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High-throughput approach to the catalytic combustion of diesel soot II: Screening of oxide-based catalysts

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ABSTRACT

Following the development of a high-throughput (HT) methodology for the evaluation of diesel soot oxidation catalysts in a 16 parallel channels reactor, a library of over 60 catalysts was tested under optimized conditions. The catalyst compositions were chosen to include solids which specific properties like oxygen storage capacity, oxygen mobility and ionic conductivity. The key parameters for high activity appear related to the presence of active and mobile surface oxygen species, and to an appropriate catalyst particle size in order to favour the number of contacts with the soot. In contrast, high oxygen storage capacity and bulk oxygen ion mobility do not appear as relevant properties for high catalytic activity. Nine new formulations were found to perform better than the reference catalyst "high surface area (HSA) ceria" (Rhodia).

1. Introduction

The implementation of EURO 5 emission standards in 2011 for passenger cars will restrict the particulate emissions of Diesel vehicles to 0.005 g/km, which will make the introduction of Diesel particulate filters (DPFs) obligatory. Despite their recent development on passenger cars, DPFs are still facing major issues, like high regeneration temperature, mechanical and chemical instability due to the severe regeneration cycles, and a significant fuel penalty due to additional fuel consumption for filter regeneration. Moreover, the filter regeneration requires the use of cerium-based additives in the fuel, which can lead to irreversible pore plugging by ceria deposits, and might represent an environmental concern if spread in the atmosphere at large scale. Therefore, the development of catalytic filters requires the discovery of catalysts to be deposited within the filter to promote soot combustion at low temperatures and avoid the use of additives in the fuel [1,2].

High-throughput experimentation (HTE) and combinatorial methods are now frequently applied to rapidly investigate broad domains of catalyst compositions [3]. However, few works have been reported in the literature to evaluate soot combustion catalysts under screening conditions. McGinn and co-workers used automated thermo-gravimetric analysis to benchmark a series of alkali metal doped oxides based on copper and cerium oxides [4], Cu–Fe oxides [5] and on Cu, Fe, Co, Mn, Mo simple or mixed oxides [6] in the combustion of synthetic soot. Potassium was the best promoter of the catalytic activity of the solids, but the degradation of catalyst performances due to potassium sublimation during soot combustion was evidenced. Olong et al. [7] applied infrared thermography for primary screening of libraries with up to 206 catalyst–soot mixtures. However, the testing conditions can be considered as relatively remote from real soot combustion in a DPF: (i) screening at fixed temperature, generally below the light-off temperature, (ii) soot/catalyst contact not precisely controlled and (iii) static reaction conditions instead of dynamic combustion.

In a previous study [8], a HT procedure was developed for HT screening of diesel soot combustion catalysts. Using a commercial high surface area ceria (HSA ceria, Rhodia), the testing conditions were optimized to provide reliable and reproducible information on the catalytic activity. These conditions were validated by synthesizing and testing the perovskite oxide $La_{0.8}Cr_{0.8}Li_{0.2}O_3$ described by Fino et al. [9]. In the present study, this high-throughput procedure has been applied to the screening of a large catalyst panel based on oxide and mixed oxide formulations, which were compared to HSA ceria used as reference catalyst.

2. Experimental

2.1. Catalysts and soot

The mixed oxide catalysts were synthesized using the citrate-gel method. An aqueous solution of metal precursors (nitrates), in

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the desired stoichiometric ratio, was mixed with citric acid in the proportion of one mole citric acid per mole of nitrate. The pH of the solution was adjusted at 6–7 by addition of concentrated ammonia. This solution was heated under stirring at 90 °C, until a viscous gel was formed. The gel was successively heated at 110, 130 and 170 °C in an oven, which led to swelling into a voluminous powder. Finally, the powder was grinded in a mortar and calcined at 600 °C in air for 3 h.

The soot samples were collected in an uncatalysed flow-through particle trap placed in the exhaust stream of a 2.2 L Renault diesel engine after an oxidation catalyst.

2.2. Characterizations

X-ray diffractograms of powder samples were performed on a Siemens D500 diffractometer, using a Cu Kα radiation ($\lambda = 0.154184$ nm). The diffractogram analysis was realised using the DiffracPlus Eva software and the ICDD-JCPDS database.

The particle size distribution of powder samples was measured by laser granulometry, assuming a particle spherical shape. The percentage of particles having a certain size was measured from the total solid volume.

Thermal analyses (DTA/TGA) were performed in air with a SETARAM analyzer.

2.3. Catalytic tests

Catalytic tests were performed in a 16 parallel reactors bench (commercialised as SWITCH 16 by AMTEC GmbH). The 16 Inconel reactors can be heated up to 650 °C with a ramp ranging from 1 to 5 °C/min. They are fed with the same stream composition at the same space velocity. Each reactor is equipped with a K-type thermocouple placed inside the catalyst/soot bed, allowing the continuous measurement of the temperature. Experimental error on the soot ignition temperature is estimated at ±3 °C [8]. A second thermocouple monitors the reactor temperature, which is increased linearly with a heating rate of 5 °C/min. A complete description of the system can be found elsewhere [10]. Reaction products of one selected reactor can be monitored by mass spectrometry using an Inficon mass spectrometer.

Catalytic tests performed previously with a loose soot/catalyst contact were found to be poorly reproducible [8]. Therefore, the screening tests were generally performed under tight catalyst/soot contact, as reported by other authors for the same reason [11]. Although this contact does not represent the reality of a catalytic DPF [12], it provides an accurate and reproducible benchmarking of catalysts. In a standard experiment of temperature programmed oxidation, the 16 stainless steel reactors were loaded with 40 mg of mixtures of 40 mg soot with 400 mg of catalyst, placed between two quartz wool plugs. The soot was manually grinded with the catalyst in an agate mortar (tight contact). Oxidation experiments were performed from 25 to 600 °C with a heating rate of 5 °C/min, under a 40 mL gas flow (50% O_2 in He). The ignition temperature T_{ign} corresponding to the light-off process was defined as the intersection between the tangent to the temperature ramp profile, when it starts to deviate from linearity, with the tangent to the temperature peak corresponding to the exothermic reaction, i.e. 458 °C in Fig. 1. The maximum temperature $T_{\max}$ corresponding to the exothermic peak (638 °C) was not considered as a reliable parameter, since it depends on the amount of soot and on the catalyst/soot ratio, whereas the shape of the exothermic peak can also vary significantly. We showed previously [8] that the exothermic peak matches exactly the CO_2 production associated to soot combustion.

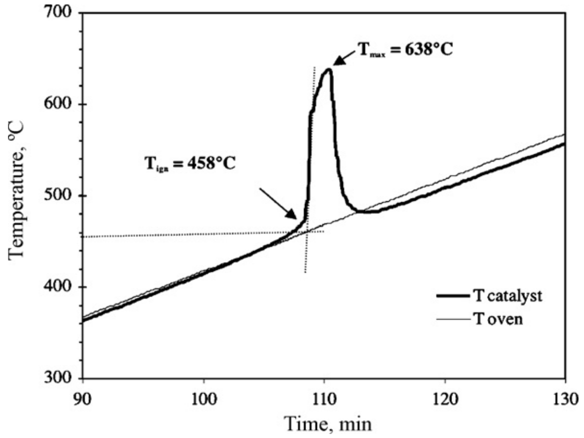

Fig. 1. Light-off temperature identification in diesel soot combustion process. Conditions: 40 mg soot/400 mg catalyst in tight contact, 40 ml/min O_2/He (50% O_2).

A few tests were performed in loose contact (catalyst powder and soot mixed with a spatula), for the purpose of performance comparison with the tight contact.

3. Results and discussion

The approach for selecting a library of catalysts was based on a stepwise screening including various simple and mixed oxides. They were selected on the basis of literature data analysis of performances as oxidation catalysts, not only for carbon but also for total oxidation of gaseous pollutants (hydrocarbons, volatile organic compounds). Their compositions were chosen to include specific solid properties like oxygen storage capacity and oxygen mobility, to increase the probability of discovering relevant and original catalyst formulations.

3.1. Soot oxidation in the presence of simple oxides

CeO_2, V_2O_5, SnO_2, Mn_2O_3, Bi_2O_3, and $K_2Al_2O_4$ were tested (Fig. 2) under tight contact with the soot.

Except for SnO_2, the soot ignition temperature is well below 400 °C with all the tested oxides. For comparison, the un-catalysed soot ignition temperature measured in similar conditions is 515 °C.

Mn_2O_3 and $K_2Al_2O_4$ show similar performances to HSA ceria. Wet impregnation of carbon blacks with manganese acetate (tan-

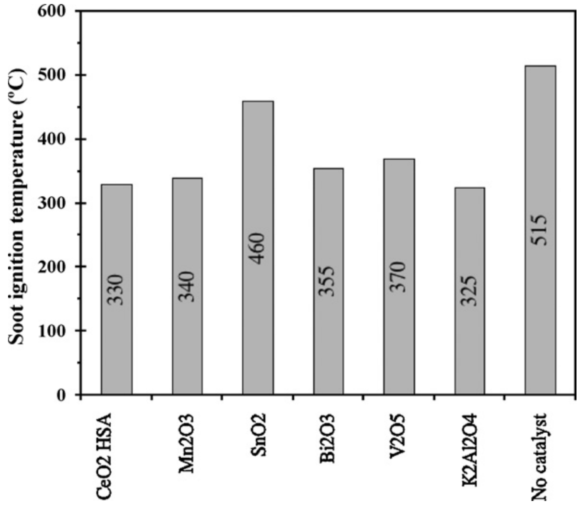

Fig. 2. Soot ignition temperature of simple oxides. Conditions: 40 mg soot, 400 mg catalyst, tight contact, heating rate 5 °C/min, 50% O_2 in He, total flow rate 40 mL/min.

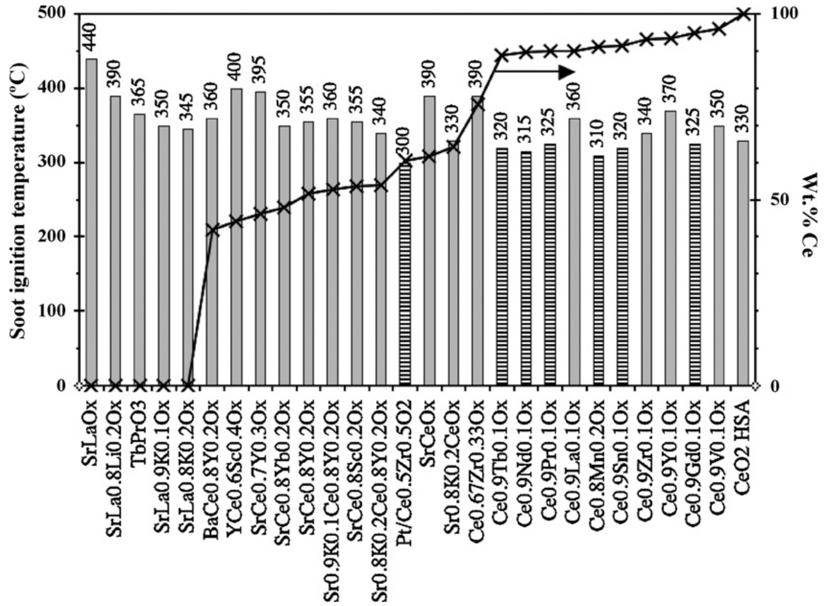

Fig. 3. Soot ignition temperature over rare-earth-based catalysts. Hatched bars highlight catalysts with a T_{ign} lower than that of HSA ceria.

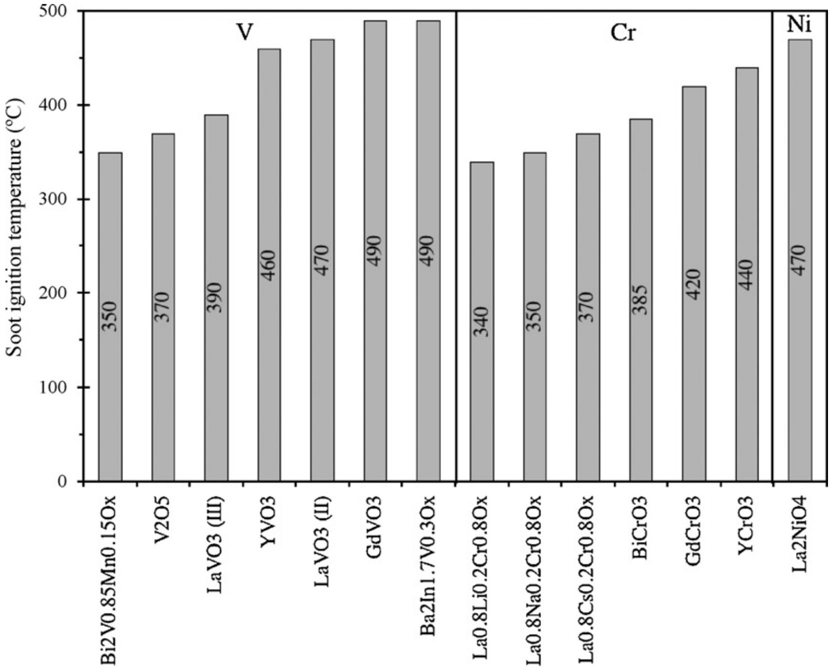

Fig. 4. Soot ignition temperature over V-, Cr- and Ni-based mixed oxide catalysts.

tamount to tight contact) has been shown to decrease the ignition temperature by 150–200 °C [13]. The high activity was ascribed to the formation of Mn_2O_3 in tight contact with the soot particles. When tested in loose contact, however, the effect of Mn_2O_3 on the carbon ignition temperature was not significant [13]. Recently, birnessite and cryptomelane (mixed oxides of Mn and K combining Mn(IV) and Mn(III) cations) were also found to be very active catalysts for carbon black combustion in loose contact conditions [14]. $K_2Al_2O_4$ confirms the strong effect of K in enhancing the activity. The promotion effect of alkali metals on the soot combustion activity has been related to the formation of a low melting point species (potassium carbonate, oxide or hydroxide), which enhance the soot/catalyst contact [15]. Potassium compounds, however, are relatively volatile, and although this property is beneficial for the mobility of active species [6,5], the main drawback is that potassium is lost in the exhaust line, leading to catalyst deactivation and to corrosion of the exhaust line.

3.2. Rare-earth-based oxides

Most of the catalysts tested were based on doped ceria, while five solids were based on lanthanum or praseodymium oxides. Fig. 3 presents the soot ignition temperature of the various solids tested, and the related cerium content in the solids (referred to the total metals content, excluding oxygen).

All catalysts exempt from cerium exhibit soot ignition temperatures in the range 345–440 °C, higher than HAS ceria (330 °C). Within those, the K-doped solids are the most active, the ignition temperature decreasing when the potassium content increases.

In the series of doped ceria with formula $Ce_{0.9}X_{0.1}O_x$ (X = V, Y, Zr, Sn, La, Pr, Nd, Gd, Tb) containing 89–100 wt.% Ce (metal-based), five solids appear more active than pure HAS ceria, which can be ranked in the following order of ignition temperatures:

$$\mathrm{Pr} = \mathrm{Gd} > \mathrm{Sn} = \mathrm{Tb} > \mathrm{Nd}$$

Pr- and Gd-doped ceria are known as solids with a mixed ionic-electronic conductivity [16]. This property, however, is not expected to contribute much to soot combustion activity, since it is effective at rather high temperatures (500–800 °C) compared to the soot oxidation temperature. Krishna et al. [17] observed that the soot combustion activity of low specific surface area ceria ($2\,m^2\,g^{-1}$) is strongly improved by Pr-doping. Despite the intrinsic reducibility of Pr, its role was essentially correlated to the higher surface area ($11\,m^2\,g^{-1}$) of the doped samples.

Doping ceria with manganese in the solid $Ce_{0.8}Mn_{0.2}O_x$ leads to a very active catalyst (soot ignition temperature 310 °C). Pure manganese oxide being slightly less active than pure ceria (Fig. 2), this suggests a synergetic effect between cerium and manganese oxides for soot combustion. A similar effect has been observed by Tikhomirov et al. [18] during soot oxidation assisted by NO over MnO_x–CeO_2 mixed oxides, which was attributed to an improved nitrate storage capacity of the mixed oxides, compared to the individual oxides. Upon heating, the nitrates were decomposed into NO_2, which oxidized the soot.

Despite the well-established improvement in the oxygen storage capacity of ceria induced by Zr substitution [19–21], the $Ce_{0.67}Zr_{0.33}$ catalyst is significantly less active for soot combustion than HSA ceria (T_{ign} = 390 °C). This contrasts with the results of Aneggi et al. [22] and Atribak et al. [23], who observed that ceria-zirconia solid solutions were more active than CeO_2 for soot combustion, for samples after thermal ageing. However, a direct comparison of HSA ceria (commercial catalyst, with a surface area of $217\,m^2\,g^{-1}$ after calcination at 600 °C) with the ceria-zirconia solid prepared in the present study ($70\,m^2\,g^{-1}$) is not straightforward, since the surface area has been show to influence the soot combustion activity of ceria-zirconia solid solutions [24]. Our results are consistent with a previous study on rare-earth doped cerias [17], which concluded that the reducibility and the bulk oxygen mobility are not critical parameters for soot combustion activity, but rather the amount of surface oxygen species transferred to the soot and their mobility by spillover. The most active catalyst in this series is Pt(1 wt.%)/$Ce_{0.5}Zr_{0.5}$, with a T_{ign} = 300 °C, which is also the only catalyst containing a noble metal in this study. However, the soot ignition temperature is only moderately affected by the presence of platinum, compared to very active ceria-based compositions in the series $Ce_{0.9}X_{0.1}O_x$ (X = Y, Sn, Nd, Tb). This suggests that the ability of platinum to adsorb and dissociate O_2 is not a decisive property to promote soot combustion activity, probably because the amount of Pt is low and the probability for a direct contact between soot particles and platinum is therefore very low. The presence of activated oxygen species at the ceria-zirconia surface (peroxide, superoxide species [25,26]), capable to diffuse at low temperature at the soot/catalyst interface, appears a more relevant parameter. The influence of platinum, however, would probably be more significant if the soot oxidation were performed in the presence of $NO + O_2$, since is this case the catalyst would operate on the principle of the NOx-aided continuously regenerated trap: Pt catalyses NO oxidation into NO_2, which is more reactive than O_2 towards the soot [27].

All the mixed oxides containing 42–64 wt.% Ce (excluding Pt/$Ce_{0.5}Zr_{0.5}$) are less active than pure ceria, with T_{ign} = 400 °C for $YCe_{0.6}Sc_{0.4}O_x$. The soot ignition temperature tends more or less to increase when the amount of cerium in the solid decreases. The most active catalysts are those doped with the highest amount of potassium (T_{ign} = 330 and 340 °C for $Sr_{0.8}K_{0.2}CeO_x$ and $Sr_{0.8}K_{0.2}Ce_{0.8}Y_{0.2}O_x$, respectively, which contain 3.6 and 3.8 wt.% K on a metal basis, respectively).

3.3. Transition metal-based oxides

3.3.1. V-, Cr- and Ni-based oxides

Fig. 4 shows the soot ignition temperature obtained with a series of V-, Cr-, and Ni-based oxides.

All the vanadium-based solids are less active than HAS ceria. The T_{ign} of pure V_2O_5 is 370 °C, and except for $Bi_2V_{0.85}Mn_{0.15}O_x$ (T_{ign} = 350 °C), all the other mixed oxides exhibit higher ignition temperatures. The $Bi_2V_{0.85}Mn_{0.15}O_x$ catalyst belongs to the family of BIMEVOX oxides, which exhibit a high ionic conductivity at moderate temperature [28]. Oxide ion mobility, however, is effective at temperatures higher than 500 °C, and is not expected to account for the low soot ignition temperature observed with this catalyst. The activity of this catalyst can be attributed to the presence of bismuth and manganese (Bi_2O_3 and Mn_2O_3 are active at lower temperatures than V_2O_5, see Fig. 2).

$LiCrO_2$ [29] and $La_{0.8}Cr_{0.8}Li_{0.2}O_3$ [9] have been shown to be efficient catalysts for diesel soot oxidation at low temperature. Although the activity of $La_{0.8}Cr_{0.8}Li_{0.2}O_3$ is very close to that of HSA ceria under our testing conditions (T_{ign} = 340 °C), all Cr-based mixed oxides tested in the present study are less active than HSA ceria. Alkali-doped La–Cr perovskites show the best activity ($T_{ign} \leq 370$ °C), with a promotion effect of alkali metals in the range Li > Na > Cs. With $BiCrO_3$, $GdCrO_3$ and $YCrO_3$, the soot ignition takes place at higher temperatures (385–440 °C).

The La_2NiO_4 spinel-type oxide has only a modest influence on the ignition temperature, which decreases by only 45 °C compared to pure soot self-ignition.

3.3.2. Mn-, Fe- and Co-based oxides

The soot ignition temperature in the presence of Mn-, Fe- and Co-based mixed oxides is depicted in Fig. 5.

Mn-based oxides appear altogether very active in catalyzing diesel soot combustion, the ignition temperature being lower than 400 °C with all the solids tested. $BiMnO_3$ and $LaMnO_3$ exhibit ignition temperatures lower than that of HSA ceria, i.e. 300 and 320 °C, respectively. The combination of Bi and Mn in a mixed oxide gives the most active catalyst in this series, with a soot ignition temperature as low as that of Pt/$Ce_{0.5}Zr_{0.5}$. $BiMnO_3$ is also more active than the simple Bi and Mn oxides (Fig. 2), suggesting a synergetic effect between these two metals when incorporated in a mixed-oxide structure. Interestingly, this synergy does not seem to operate between Bi and Fe (T_{ign} = 350 °C for $BiFeO_3$), nor between Bi and Cr (T_{ign} = 385 °C for $BiCrO_3$).

Fe- and Co-based mixed oxides based on perovskite-type structures are also fairly active for the catalytic combustion of diesel soot ($T_{ign} \leq 360$ °C, except for $YFeO_3$ which shows a T_{ign} of 410 °C). Only $Ba_{0.5}Sr_{0.5}Fe_{0.6}Bi_{0.4}O_x$, however, is more active than HSA ceria (T_{ign} = 310 °C), which cannot be related only to the bismuth present in this solid, since $BiFeO_3$ is significantly less active (T_{ign} = 350 °C).

3.4. Catalyst characterization

Among the various catalysts tested in the present study, around 25 were submitted to X-ray diffraction analysis. The diffractograms revealed that some samples were well crystallized and corresponded to the targeted oxide phase (i.e. $LaFeO_3$, $La_{0.8}Cr_{0.8}Li_{0.2}O_3$), whereas others were mostly amorphous. Other samples actually consisted in mixtures of crystallized phases, particularly the Sr-containing samples which always exhibited the diffraction lines of strontium carbonate. Although the synthesis procedure by the

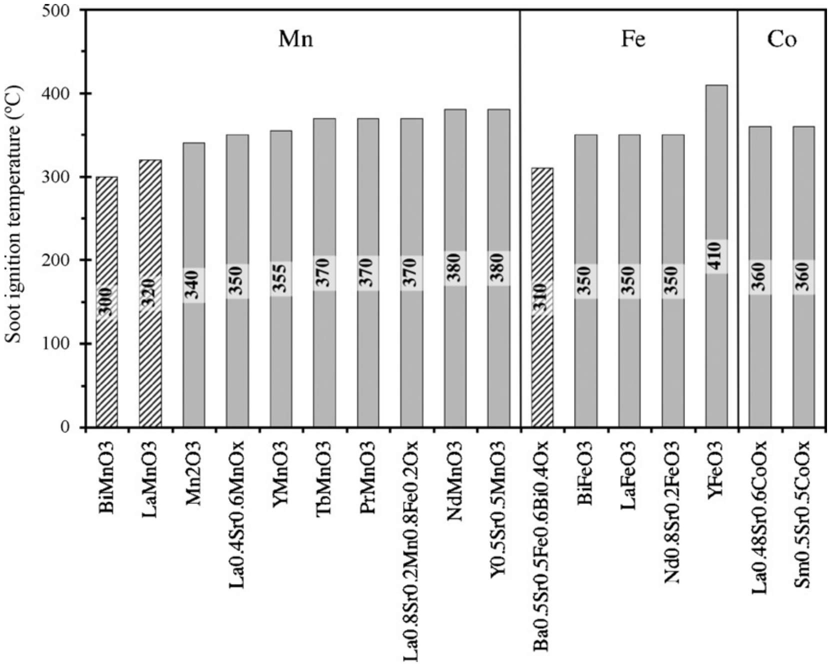

Fig. 5. Soot ignition temperature over Mn-, Fe- and Co-based catalysts.

citrates-gel method is very general, the calcination step clearly requires a specific adjustment for each solid, as long as single phase formation is targeted.

The diffractogram of the most active composition, targeted as the bismuth manganite $BiMnO_3$, is shown in Fig. 6. Most of the diffraction lines actually correspond to a Mn_2BiO_5 phase (JCPDS file 04-011-5148), whereas the remaining lines can be attributed to two different crystallographic Bi_2O_3 phases (monoclinic: JCPDS file 00-041-1446 and tetragonal: JCPDS file 01-074-1374).

The "$BiMnO_3$" solid was submitted to DTA/TGA measurements in air, up to 850 °C, in order to check its thermal stability. A slow and continuous mass loss was observed between 50 and 700 °C, which remained however <1.1% of the total mass and was not associated to clear endo/exo peaks. This is probably related to a slow dehydration/dehydroxylation of the solid, which appears stable up to 850 °C. However, further characterizations and tests of various Bi/Mn ratios are be necessary to identify the optimal composition of this catalyst, and to relate its activity to a single crystallographic phase (Mn_2BiO_5) or to the mixture of phases and their relative ratios. Combinatorial methods should here offer powerful tools to reach this goal. In addition, this suggests that the high activity of this catalyst formulation might be related either to an optimal composition of the solid solution Mn_2BiO_5 leading to high mobility of surface oxygen species (appropriate strength of the metal oxygen bonds, presence of oxygen vacancies [30]) or to a synergetic effect between physically mixed oxide phases (Mn_2BiO_5 and Bi_2O_3) on the activation of mobile oxygen species, similarly to the "remote control mechanism" evidenced by Delmon and Froment [31] for selective oxidation catalysis on oxides.

3.5. Effect of soot/catalyst contact

In order to assess the catalyst performances under more realistic conditions, HSA ceria and $BiMnO_3$ were evaluated under loose contact with the soot, i.e. by mixing the catalyst powder and the soot with a spatula. The results are shown in Fig. 7.

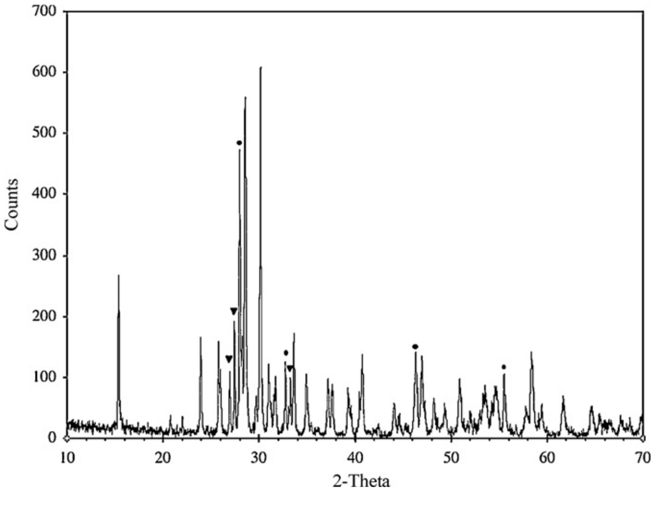

Fig. 6. X-ray diffractogram of the "$BiMnO_3$" mixed oxide fresh catalyst. Unmarked lines: Mn_2BiO_5 phase; (▼) Bi_2O_3 (monoclinic); (●) Bi_2O_3 (tetragonal).

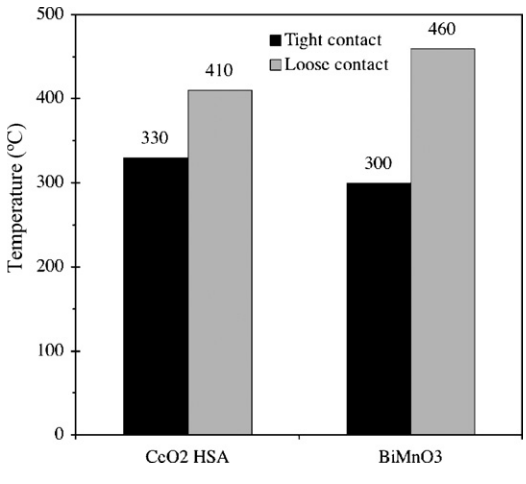

Fig. 7. Comparison of soot ignition temperatures over HSA ceria and $BiMnO_3$ catalysts under tight or loose contact.

As expected, the soot ignition temperatures are higher in loose contact than in tight contact: it increases by 80 °C for ceria, but by 160 °C for $BiMnO_3$. This might be related to a larger amount of soot/catalyst contact points with HSA ceria, due to the small ceria particle size. We have characterized previously the average particle size of HSA ceria (20 μm) and of the Diesel soot (40 μm) [8]. This suggests that, even in loose contact, the small ceria particles should spread around the larger soot particles. In contrast, the particle size of the "$BiMnO_3$" powder ranges from 100 to 300 μm, with a mean diameter of 200 μm. Consequently, the number of contacts is probably critical under loose contact conditions. This underlines the importance of an appropriate particle size for solid–solid contact, and suggests that the particle size of the catalyst powder should be controlled for an optimal contact with the soot.

Although the loose contact should be more representative of realistic conditions than tight contact, these results should however be considered with caution, since we also observed that the ignition temperatures measured under loose contact conditions were poorly reproducible [8]. This is probably related to the difficulty of preparing a reproducible mixture of catalyst and soot when mixing with a spatula.

4. Conclusion

High-throughput screening of diesel soot combustion catalysts by identification of the soot combustion ignition temperature has proved a valuable technique to rapidly identify efficient catalysts among a broad set of formulations. Appropriate testing conditions have to be carefully selected in order to obtain reliable results.

Among the 62 catalyst formulations tested, 9 compositions exempt of noble metals were found more active than the reference catalyst, HSA ceria (Rhodia). Six of them were based on doped ceria, but two original formulations based on Bi–Mn or Bi–Fe mixed oxides were also discovered.

This screening also highlights that some catalyst properties appear un-relevant to the activity for soot combustion: high lattice oxygen ion mobility as well as high oxygen storage capacity do not significantly improve the catalytic activity. Key parameters for low-temperature soot ignition appear rather related to the presence of mobile surface oxygen species, and to an appropriate catalyst particle size allowing a maximum of physical contacts with the soot particles.

From this primary screening, which identified "hit" compositions, combinatorial methods will now be applied to identify the favourable elements combinations and optimize catalysts composition. Further characterizations of the best catalysts will also be required to assess the relationships between the physico-chemical properties of the solids and the catalytic activity.

Acknowledgments

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