced by spark-ignition engines. However, even the most recent diesel engines (e.g., common-rail ones) generate nitrogen oxides and carcinogenic particulate, whose size (50–200 nm) falls in the so-called lung-damaging range [1]. The automotive industry is currently facing serious challenges to meet the specific requirements of future regulations concerning both NO_x and particulate emissions. Less than $0.005\ g\ km^{-1}$ for particulate and $0.20\ g\ km^{-1}$ for NO_x will be prescribed by the pending "EURO 5" regulations to be enforced by the year 2009. Either engine modifications [2] or fuel pre-treatments [3] or more simply by a better tuning of the combustion process [4] could not alone meet these limits: a convenient way of treating diesel off-gases has thus to be worked out. In this context, diesel particulate traps appear to be mandatory to most researchers in the field and the authors have already been involved for several years in some European R&D programs (CATATRAP, DEXA-cluster, TOP-Expert, ATLANTIS) aiming at the development of catalytic traps [5]. Catalysis can indeed play a significant role, by possibly achieving as good results as those obtained with catalytic converters in gasoline-fired engines [6]. Research in this field is carried out worldwide towards two different solutions:

1. Filtration of particulate in a first catalytic converter (the soot is trapped and burned out owing to the presence of a catalyst thoroughly deposited onto the filter [7]), whereas NO_x are eliminated in a second catalytic converter either by reaction with suitable reducing agents (light hydrocarbons, fuel, ammonia, etc., [8]) or by direct decomposition [9].
2. The lean NO_x trap (LNT) concept, which is one of the leading candidates among denoxing technologies. LNTs are formulated with a storage component, usually oxides of alkali or alkaline earth metals, such as barium or potassium, and precious metals over a high surface area γ-Al_2O_3 support. During lean operation, NO_x is "trapped" in the storage component in the form of nitrates. For short periods the engine is operated slightly rich such that the stored nitrates are released as NO_x and subsequently reduced to N_2 by CO, H_2 and UHC over the precious metal sites [10]. Through successive cycles of lean and rich operating conditions, LNTs

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can achieve NO_x conversion efficiency greater than 90%. Even though significant progress in improving LNT performance has been recently achieved, sulphur tolerance is a major hurdle that needs to be addressed before LNTs can be commercially viable.
3. Contemporary removal of soot and nitrogen oxides in a single filter catalyzed with a suitably developed catalytic material [11].

This latter way is clearly more ambitious than the former one, and has been very recently explored in view of the considerable advantages it might enable in terms of both investment cost and pressure drop reduction. It is well known that 10–15% of NO_x is generally reduced during regeneration of soot laden filters. This reduction mostly involves NO_2 molecules by direct reaction with soot to form NO and to a lesser extent N_2 and N_2O [12]. This reaction, exploited in the so-called CRT system by Johnson Matthey, stands at the grounds of the present study [13]. Despite the existence of several technical problems to be solved (e.g., finding out the best catalyst deposition on the trap, the best trap design so as to allow an intimate contact between the reactants and the catalyst itself, etc.), the more urgent research topic to be addressed for the time being is the development of a suitable catalyst, capable of promoting both soot oxidation and NO_x reduction at comparatively low temperatures (possibly within the range typical of diesel exhaust: 150–380 °C). On the grounds of previous investigations of ours [14,15], mixed oxides seem to be the most promising catalysts for such a purpose. In the present work, the $La_{1-x}A_xFe_{1-y}B_yO_3$ (where A = Na, K, Rb and B = Cu) perovskite catalysts were prepared, characterized and tested for their activity in the simultaneous abatement of NO_x and soot.

2. Experimental

2.1. Catalyst preparation and characterization

Several La-Fe perovskite catalysts ($La_{1-x}A_xFe_{1-y}B_yO_3$ where A = Na, K, Rb and B = Cu) were prepared via a highly exothermic and self-sustaining reaction, the so-called "*solution combustion synthesis*" SCS method [16]. This technique is particularly suitable for production of nano-sized particles of catalyst. The combustion synthesis process can be formally split into two steps (the preparation of $LaFeO_3$ is here considered as an example):

$$2La(NO_3)_3 + 2Fe(NO_3)_3 + 10CO(NH_2)_2 \rightarrow 2LaFeO_3 + 10CO_2 + 16N_2 + 20H_2O \quad (1)$$

The whole reaction can be formally regarded as the combination of two different contributions:

$$2La(NO_3)_3 + 2Fe(NO_3)_3 \rightarrow 2LaFeO_3 + 15O_2 + 6N_2 \quad (2)$$

$$10CO(NH_2)_2 + 15O_2 \rightarrow 10CO_2 + 20H_2O + 10N_2 \quad (3)$$

The first one (2) is the endothermic reaction for the real perovskite synthesis starting from the metal nitrate precursors, while the second one (3) is exothermic and accounts for the reaction between urea and oxygen derived from nitrates decomposition. Some direct combustion of urea with atmospheric oxygen cannot of course be excluded as the preparation is carried out in air within an electric oven kept at 500 °C hosting the precursors mixture placed in a porcelain pot. The overall set of reactions is markedly exothermic, which leads within the reacting solid mixture to a thermal peak well exceeding 1000 °C for a few seconds. To increase this sudden heat release, a varying amount of NH_4NO_3 was also added and kept into account in the fuel-to-oxidizers ratio.

Under these conditions nucleation of perovskite crystals is induced, their growth is limited, and nano-sized grains can be obtained, as earlier anticipated. After preparation, all catalysts were ground in a ball mill at room temperature and submitted to physical and chemical characterization. The nano-structured nature of the catalyst is pursued so as to increase the number of contact points between the catalyst and the particulate, a key feature of the reaction pathway, as later discussed.

Chemical analysis of the catalysts was performed by atomic absorption spectroscopy (AAS) in a PerkinElmer 1100B spectrophotometer after dissolution in strongly acidic media, to verify the amount of each constituting element in the prepared perovskites. The catalysts were analyzed by X-ray diffraction (PW1710 Philips diffractometer equipped with a monochromator, Cu Kα radiation) in order to asses their purity, crystalline structure and approximate crystal grain size. Field Emission Scanning Electron Microscope (FESEM - Leo 50/50 VP with GEMINI column) was employed to analyze the microstructure of the crystal aggregates of the catalysts both as prepared and after aging. The BET specific surface area of the catalysts has been finally evaluated from the linear parts of the BET plot of the N_2 isotherms, using a Micromeritics ASAP 2010 analyzer.

2.2. Catalytic activity assessment

The catalytic activity of the prepared catalysts was tested in a Temperature Programmed Surface Reaction apparatus (TPSR). A standard gas mixture (1000 ppmv NO; 10 vol.% O_2, He = balance) was fed at the constant rate of $100\ N\ ml\ min^{-1}$ via a set of mass flow meters (Brooks) to a fixed-bed microreactor enclosed in a quartz tube placed in an electric oven. The tubular quartz reactor was loaded with 50 mg of a 1:9 by weight mixture of carbon and powdered catalyst and 150 mg of SiO_2 granules (0.3–0.7 mm); this inert material was added to reduce the specific pressure drop across the reactor bed and to prevent thermal runaways. Experiments were performed by using, instead of real diesel soot, an amorphous carbon (Printex U by Degussa) having the following properties: average particle size = 45 nm; 0.34 wt.% ashes after calcination at 800 °C; 12.2 wt.% of moisture lost after drying at 110 °C. This material was preferred so as to avoid any interfering effect due to the presence of adsorbed HCs, sulphates or fly ashes present in real diesel soot. Moreover, the carbon employed is more difficult to burn than diesel soot, which renders the achieved results conservative [5]. A proper and reproducible mixing between catalyst and carbon was routinely carried out grinding carefully together such substances for 15 min in a ball mill. These mixing conditions are normally addressed as “tight-contact” ones [7]; they were ensured in the present study because they allow a better reproducibility. The real soot–catalyst contact conditions in a real catalytic trap are though less intensive. The catalyst–carbon–SiO_2 fixed bed was sandwiched between two quartz-wool layers, while the tip of a K-type thermocouple was located well inside the bed itself to determine the reaction temperature. The oven temperature was controlled through a PID regulation system based on the measurements of an external K-type thermocouple and varied during each TPSR runs from 200 to 700 °C at a 5 °C min^{-1} rate. A 30 min stay at 200 °C under He flow was adopted as a common pre-treatment in order to eliminate possible contaminants such as adsorbed water. The analysis of the outlet gas was performed via a N_2O/CO/CO_2 NDIR analyzer and a NO/NO_2 chemiluminescence analyzer by ABB (the amount of N_2 was deduced by balancing the measured nitrogen oxide species). The temperature of maximum CO_2 production (T_p) and of maximum NO conversion could be pointed out as indexes of the catalytic activity.

2.3. Catalytic filter preparation and diesel engine bench tests

The best catalyst selected (La-K-Cu-FeO_3, as detailed later) was deposited by *in situ* SCS directly over the wall-flow filters. The ceramic support was dipped in the aqueous solution of its precursors and then placed into an oven at 600 °C. The aqueous phase was rapidly brought to boil, the water evaporated, the precursors mixture ignited and the synthesis reaction induced *in situ*. The support selected was a silicon carbide (SiC) filter produced by NGK (cell structure = 17/200; diameter 35 mm; length = 10 in.; pore diameter of channel walls = 23 μm; porosity of channel walls = 52%) which was found to be chemically compatible with the selected catalyst. The load of catalyst deposited was assessed by gravimetric analysis and resulted equal to 4.8 wt.%.

The developed filter was tested over real Diesel exhaust gases on an engine bench (Iveco F1C Unijet 4 cylinders, 3000 cc, 122 kW at 3500 rpm, maximum torque 380 Nm), where the temperature and gas composition upstream and downstream the trap can be controlled and monitored, as well as the evolution of the pressure drop through the trap (a sign of soot accumulation therein). A detailed description is provided in Ref. [17].

The gas superficial velocity across the filter could be controlled by measuring the exhaust flow rate through a volumetric flow controller connected to a throttling valve placed on a by-pass exhaust stream. The pressure drop across the filter could be measured by suitable differential pressure transducers (VIKA), whereas K-type thermocouples were mounted at axial position just upstream and downstream the trap so as to measure the local temperatures. In line with the pending “EURO 5” regulations, all the tests were carried out by using a low-sulphur (<50 ppmw) diesel fuel produced by Agip Petroli.

The following standard bench test procedure was adopted. The filter was loaded by letting comparatively cold exhaust gases flow through it until a 170–180 mbar pressure drop was reached. Then, regeneration was induced by post-injecting some fuel (0.025 kg fuel/kg exhaust gases) with a metering pump (ISMATEC - Reglo CPF analog) and by burning it with an oxidizing honeycomb catalyst (OXICAT by Johnson Matthey) placed just upstream the filter so as to get to an inlet trap temperature of about 550 °C. The time needed for the complete filter regeneration (e.g., combustion of soot hold-up) is an index of catalyst performance. The higher the catalyst activity for soot oxidation by O_2 assisted by NO_2 the lower the time required to regenerate the filter. The completeness of the regeneration process was indicated by the decrease of the filter pressure drop till a value practically equal, or lower, to that measured at the beginning of the loading phase. As far as the NO conversion is concerned, the temperature and gas composition upstream and downstream the filter were controlled and monitored, in order to evaluate the catalytic activity towards NO reduction. A twin run on a virgin, non-catalytic filter was also performed for the sake of comparison.

3. Results and discussion

All perovskite catalyst samples were found to be well crystallized by XRD analysis (Fig. 1). All the main diffraction peaks listed in the reference JPCDS card ($LaFeO_3$: PDF 37-1493) were observed. No secondary phases could be detected by this technique. X-ray diffraction has a ±2/3% precision, and therefore, the presence of amorphous phases cannot be completely excluded. Besides, chemical analysis (dissolution + atomic absorption, O_2 titration) confirmed, with its intrinsic precision (±5%), that the amount of the various constituting elements (La, Ni, Na, K, Rb, Cu, Fe, O) was consistent with that used in the precursors and was compatible with the phases detected by XRD. In the present context, perovskite crystals having a size of the same order of magnitude of that of the particulate agglomerates are expected to provide the highest specific number of contact points between the soot directly captured over the catalyst layer and the crystals constituting

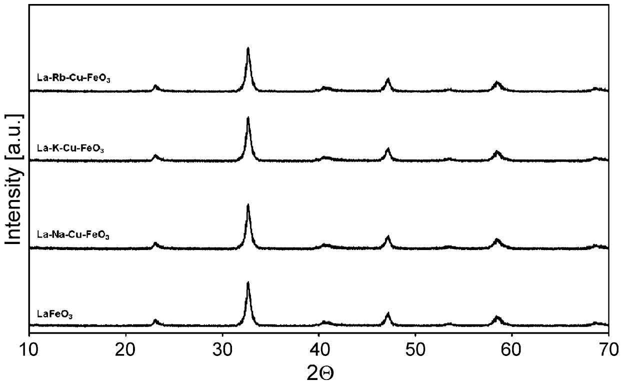

Fig. 1. XRD diffraction patterns of the perovskite-type catalysts prepared.

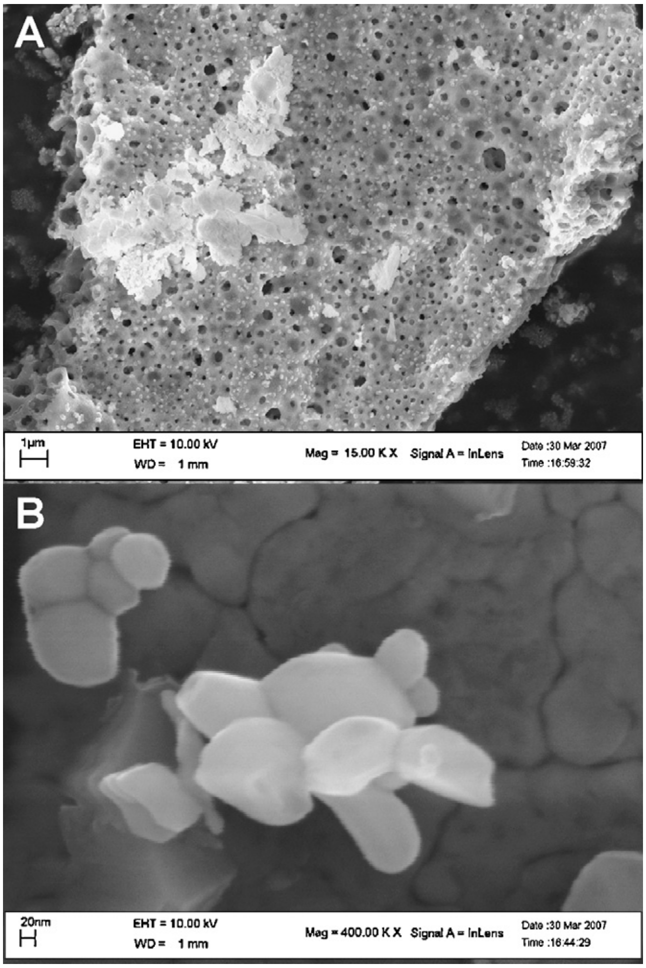

Fig. 2. FESEM electron microscopy pictures of $LaFeO_3$ catalyst: (A) catalyst microstructure and (B) catalyst crystals.

such a layer. Fig. 2A shows a FESEM picture of $LaFeO_3$ perovskite-type catalyst produced via SCS. It refers to the catalyst that showed the highest BET area among those prepared (see Table 1). This FESEM picture enlightens a very foamy structure, a typical feature of catalysts synthesized by SCS. This should once again remarkably favor the contact between soot and catalyst even under loose contact conditions and, once coated upon the wall-flow trap channel walls, it should allow just a slight increase of pressure drop. Fig. 2B shows a close view of the same catalyst. The perovskite crystals of the considered catalyst have a size below 100 nm, which is perfectly in line with the dimensions of particulate nuclei and is in substantial agreement with the specific surface area measured. Anyway the perovskite crystals of all catalysts prepared have a size ranging between 60 and 100 nm, which satisfies the above requirement.

Table 1
BET analysis and catalytic activity results

Catalyst	BET [m^2/g]	T_p [°C]	Max NO conversion [%]	T of max NO conversion [°C]
$LaFeO_3$	25.4	415	21	451
La-Na-Cu-FeO_3	9.4	370	84	388
La-K-Cu-FeO_3	7.9	359	79	384
La-Rb-Cu-FeO_3	10.7	375	76	387

With these catalytic materials, without a very significant internal porosity (as opposed to carriers like γ-Al_2O_3), the specific surface area is coincident with the geometrical surface area exposed by the crystals. As direct contact between soot and catalyst is essential, foamy and nano-structured catalysts having the same size of soot can indeed increase the number of contact points between catalyst and soot.

Shifting to the activity screening results, Table 1 summarizes the BET specific surface area, the carbon combustion peak temperatures, the maximum NO conversion and the relative peak temperature of all the catalysts investigated. A detailed investigation was carried out on these oxides by temperature programmed oxidation of soot and reduction of NO_x (TPSR) tests in fixed bed microreactors. As expected, all the catalysts significantly lower the combustion peak temperature compared to that of the non-catalytic combustion ($T_p = 560$ °C). An activity order can be outlined as follows:

(1) despite the lowest specific surface area the La-K-Cu-FeO_3 catalyst appears to be the one displaying the highest catalytic activity towards carbon combustion combined with a very high NO conversion activity, as well ($T_p = 359$ °C; max NO conversion = 79% at 384 °C). For this reason, further investigations at a catalytic trap level were carried out on this catalyst only;
(2) the other perovskite catalysts characterized by both lanthanum and iron substitutions with alkali metals and copper, respectively, exhibit quite similar activity (T_p ranging from 370 to 375 °C; max NO conversion ranging from 76 to 84%);
(3) the un-substituted $LaFeO_3$ is by far the less active catalyst ($T_p = 415$ °C; max NO conversion = 21% at 451 °C) despite its highest specific surface area.

In line with earlier papers of ours [16,19], it can be deduced that substitution of part of lanthanum with a lower valence alkali metal brings about formation of high valence iron to maintain electro-neutrality and possibly to more active or more concentrated oxygen species over the catalyst surface. It seems to be clear that the presence of both alkali metals at the A site and copper at the B site is directly involved with the activity towards both carbon combustion and NO reduction.

Fig. 3 shows the complete set of gas outlet concentrations plots of the various components involved in a standard TPSR run on the La-K-Cu-FeO_3 catalyst but it is representative of the catalytic behavior of all the prepared catalysts.

It seems clear that the two reaction processes (soot combustion and NO reduction) occur simultaneously. To strengthen this conclusion, explorative TPSR runs (not reported for the sake of briefness) performed by feeding fixed beds of the best catalyst in the absence of carbon or with carbon in the absence of catalyst, lead to just a negligible reduction of nitrogen oxides. Coming back to Fig. 3, the early stages of soot combustion, at temperatures below 330 °C, seem to involve a

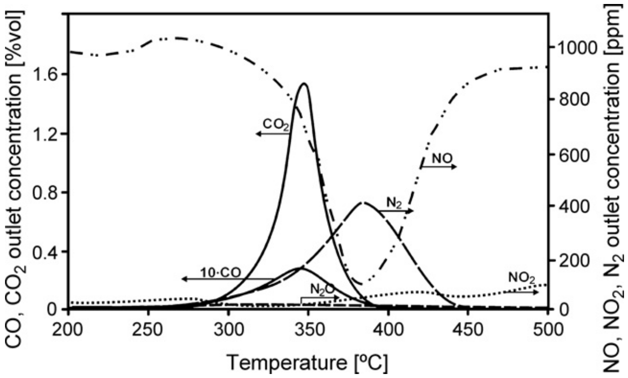

Fig. 3. Outlet gaseous species concentration in a TPSR run performed with La-K-Cu-FeO_3 catalyst.

certain amount of oxygen derived from NO_x reduction. This effect seems to progressively vanish as long as the combustion progresses and higher temperatures are reached. It is very likely that a key player in the NO_x reduction and soot oxidation processes is NO_2 whose production gets maximized in the 300–400 °C range as a consequence of kinetics (below 300 °C) and thermodynamic (above 400 °C [18]) limitations. Deeper investigations are needed to elucidate this point, in line with earlier papers of us [14,15]. Anyhow, the scenario is rather complex. CO_2 is generally by far the main carbon oxidation product with a selectivity well exceeding 95%. A certain role of CO as a reducing agent may be present. However, this role should be minor as it is known that NO reduction by CO on metal oxide catalysts (e.g., [20]) is strongly inhibited by the presence of oxygen, which was accounting for 10 vol.% of the feed gas mixture in any TPSR experiment performed. The main product of NO reduction is N_2. N_2O formation is rather low (less than 30 ppm). Conversely, NO_2 outlet concentrations, formed to some extent from the fed NO and O_2 according to the $NO + 1/2O_2 \leftrightarrow NO_2$ equilibrium, generally increases with the temperature. By the way, NO_2 should actually contribute to direct carbon combustion, but with minor impact on N_2 generation as the main reduction product in this case is known to be NO itself.

Shifting to the analysis of the developed filter systems performance, Fig. 4 compares the results of the runs obtained with the catalytic and the reference, non-catalytic SiC wall-flow monolith. After filter loading up to a pressure drop of about 170–180 mbar, the post-injection of about 0.025 kg fuel per kg exhaust gases was operated in both cases. This entailed a rapid increase up to 550 °C in the inlet trap temperatures, as a common operating condition. The results are rather encouraging. The regeneration of the La-K-Cu-FeO_3-catalyzed trap was much faster than that of the non-catalytic ceramic filter; the soot combustion period resulted nearly one third shorter (see the regeneration time evaluated from the starting point of the fuel post-injection). By these means, it becomes possible to save a significant amount of fuel at any filter regeneration, thereby reducing the operating costs.

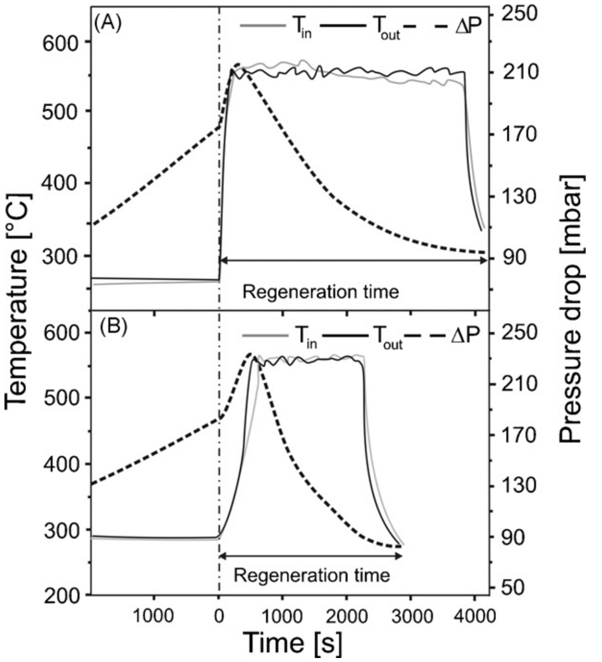

Fig. 4. Results of loading and regeneration runs for: (A) non-catalyzed and (B) catalyzed (La-K-Cu-FeO_3) wall-flow filters.

Another important feature lies in the more complete regeneration of the catalytically promoted filter: the pressure drop value at the end of the regeneration is about 15 mbar lower than that of the non-catalytic filter. As a consequence of this higher completeness in the regeneration, a lower regeneration frequency can be expected for the catalytic filter, compared to the non-catalytic one, which should result in further savings in post-injected fuel.

It has to be kept into account, however, that the fuel saving might in part be compensated by a small filter permeability decrease for the presence of the catalyst which brings about a slightly higher back pressure.

As far as the NO conversion is concerned (Fig. 5), a reduction of the nitrogen oxide emitted during regeneration of the catalytic trap, as opposed to the non-catalytic one, was observed. However, the results are a bit less satisfactory than those obtained during the TPSR experiments. During the regeneration phase the NO conversion to N_2 was about 10%

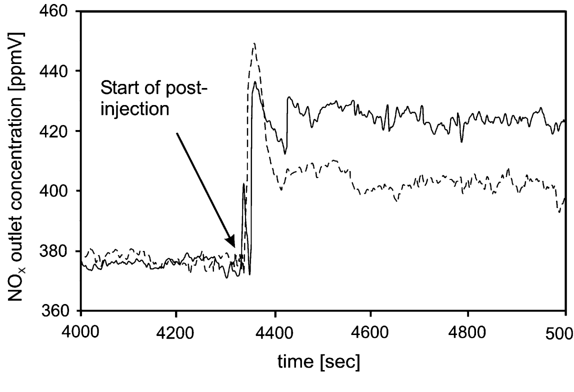

Fig. 5. Highlight of the transition between the loading and regeneration phases for the catalyzed (La-K-Cu-FeO_3, dot line) and a non-catalytic (solid line) wall-flow filter in terms of NO_x reduction performance. Same operating conditions as in Fig. 4.

which is interesting, even if quite lower than the 79% value recorded for the TPSR runs. In any case, one can conclude that even under loose contact conditions the catalysts are generally performing significantly better than the reference non-catalytic combustion/reduction. A possible explanation of the NO_x conversion obtained, compared to the microreactor runs on catalyst–soot mixtures, may lay in the fact that filter regeneration occurs at temperatures rather high (about 550 °C), compared with those at which NO reduction was observed in the microreactor runs (300–450 °C). At 550 °C NO conversion to NO_2 is actually less favored thermodynamically [21], which may result in a further sign of the key role played by NO_2 in this simultaneous combustion/reduction process.

In any case, any little reduction of NO_x obtained by the catalytic system developed will turn in an advantage of any downstream NO_x removal system: LNT traps would indeed be regenerated less often, whereas less urea would be consumed in the SCR system.

4. Conclusions

Some perovskite-type catalytic materials were prepared, characterized and tested for potential application in the treatment of diesel exhaust gases as promoters of both NO_x and soot removal. A common feature of all the catalysts tested was the simultaneity of the denoxing and the soot combustion reactions, but the catalyst containing both K and Cu elements showed particularly a higher NO reduction activity and the lowest combustion peak temperature. In this context, the present investigation proved that a wall-flow ceramic filter catalyzed with the La-K-Cu-FeO_3 obtained by *in situ* SCS looked very promising, as it entailed a one third reduction of the time required for trap regeneration (and of the related fuel penalty) as well as a more complete regeneration compared to that of a non-catalytic filter. Moreover, the La-K-Cu-FeO_3-catalyzed filter facilitates the conversion of NO, especially during the regeneration phase, which can turn into savings in any of the NO-reduction techniques (LNT or SCR) possibly adopted downstream the particulate filter. An experimental test campaign is currently in progress to better verify this potential at a catalytic filter level on diesel commercial vehicles. Alongside with this, studies are in progress to better understand the reaction mechanism leading to simultaneous NO_x reduction and soot oxidation in this rather complex solid–solid–gas system.

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