Mobile and Non-mobile Catalysts for Diesel-Particulate Combustion: A Kinetic Study

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Abstract−One of the potential ways to solve the problem of diesel particulate emission from both stationary and mobile sources is the use of traps carrying a suitable catalyst for promoting particulate combustion as soon as it is filtered. A Cu-K-V based catalyst, considered among the most promising in the literature, the KVO_3+CsCl and the $K_{0.7}Cu_{0.3}VO_3+KCl$ catalysts were prepared and investigated. Their performance was compared to a reference V_2O_5 catalyst. The superior performance of the Cu-K-V catalyst is based on the grounds of both microreactor (temperature programmed combustion) and catalytic trap tests. Based on experimental data and modelling calculations, this paper elucidates how the mobility of catalyst components is the main reason for such an outcome performance and is a pre-requisite to achieve an activity sufficient for trap self-regeneration.

Key words: Diesel Particulate, Mobile Catalysts, Combustion, Kinetics, Cu-K-V Catalysts

INTRODUCTION

The continuous growth of the diesel engine market has brought to the urban areas several environmental effects [Rah, 1984, 1985]. Diesel engine emissions are harmful for human health, especially as a consequence of their high particulate content. Diesel particulate consists of spherical carbon particles with a diameter ranging from 0.01 to 0.08 μm (soot), on whose surface condensed hydrocarbons are adsorbed. Because of its small dimensions, diesel particulate raises a special health concern: small particles are in fact more likely to be deposited in the alveolar region of the lungs and require much longer periods of time to be cleared away from the respiratory tract than the larger ones. Moreover, diesel particulate has been pointed out as particularly hazardous due to its composition: the carcinogenic effect is related both to the solid carbon nuclei and to the adsorbed hydrocarbons.

In order to maintain the air quality under tolerable levels, owing to the continuous growth of the diesel-vehicle market, many countries are tightening their regulations of diesel emissions (DieselNet web site) not only for land transports [Kandylas and Stamatelos, 1997], but also for those industries [Fino et al., 2002b] that produce flue gases rich in carbon particles having a negative impact on the air quality [Schlesinger, 1989], which affects both the environment and human health. In particular, the US, Europe and Japan have recently adopted particulate emission standards which will require significant technological progress in the abatement of these substances. EURO IV standards will be in force from 2005 and will impose particulate emission levels lower than 0.025 g/km. This will force car manufactures to adopt a catalytic aftertreatment device.

One of the possible ways to eliminate this pollutant is that of trapping it and simultaneously burning it out in catalysed deep-filtration traps before releasing the flue gases into the environment [Ambrogio et al., 2001; Fino et al., 2002a; Saracco and Fino, 2001; van

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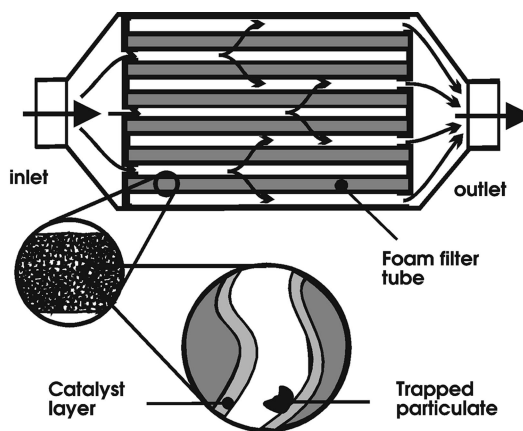E-mail: debora.fino@polito.it

Fig. 1. Scheme of a catalytic trap based on a shell-and-tube assembly of tubular foam filters.

Gulijk et al., 2001]. By using such catalytic filters, particulate emissions could in principle be reduced up to 85% [Bar Ilan et al., 1995].

Once suitable catalysts are deposited over the pore walls of the trap, these structures are able to collect the soot particles bringing them in contact with a catalyst that can favour their oxidation [Saracco et al., 1999b]. Fig. 1 shows a scheme of a trap based on ceramic foam tubular filters [Matsunuma et al., 1996].

From previous studies conducted at the Politecnico di Torino the present contribution shows the results concerning the development of suitable catalysts for the envisaged application. In this context, very promising catalysts are: the Cu-K-V [Watabe et al., 1983; Ciambelli et al., 1994, 1996] and Cu-K-Mo-Cl [Neeft et al., 1997] ones. Earlier studies concerned the stability of these catalysts [affected by loss of chlorides at high temperatures; Badini et al., 1996], its chemical characterisation [Badini et al., 1998a] and the analysis of its reaction mechanism [Serra et al., 1997]. $CuCl_2$, one of the Cu-K-V catalyst components, raises a special concern due to its intrinsic toxicity and its well known oxychlorination activity which can lead, in contact with diesel exhausts, to the formation of toxic chlo-

rinated compounds, such as dioxins [Luijk et al., 1994]. For this reason, further investigations were then addressed to binary catalyst so as to discover catalysts possibly as active as the Cu-K-V one, but less detrimental to the environment. Two different approaches were followed. A first class of materials was based on single vanadates, suspected to be primarily responsible for the catalytic activity, in combination with KCl [Badini et al., 1996]. A second class of catalysts was based on KVO_3 coupled with different halides [Saracco et al., 1997].

The major goal of this communication is to demonstrate how the mobility of the catalyst components owing to formation of eutectic liquids is an essential condition in combination with catalyst stability to ensure feasibility of the catalytic trap concept (Fig. 1). For such a purpose, new experimental runs were performed on the most promising catalysts of the two above classes (i.e., KVO_3+CsCl and $K_{0.7}Cu_{0.3}VO_3$+KCl), on the Cu-K-V catalyst, and on V_2O_5 used as a reference material which, as opposed to its former counter parts, does not form low temperature melting liquids. A model was also assembled, as hereafter described, for the prediction of the performance of the catalytic traps. Special attention is paid to the role of mobility of active species on the catalyst and catalytic trap performance, assessed by both experimental and modeling tools.

EXPERIMENTAL SECTION

The Cu-K-V based catalyst was prepared by impregnation of a support of α-aluminium oxide powder (50 wt%) with solutions of KCl, NH_4VO_3 and $CuCl_2 \cdot 2H_2O$ so as to achieve the optimum Cu : K : V atomic ratio of 2 : 2 : 1. Impregnation was followed by drying at 120 °C and calcination at 700 °C, leading to several reactions (decomposition, synthesis of new compounds, etc.) among the catalyst precursors, which give rise to the active components [mostly KCl and copper and potassium vanadates, Badini et al., 1997].

The $K_{0.7}Cu_{0.3}VO_3$+KCl catalyst was prepared by impregnation of α-Al_2O_3 powder with a water solution of these two compounds in a 1 : 1 molecular ratio (50 wt% of alumina). Afterwards, the above drying and calcination steps were performed. The KVO_3+CsCl catalyst was prepared according to the same procedure.

On the three above catalysts twin differential scanning calorimetry (DSC) runs were performed, some measuring the heat released by the catalytic combustion of carbon, some performed on a pure catalyst sample in order to detect any endothermic peak attributable to liquid formation.

Moreover, the effect of the catalyst-to-carbon ratio on the DSC peak temperature T_p (temperature at which carbon conversion rate is maximum) was assessed for any of the three selected catalysts. T_p was regarded as an index of catalytic activity. The lower the T_p value, the higher the catalytic activity.

DSC runs were performed on catalyst-carbon mixtures (obtained by careful mixing in an agate mortar) according to the experimental procedures described by Serra et al. [1996]. In the activity tests, amorphous carbon (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) was preferred over diesel particulate in order to get higher reproducibility. Diesel particulate is in fact characterised by a variable content of adsorbed hydrocarbons, which favours, to some extent, carbon combustion. In each test, 10 mg of the catalyst-carbon

mixture was submitted to a linear rise of temperature (scanning rate: 10 °C/min) in the DSC apparatus (Perkin Elmer DSC 7). Air was delivered to the calorimetric cell at a flow rate (7.5 ml/min) which was enough to provide a large excess of oxygen. The DSC measurements were performed using α-Al_2O_3 as a reference.

A counterpart of the above low-temperature melting and mobile catalysts, $V_2O_5 \cdot \gamma$-Al_2O_3 was also considered in the present study on the grounds of the experimental work by Ahlström and Odenbrand [1990].

Finally, some comparative experimental tests were carried out on zirconia toughened alumina foam traps (pore size=50 pores per inch, diameter=70 mm; thickness=17 mm; overall density=904 kg m^{-3}; thermal capacity=1.16 J·$kg^{-1}K^{-1}$; thermal conductivity=1.13 W·$m^{-1}K^{-1}$) lined with 3 wt% of each of the above considered catalysts according to the procedure described in [Saracco et al., 1999a]. Particularly, the pressure drop across the traps (a sign of soot accumulation therein) was monitored as a function of time meanwhile feeding them at a constant temperature (460 °C) with a constant flow rate of particulate-laden flue gases (0.106 g/m^3 at 0.7 m/s superficial velocity) derived from an acetylene burner operated sub-stoichiometrically. This pilot plant is described in detail in Saracco et al. [1999b] where more details on the experimental procedures can be found.

RESULTS AND DISCUSSION

The results of the twin DSC runs are reported in Fig. 2. Several
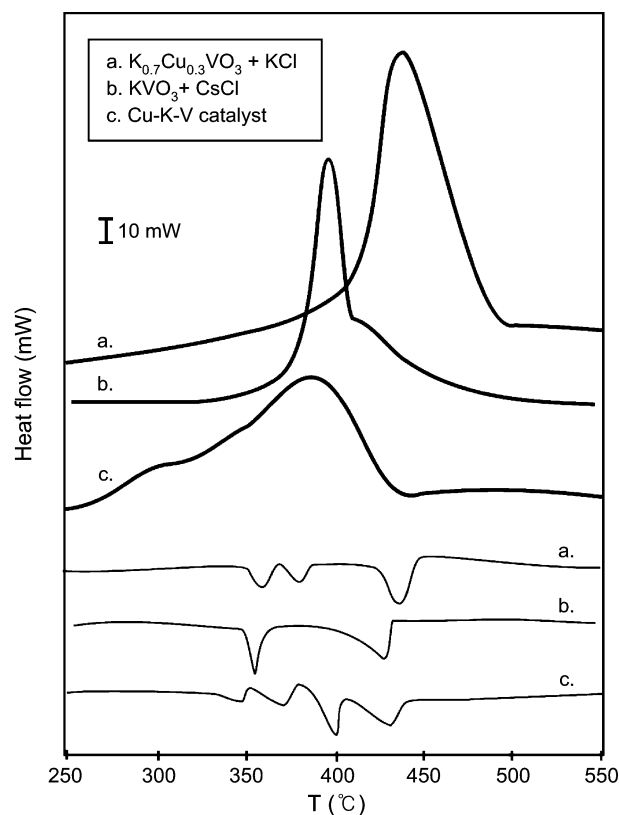

Fig. 2. Results of DSC combustion (thick lines) and melting (thin lines) runs on three selected catalysts [adapted from Serra et al., 1996; Saracco et al., 1996]. Catalyst to carbon weight ratio in the combustion runs=1 : 1.

endothermic peaks were found for each catalyst, which were attributed to the formation of different eutectic liquids between the different catalysts components generated during calcination. The formation of the above eutectic liquids occurs at the same temperature at which the catalyst displays its activity. It can therefore be argued that the reaction mechanism of the three catalysts is the same: above certain temperatures eutectic liquids are formed among the various catalyst components; such liquids should wet diesel particulate and promote its combustion through redox processes enabled by the presence of vanadates.

Since the contact under practical conditions when filtering soot with a catalytically coated trap is rather poor, Neeft et al. [1997] performed combustion experiments on mixtures either prepared by shaking soot and catalyst in a sample bottle ("loose contact"), so as to simulate the contact conditions typical of a trap, or by ball-milling the components in an agate mechanical mill ("tight contact"), so as to define an intrinsic catalytic activity under optimal contact conditions. Some catalysts showed a high or moderate activity in tight as well as in loose contact mode, while others found to be active in tight contact mode, showed no significant activity in loose contact mode. On the basis of the above considerations, Neeft and co-workers concluded that the intrinsic catalytic activity is not the only requirement needed to obtain superior activity, but that mobility of the catalytic material is an important prerequisite for applications in diesel exhaust treatment. In Fig. 3 the possible contact conditions for non-mobile catalysts (such as noble metals or V_2O_5) and for mobile catalysts are sketched. Moreover, it is enlightening that two different mobility mechanisms exist. Catalysts forming volatile com-
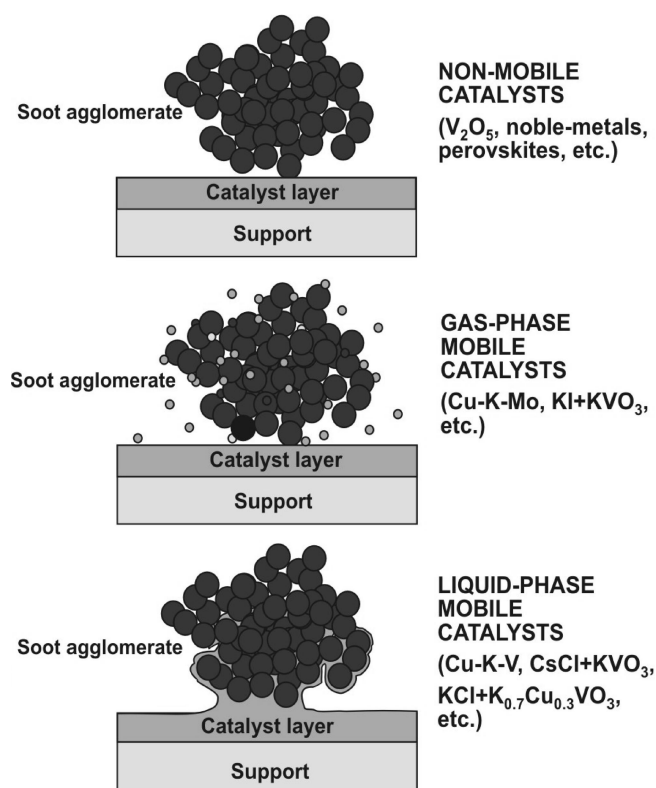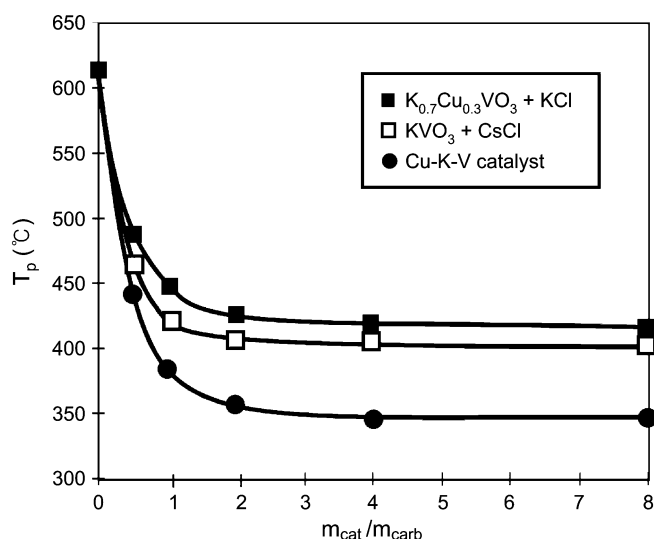

Fig. 4. DSC peak temperatures vs. catalyst-to-carbon weight ratio (m_{carb}=1 mg) for the three catalysts tested.

pounds at their operating temperatures (such as a Cu-K-Mo based catalyst or KI coupled with KVO_3, Badini et al., 1998b) work through a gas-phase transport, while catalysts forming liquids at their operating temperatures (such as the materials here tested) work through a moving liquid-phase contact. The first option is not suitable for diesel engine applications. Due to the high space velocities of the exhausts, loss of catalyst is actually unavoidable. Conversely, the materials, which show liquid-phase mobility, seem to have potential of practical application, if they are stable under working conditions.

Further strength to the hypothesis of mobility characteristics due to liquid formation of Cu-K-V, KVO_3+CsCl and $K_{0.7}Cu_{0.3}VO_3$+KCl catalysts is given by the tests to assess the effect on the catalyst-to-carbon weight ratio on the catalyst activity. The results obtained with DSC combustion runs are shown in Fig. 4: when the above ratio is increased, the DSC peak temperature is at first significantly lowered, but beyond a 2 : 1 weight ratio no further appreciable decrease is observed. Two main comments can be drawn concerning the data shown in Fig. 4:

• For low catalyst-to-carbon weight ratios (below 1 : 1) the carbon is simply too much to achieve an efficient contact with the catalyst. Probably, a part of carbon is ignited immediately by the catalyst; afterwards, the combustion of the rest of the carbon is delayed because a certain time-lag is required to get in touch with the catalytic species;
• For higher catalyst-to-carbon ratios the catalytically active liquid phases are capable of getting rapidly in contact with considerable fractions of the total amount of the carbon present. For ratios higher than 2 the maximum catalyst performance is nearly achieved (all the carbon is reached with negligible time-lag, despite the fact that, due to the remarkable difference between catalyst and carbon particle sizes, only a very small fraction of carbon is directly touching the catalyst surface).

A peculiar phenomenon can be observed for all the tested catalysts: above a critical value of the catalyst-to-carbon weight ratio,

Fig. 3. Mobile and non-mobile catalysts: sketches of the soot-catalyst contact conditions.

$(m_{cat}/m_{carb})^*$, approximately equal to 4 for the Cu-K-V catalyst and perhaps less for the other two catalysts studied, T_p is no longer influenced by the catalyst mass used in the DSC experiments. Therefore, it can be concluded that beyond a certain m_{cat}^* value, referred to the amount of carbon available, the mass of catalyst should no longer influence reaction kinetics.

The particular behaviour observed can be explained through mobility of the catalyst components, which governs, as above underlined, the reaction mechanism. For a given carbon mass, a critical mass of catalyst exists which generates enough liquids to reach the entire geometrical surface exposed by the carbon particles with negligible time lag. If the catalyst were not mobile, an analogous limiting condition would be achieved when all the carbon particles are in contact with the catalyst (monolayer catalyst coverage). However, it can be demonstrated [Serra et al., 1996] that the critical $(m_{cat}/m_{carb})^*$ ratio would be well above 50, for typical values of the carbon (~1 μm) and catalyst (~10 μm) particle diameter. Similar runs were also performed by Ahlström and Odenbrand [1990] employing a $V_2O_5 \cdot \gamma\text{-}Al_2O_3$ catalyst, which did not form liquid phases at its best operating temperatures. Their results (Fig. 5) clearly show that the carbon conversion rate per unit carbon mass (R_c) remains proportional to m_{cat}/m_{carb} at least up to a value of 20, well above the $(m_{cat}/m_{carb})^*$ values detected for the catalysts here studied.

Based on the above considerations a mobility index was defined as:

$$MI = \frac{100}{W_{cat}(m_{cat}/m_{carb})^*}, \tag{1}$$

where W_{cat} is the percent weight fraction of catalytically active species in the overall catalyst mass, which includes the inert support. The higher the catalyst mobility, the higher the mobility index.

It has to be admitted that the mobility index determined via Eq. (1) on the grounds of the experimental data shown in Fig. 4 might not be very representative of what takes place inside a real catalytic trap for several reasons:

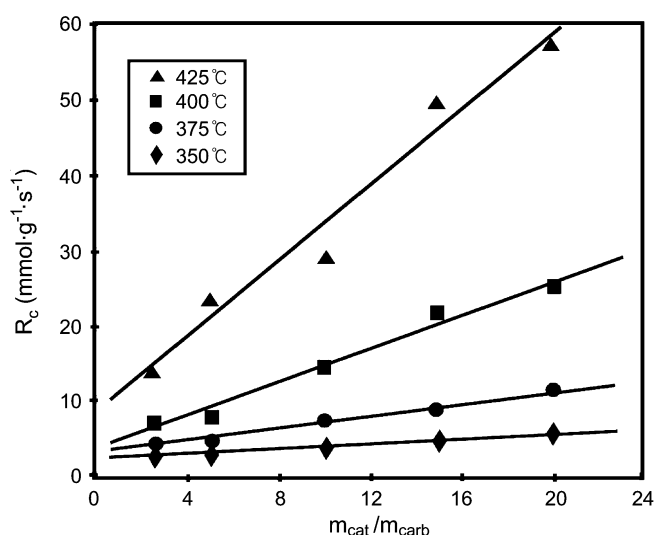- ball-mill mixing of carbon and catalyst leads to quite reproduc-ible results but the obtained *tight-contact* conditions between these two counterparts are often too intensive compared to what occurs inside a catalytic foam operating on diesel exhaust gases;

- the ball milling procedure may lead to rupture of the catalyst or carbon agglomerates changing their particle size, which should have an influence on the catalyst performance and, to some extent, on the mobility index, as well.

Nonetheless, a model was assembled and validated by fitting the data published by Watabe et al. [1983] and by Matsunuma et al. [1996]. The model, thoroughly described in Ambrogio et al. [2001], includes differential heat and mass balance equations inside a catalytic foam trap. Particularly, inertial impaction, interception and brownian diffusion were considered as basic mechanisms for diesel particulate trapping [Oh et al., 1981], whereas literature data were used for the kinetics expression [Ciambelli et al., 1994, 1996]:

$$-\frac{dm_{carb}}{m_{cat}dt} = kS_t p_{O_2}^{1/2} \tag{2}$$

with $k = A \cdot \exp(-E_a/RT)$, $\tag{3}$

where m_{carb} and m_{cat} are the carbon and catalyst masses brought into contact, k the kinetic constant, S_t the geometrical surface area of the carbon particles, p_{O_2} the oxygen partial pressure. The values of the
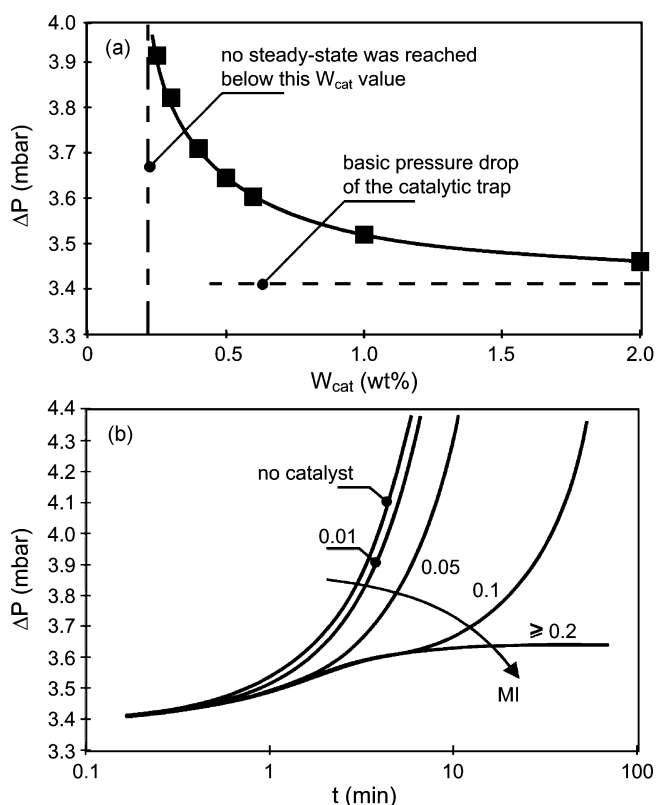

Fig. 6. Model results. (a): effect of the catalyst weight load of the foam trap on the steady state pressure drop (MI=0.5); (b): effect of MI on ΔP variation vs. time (W_{cat}=0.5 wt%). Feed flow rate=150 kg/h; particulate concentration=0.05 g/m³; O_2 inlet concentration=6 vol%; average particulate size= 0.2 μm; inlet temperature=450 °C; filter porosity=70%; average pore radius=200 μm; total filter area=0.18 m²; filter thickness=5 mm.

Fig. 5. Combustion rate per unit carbon mass (R_c) vs. the m_{cat}/m_{carb} ratio when using a non-mobile catalyst (V_2O_5) (adapted from Ahlström and Odenbrand, 1990).

pre-exponential factor A and of the activation energy E_a are $4.86 \cdot 10^{-3}$ $g_{carb} \cdot g_{cat}^{-1} \cdot min^{-1} \cdot m^{-2} \cdot atm^{-1/2}$ and 71 kJ·mol^{-1}, respectively, for the Cu-K-V catalyst. Since for mobile catalysts a critical m_{cat}^* value (referred to as the amount of carbon available) exists, above which the mass of catalyst does not longer influence the reaction kinetic, m_{cat} can be replaced with m_{cat}^* in the first member of Eq. (2).

Simulations (Fig. 6) were then performed on the grounds of the model thoroughly described by Ambrogio et al. [2001] in order to assess the role of the load of catalytic components (W_{cat}) in such a foam filter as that drawn in Fig. 1, and of MI on the pressure drops across the filter (the higher the pressure drops, the higher the particulate amount deposited in the trap). The parameters used in model calculations are listed in the figure caption. In particular, the reaction kinetics of the Cu-K-V catalyst were used. As concerns Fig. 6b, the mobility index was varied below its original value 0.5, so as to elucidate the effect of this parameter on the trap performance.

Fig. 6a shows that the lower the W_{cat} the higher the pressure drops across the filter at steady state conditions (generally reached within 1 hour). Further, a limiting W_{cat} value was found below which no steady state can be reached: the trap is rapidly plugged since it is not active enough to burn out all the trapped particulate. Fig. 6b stresses the importance of the catalyst mobility on the performance of the catalytic trap. For the given values of the operating parameters, an MI value higher than 0.1 is strictly required in order to avoid rapid plugging of the trap. For very low MI values, typical of non-mobile catalysts (i.e. ~0.05), the catalytic trap does not behave quite differently from the non-catalytic one.

Such conclusions drawn by modelling means are confirmed by the results obtained from catalytic filtration tests performed on foam traps. The data plotted in Fig. 7 do confirm that the presence of a non-mobile catalyst such as V_2O_5 does not significantly improve the performance of the non-catalytic trap. In both cases the pressure drop rises as long as the filtration process goes on, a sign that the particulate conversion rate is not sufficient to compensate for the hold-up increase due to soot capture. Conversely, the mobile Cu-K-V catalyst does enable so high particulate conversion rates that a balance point between the trapped and the burned soot can be found, resulting in steady pressure drops and continuous trap self-regeneration.

As an obvious conclusion, it can be stressed that mobile catalysts have to be adopted if the catalytic trap concept has to become practical. In this context, issues like catalyst chemical and thermal stability become of crucial importance [Badini et al., 1998].

CONCLUSIONS

Catalyst mobility is a necessary prerequisite for its application as a coating for diesel traps. It has been shown that the catalysts tested displayed mobility due to the formation of eutectic mixtures that melt at the operating temperatures and promote the wetting of the carbon particles. In particular, a parameter has been conceived so as to define this property, called the Mobility Index (MI). Model calculations and experimental tests based on a catalysed foam trap put into evidence the importance of the catalyst mobility on the performance of the catalytic trap. It is shown that a certain mobility level of the catalytically active species is required in order to avoid rapid plugging of the trap and that non-mobile catalysts do not significantly improve the performance of the virgin trap itself.

Nevertheless, the application of a mobile catalyst for the aftertreatment of diesel exhausts requires a careful evaluation of its stability, due to the severe operating conditions (temperature peaks, high space velocities, etc.).

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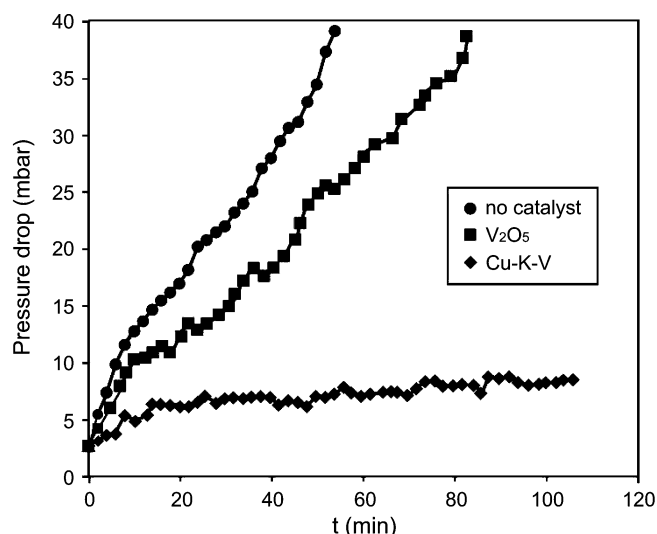
Fig. 7. Pressure drop vs. time for a virgin trap and traps deposited with a mobile (Cu-K-V) and a non mobile (V_2O_5) catalyst. Operating conditions: soot concentration=0.106 g/ Nm^3; temperature=460 ℃; outlet pressure=1 bar; catalyst in the foam=30 wt%; superficial velocity=0.7 m/s.

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