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Automotive Catalysts: Performance, Characterization and Development

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1. Introduction

Nowadays, automotive catalysts have been used to reduce atmospheric emissions, due to significant parcels of the global emissions of pollutants agents provoked by vehicles. Automotive exhaust catalysts were introduced in the 70's decade, because some countries established restricting laws related to emissions of carbon monoxide (CO), nitrogen oxides (NO_x) and hydrocarbons (HC) by the engines. These products generated by the combustion process are extremely harmful to health and the environment (Massad et al, 1985). For example, CO combined with hemoglobin in the bloodstream promotes the reduction of oxygen-active sites that provokes asphyxia. Nitrogen dioxide (NO₂) contributes to photochemical smog and acid rain, and is irritating to the eyes, skin and respiratory system. Nitrogen monoxide (NO) is toxic by inhalation and irritating to the eyes and skin. Polycyclic aromatic hydrocarbons (PAHS) have been identified as carcinogenic compounds. Other combustion products are: ash, formed mainly by particulate residues of components of the lubricating oil, and soot, combustible matter in the exhaust gas (smoke). Automotive catalytic converters have been developed precisely to make these products less toxic (Morterra et al, 1995; Ismagilov et al, 1998).

The development of catalysts only was possible with the improvement in automotive engines as the replacement of carburetion system for electronic injection and introduction of the catalyst in the exhaust systems (Kaspar et al, 2003).

The catalysts of three ways (TWC -Three-Way Catalyst) are advanced systems of emission treatment of gasoline vehicles that reduce significantly the emissions of carbon monoxide (CO), hydrocarbons (HC) and nitrogen oxides (NO_x) in atmosphere (Collins & Twigg 2007). Nevertheless, there is a steady increase in world production of vehicles powered by gasoline and by other types of fuel such as alcohol, gas and mix of fuel, leading to constant research in order to improve the catalysts already known and to develop new models (Mizukami et al, 1991; Silva et al, 2009 & Sideris, 1997).

Automotive catalysts are generally available in the form of monolith ceramic as cordierite and zeolites or metal substrate. The catalyst substrates more used are composed of magnesium cordierite (2MgO.2Al₂O₃.5SiO₂) with a honeycomb structure, which provides a high geometric surface area, coated with γ -alumina (catalyst wash-coat). This wash-coat is designed to increase the specific surface area and is the support for precious metals, mainly platinum (Pt), palladium (Pd) and rhodium (Rh), which promotes the catalytic reduction and oxidation of

pollutants gases to more harmless ones, like carbon dioxide, water and nitrogen molecular. Cerium and zirconium oxides are used in the coating by their oxygen storage capability to improve catalytic efficiency. (Angelidis & Sklavounos, 1995). A variety of other additives also are used to stabilize the alumina wash-coat at high temperatures (operational temperature). Figure 1 illustrates the operation of a three-way catalyst. This catalyst transforms the toxic gas CO in CO₂, which although not so toxic contributes to the increase in greenhouse effect.

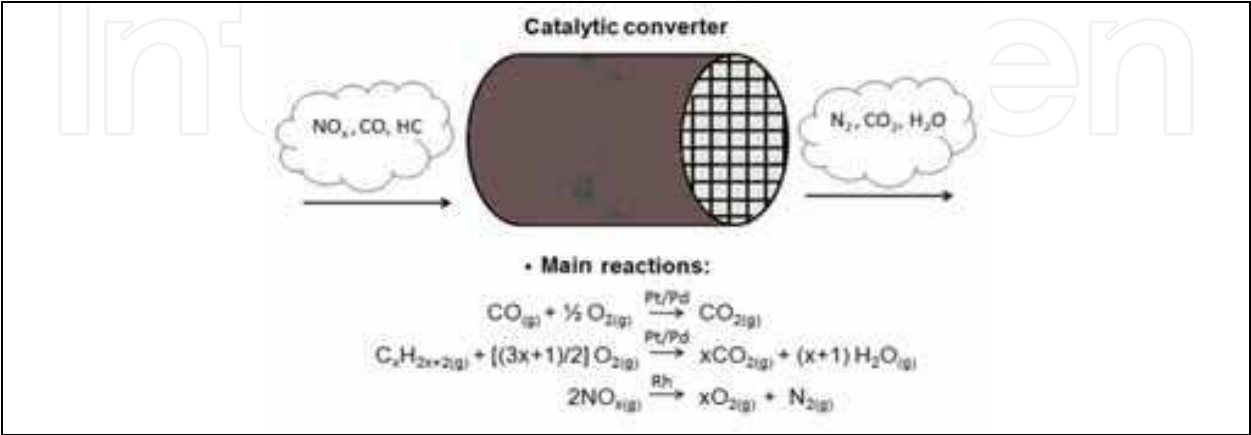


Fig. 1. Schematic illustration of the TWC operation.

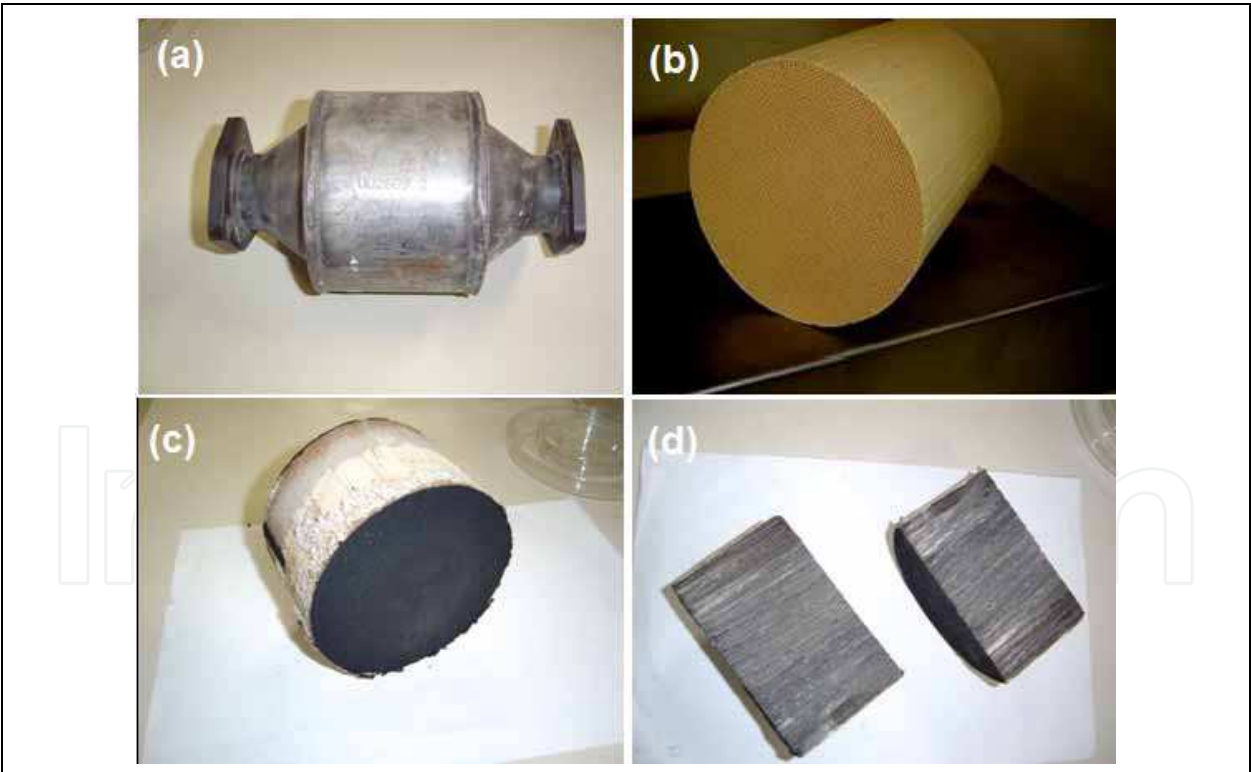


Fig. 2. (a) Catalyst module, (b) new catalyst (c) poisoned catalyst (d) samples of poisoned catalyst.

The catalyst can be deactivated by chemical, mechanical or thermal phenomena after some time of operation, depending on the composition of the used fuel and lubricants, and of the vehicle adjustment. The chemical deactivation can be promoted by poisoning due to the

chemisorptions of compounds on the surface of the catalyst; and due to chemical reactions that produce volatile compounds or inactive phases. The thermal deactivation occurs due to the sintering process and active metal diffusion. The mechanical deactivation is due to the deposition of particles from the gas phase onto the pores and catalyst surface, and effects of abrasion caused by mechanical crushing of the catalyst.

Figure 2a shows a typical catalyst module formed by a metal housing containing the catalyst. Figure 2b shows a new catalyst and the visual effect of deactivation in a poisoned catalyst is shown in Figure 2c and 2d. The amount of soot deposited in an used catalyst depends of the fuel quality, since gasoline contains some amount of contaminants such as sulfur, and oxygen and nitrogen compounds. A new catalyst sample is shown in Figure 3a and 3b, and Figure 3c shows the clogging of the honeycomb structure caused by the poisoning of the catalyst.

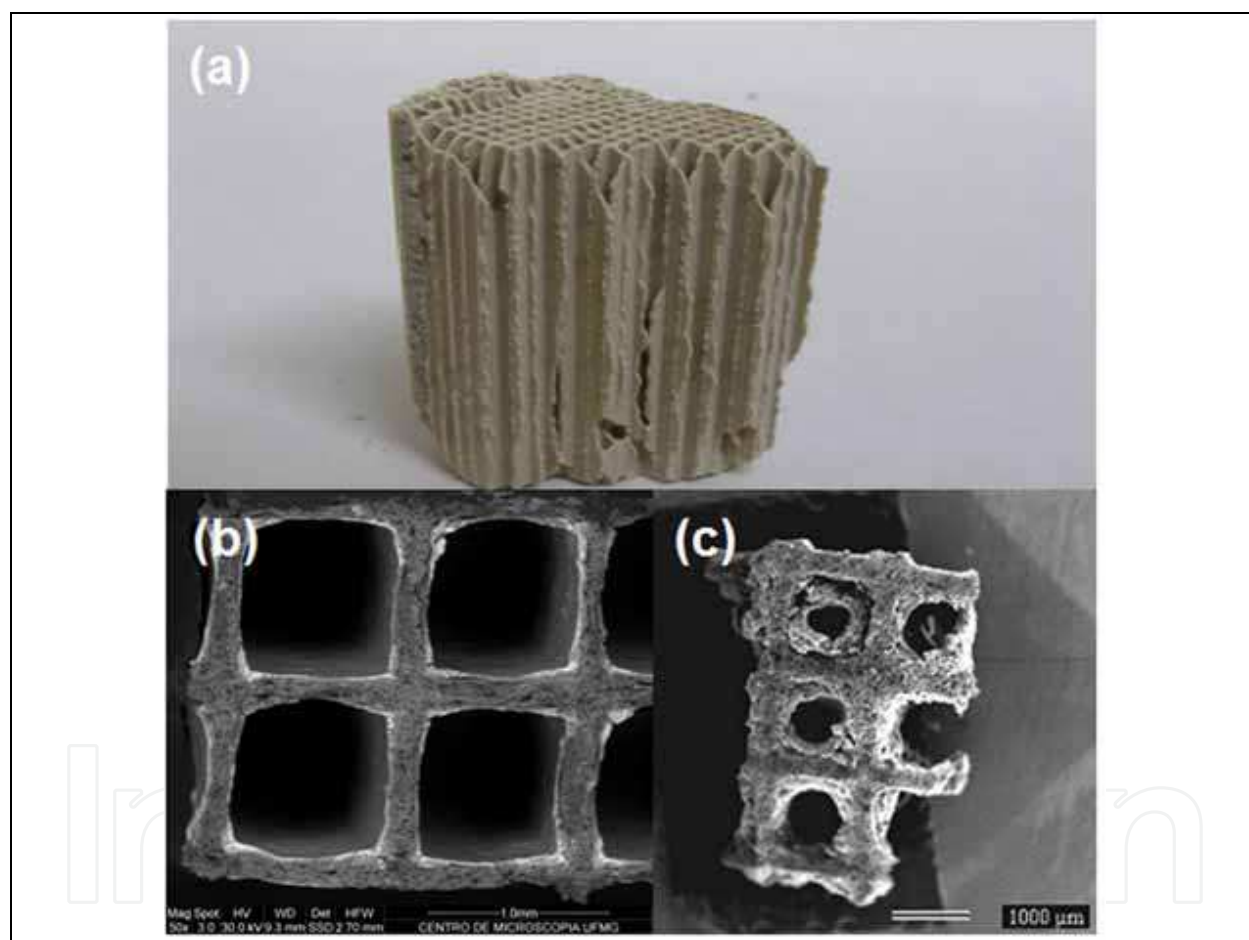


Fig. 3. (a) Photography of a catalyst sample, (b) SEM micrograph of a honeycomb structure, (c) clogging of the honeycomb structure caused by the catalyst poisoning.

Beyond the vehicles powered by gasoline, there has been a move for the utilization of other types of vehicles that have been developed to use different fuels that produce less CO_2 , which cause the greenhouse effect. These fuels are the alcohol, renewable bio-fuel derived by sugar-cane or corn, gases such as liquefied petroleum gas (LPG) and compressed natural gas (CNG), or mix of fuels as the used in flex-fuel technology. These alternative fuels have a lower carbon to hydrogen ratio than gasoline, producing less CO_2 per travelled distance, and reduce the needed of fossil fuel consumption (Cohn, 2005).

Some technologies have been developed, adapting the engine for the mixtures of fuels like gasoline and ethanol with predetermined composition. Moreover, there are the new flex-fuel technology that is related to the flexibility of choice of the car fuel, where is possible to use only hydrated alcohol or gasoline, or a mixture of these fuels in any concentration (Delgado et al, 2007). The people can buy the cheapest fuel, whose prices depend on the economic moment. The flex-fuel technology is based on sensors that detect the concentration of the mixture of gasoline and hydrated alcohol, and in the subsequent automatic adjustment of the engine. The addition of ethanol in gasoline decreases the concentration of CO emissions, making this process a very interesting technology. Some countries are planning to employ this technology, since about 85% of the cars produced in Brazil are equipped with this technology. All of these factors impact the design of TWC, since its geometric surface area until the alumina thin film formulations. It would be necessary a corresponding catalyst for each type of used fuel, leading in consideration the type of chemical reaction that occurs in the engine. But in the reality the catalysts of these new vehicles have been adapted without rigorous criteria, and they are adjusted according to need (Silva, 2008).

Other factors that influenced the development of TWCs were the economics ones, mainly the related to the prices of the platinum group metal and of the fuels. The constant increase and instability in the gasoline price led to the development of more economic engines that also need different design of catalyst. In this sense, various types of substrate as *zeolites* or metallic have been tested and/or used (Collins & Twigg 2007).

Actually, recycling and regeneration of catalysts are common practices. Regeneration consists in a controlled oxidation at high temperature to eliminate soot and convert sulfides to oxides. After this process, some catalysts also require additional treatments to recover the full activity. Non-regenerable catalysts have to be recycled for metals recuperation. This can be performed either by hydrometallurgy or pyrometallurgy (Angelidis et al, 1995; Silva, 2008; Dufresne, 2007 & Hirokazu, 1999).

In this chapter, textural, morphological and structural characteristics of selected new and used catalysts, analyzed by gas adsorption, pycnometry, X-ray diffractometry, thermal analyses and scanning electron microscopy, are shown. EDS and WDS electronic microprobe were used to detect the composition of the catalysts and their contaminants. Subsequently, we discuss the textural and morphological changes of automotive catalyst by effect of high temperatures, which lead to its deactivation. New commercial automotive catalysts were thermally treated at various temperatures. Micrographies and adsorption-desorption isotherms were used to verify the changes in the catalyst characteristics with thermal treatments. Finally, problems about gas emission and the soot present in exhaust gas are discussed, beyond some aspects about reuse and recycling are considered. Some solutions about this theme are shown.

2. Textural, morphological and structural characteristics of new and used catalysts

2.1 Experimental

Some new and used automotive catalysts of vehicles powered by gasoline, by alcohol, and by flex fuel, of diverse suppliers, have been analyzed.

The samples have been analyzed by X-ray diffractometry (Rigaku, Geigerflex 3034) with CuK α radiation, 40kV and 30mA, time constant of 0.5s and crystal graphite monochromator to identify the phases present (metals and transition metal oxides).

The composition, metal distribution on the alumina thin film and morphology of the catalysts have been evaluated by an electron microprobe (Jeol JXA, model 8900RL) with an energy dispersive and wavelength dispersive spectrometers (EDS/WDS), and by scanning electron microscopy (Quanta 200, FEG-FEI).

Density measurements of the catalysts have been obtained by helium picnometry (Quantachrome) and sample textural characteristics were determined by nitrogen gas adsorption (Autosorb - Quantachrome) at liquid nitrogen temperature. Nitrogen gas has been used with a 22-point adsorption-desorption cycle. The samples have been outgassed at 200 °C for 12 hours before each analysis. Experiments have been made in triplicate. Specific surface area and total pore volume have been obtained by the application of Brunauer-Emmett-Teller (BET) equation and the BJH method, respectively (Lowell & Shields, 2005).

2.2 Results and discussion

2.2.1 X-ray diffraction

The diffractogram of the new catalyst (Figure 4a) is characteristic of nano and/or porous materials and shows a good correspondence with the cordierite diffractogram standard,

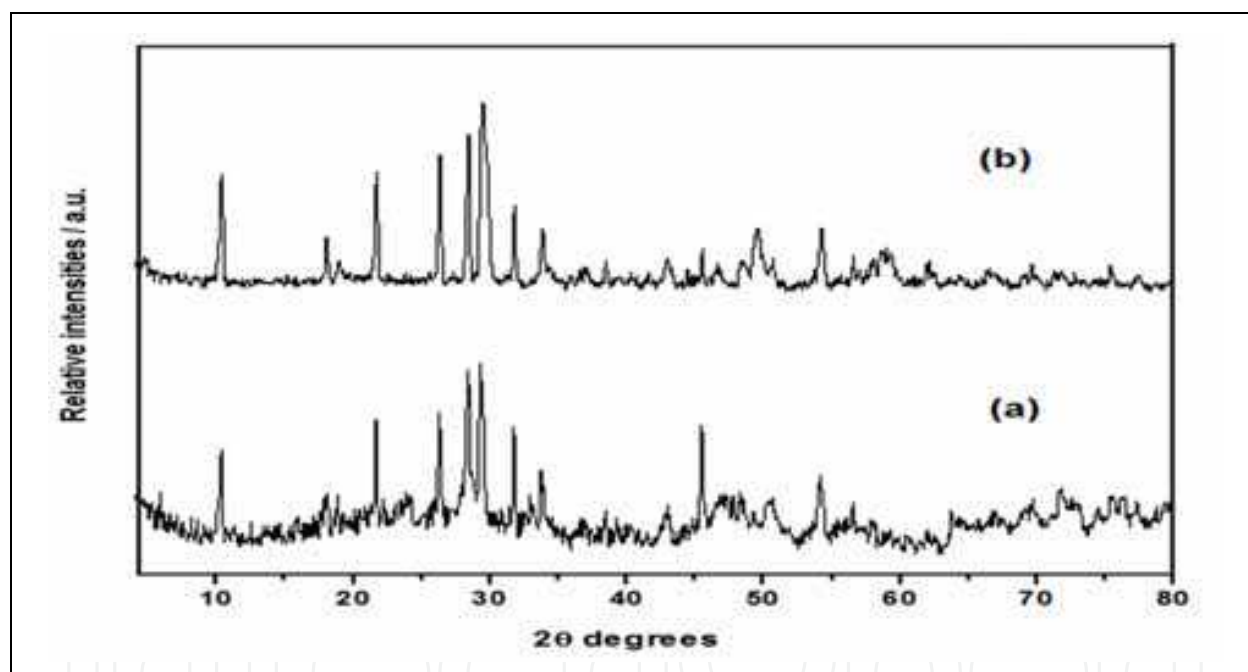


Fig. 4. X-ray diffraction patterns of (a) new and (b) used catalysts.

beyond characteristic peaks of the gamma-alumina film and of the metals dispersed in the wash-coat. A reasonable structural variation is evidenced in the diffractogram of the used catalyst (Figure 4b), that presents more crystalline behavior and characteristic peaks of precious metallic oxides.

2.2.2 Microanalysis and scanning electron microscopy

Fig. 5 shows an image of scanning electron microscopy of the catalyst obtained by back-scattering electrons. It is possible to observe the porous alumina thin film with precious metal heterogeneously dispersed (white dots) deposited on cordierite (macroporous material). The precious metal particle size varied from 1 to 15 μm. The chosen points of the

Fig. 5a have been analyzed with an EDS detector, confirming the expected basic cordierite compositions in region 1 (dark region), formed by Al, Mg and Si (Figure 6a). Region 2 also has the same composition of the cordierite, with some impurities such as TiO_2 , Fe_2O_3 , CaO and ZrO_2 (Figure 6b).

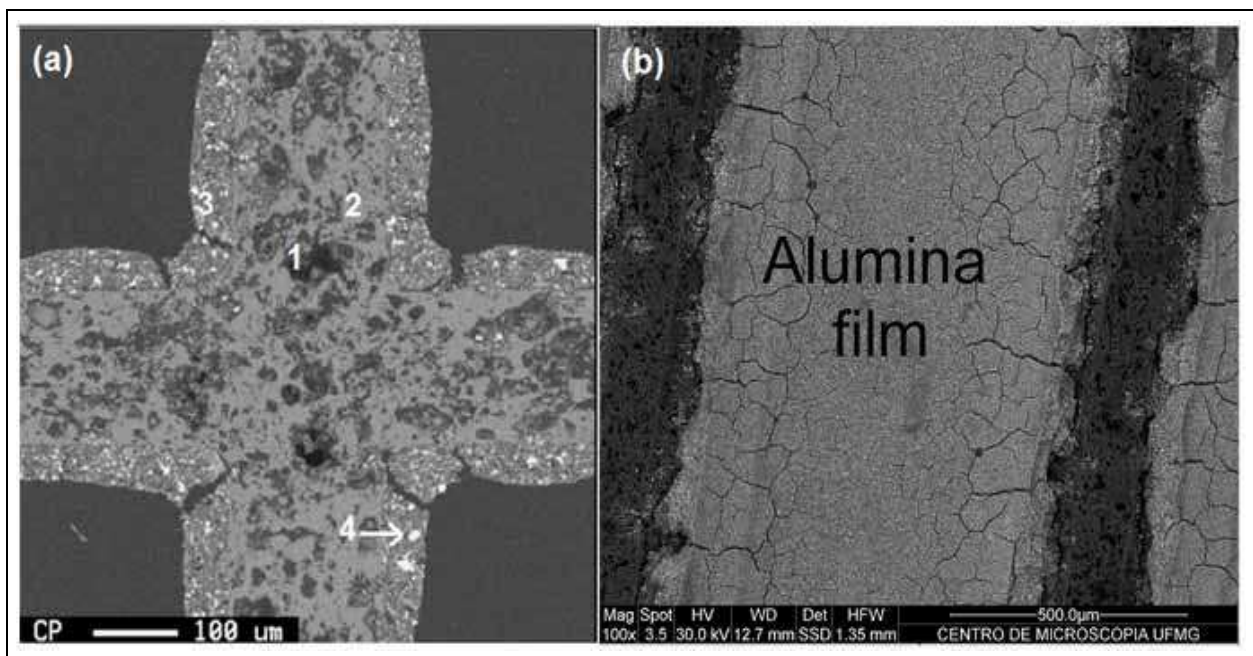


Fig. 5. (a) Backscattering SEM micrograph of a piece of a new automotive catalyst, and (b) detail of the alumina thin film on the cordierite.

The alumina wash-coat is pure (region 3 of Fig. 5a and Figure 6c) with metals and oxides dispersed such as cerium and zirconium oxide (Ce_2O and ZrO_2) in more quantity and traces of palladium (Pd) characterized by region 4 of Figure 5a and Figure 6d. Platinum and rhodium particles have been observed only by WDS detector because their minor quantity dispersed in the thin film.

After some time of utilization (months or years), the catalyst suffers poisoning due to the fuel and lubricant residues, chemical reactions and also effects of sintering due to the high operating temperatures, which generally can reach 900 °C. The images of Figure 7 show the morphological and textural comparison between the alumina films of a new and an used catalyst. The new catalyst surface (Fig. 7a) is porous with disperse precious metal particles, while the used (Fig. 7b) shows an eroded surface with agglomeration of the precious metal particles and the formation of microcracks. Texturally, the used catalyst shows a decrease in the porosity related to the new catalyst, due to the beginning of sintering caused by the operational temperature.

Figure 8 shows with more detail a morphological comparison of new and used catalysts of vehicles powered by gasoline. Column (a) shows a new cordierite substrate more macroporous and an alumina thin film more porous and preserved than those of the used catalyst (column b). It is possible to observe the precious metal diffusion inside the cordierite of the used catalyst, beyond an increase of the precious metal agglomerates also due to the diffusion process.

In general, used catalysts show a large quantity of ash and/or soot in the surface and inside of their pores. Figure 9a illustrates the obstruction of a catalyst by these contaminants. These

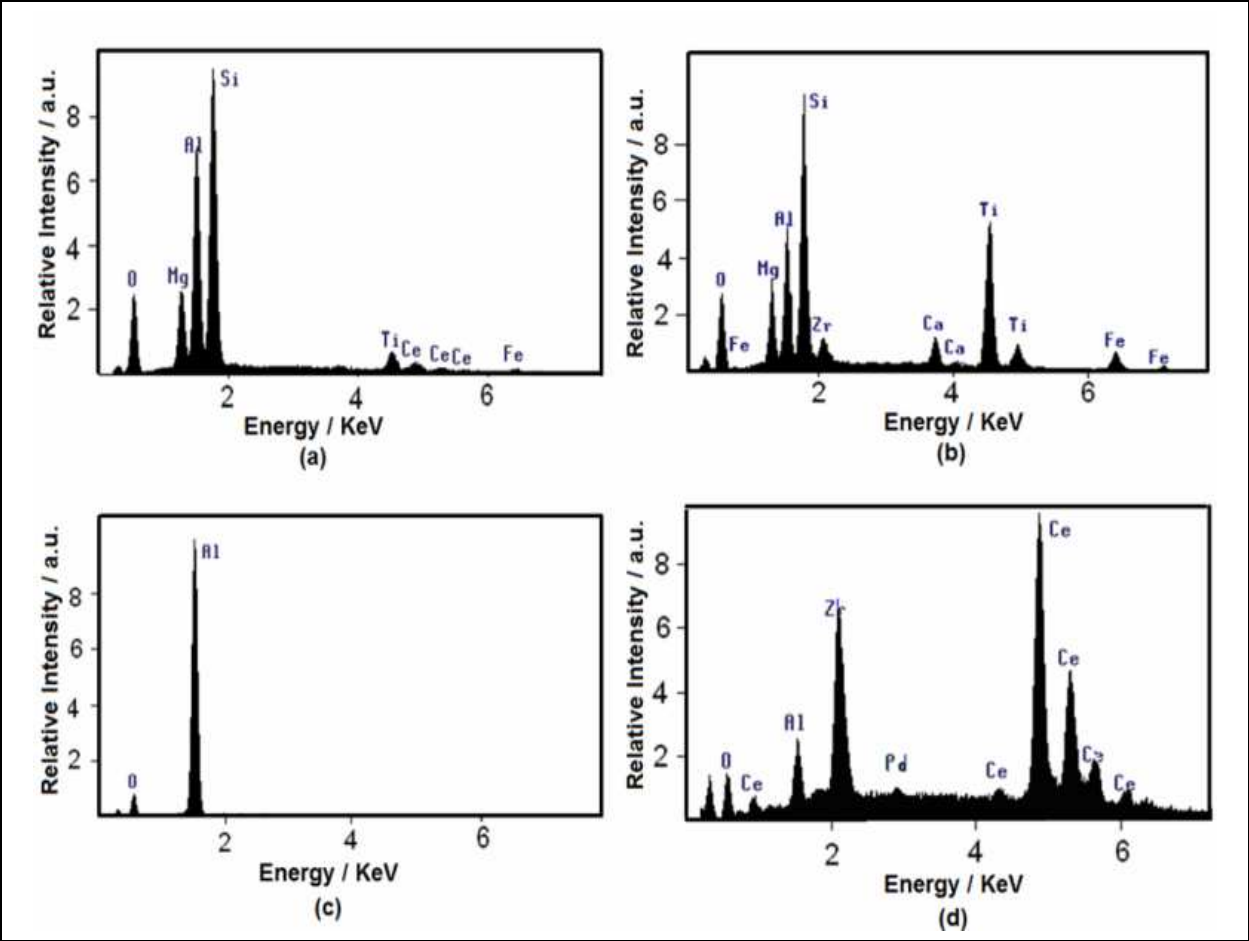


Fig. 6. EDS spectra of new automotive catalyst. a: cordierite (region 1), b: cordierite impurities (region 2), c: alumina film (region 3), d: active metals and oxides (region 4).

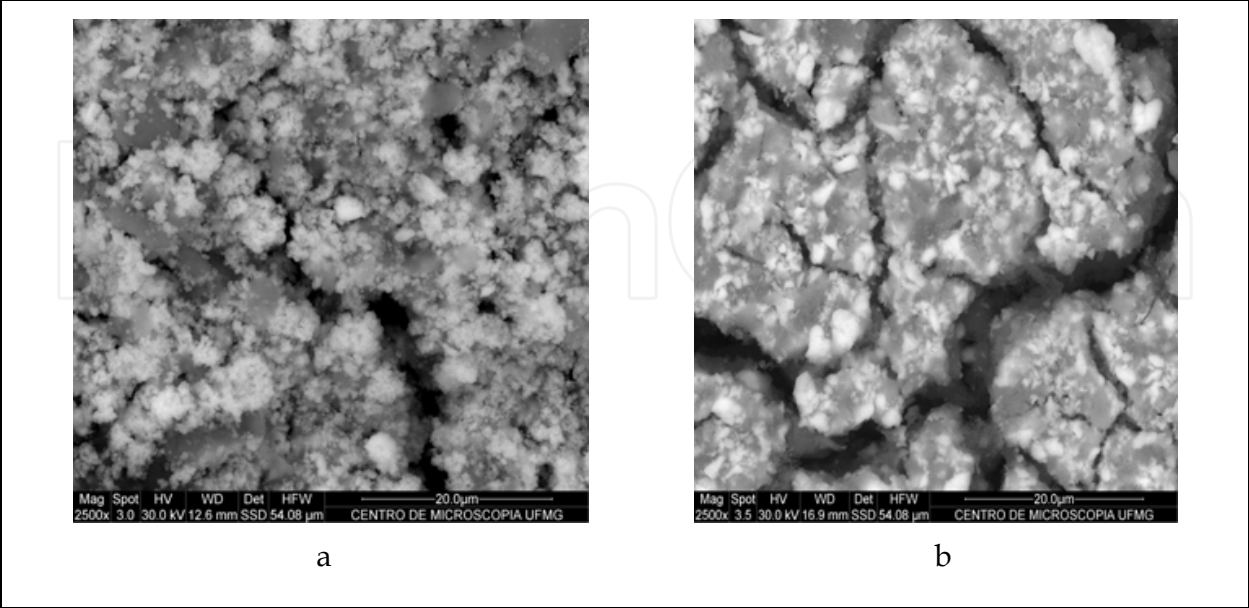


Fig. 7. Backscattering SEM micrograph of the alumina film of the (a) new and (b) used automotive catalyst.

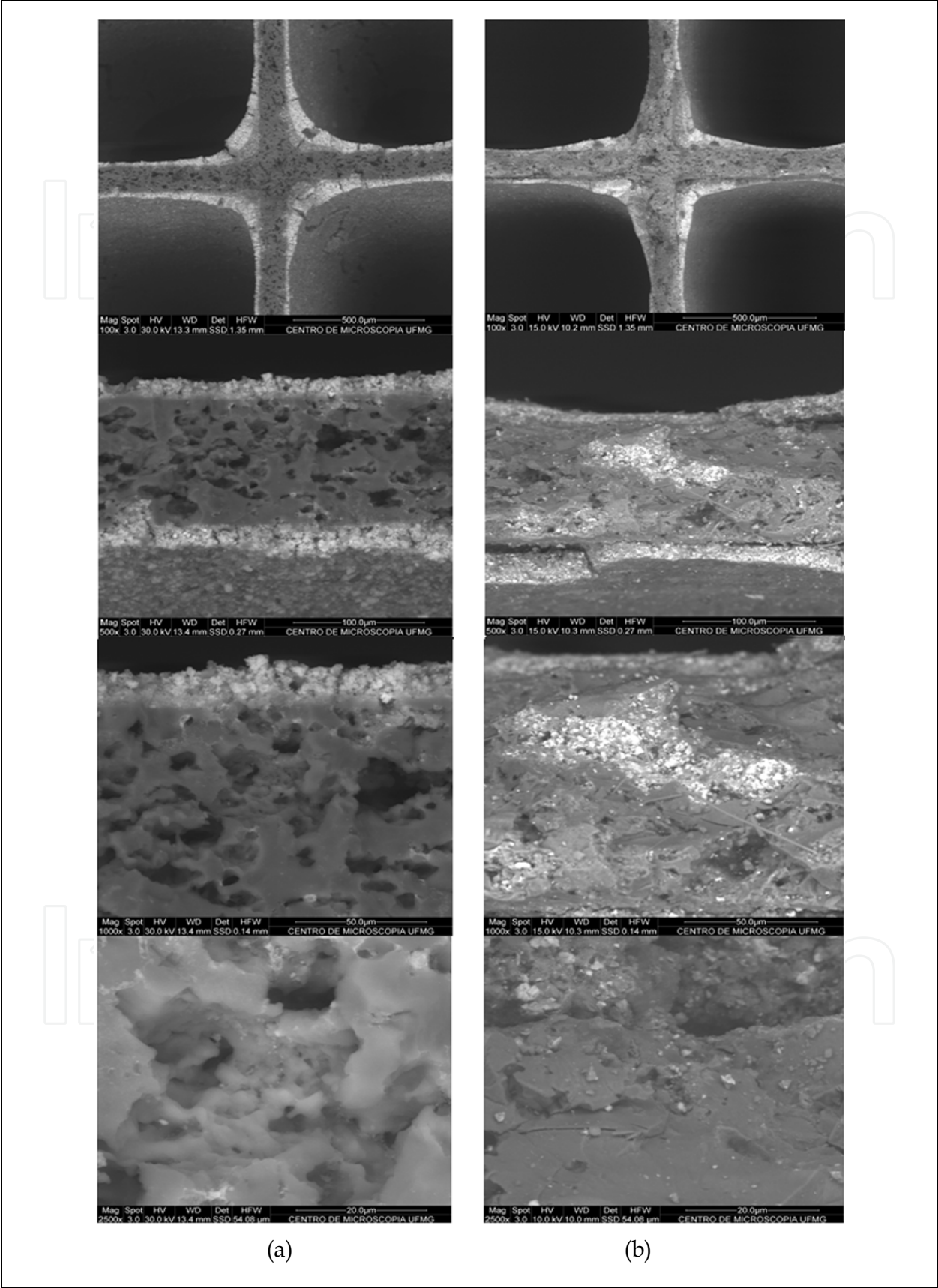


Fig. 8. Backscattering SEM micrographies of the (a) new and (b) used catalyst samples.

particles penetrate inside the pores of the catalyst, clogging the monolith cordierite channels (honeycomb structures). EDS analyses showed that the used catalysts has the same composition of the new catalysts, but also has a considerable amount of carbon, potassium, sulfur and chlorine that can come from of fuel and lubricating oil (Figure 9b). Particulate

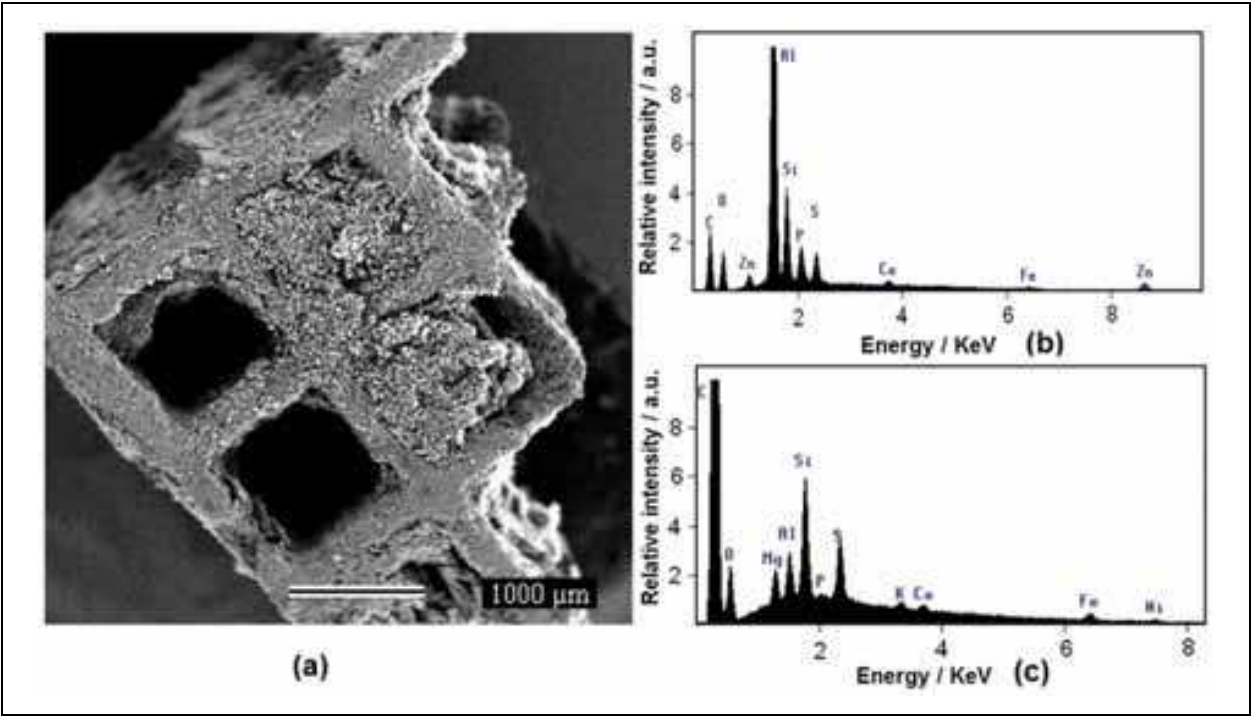


Fig. 9. (a) SEM micrograph of the obstructed used catalyst; (b) EDS spectra of the used catalyst; and (c) EDS of the particulate material (ash and soot).

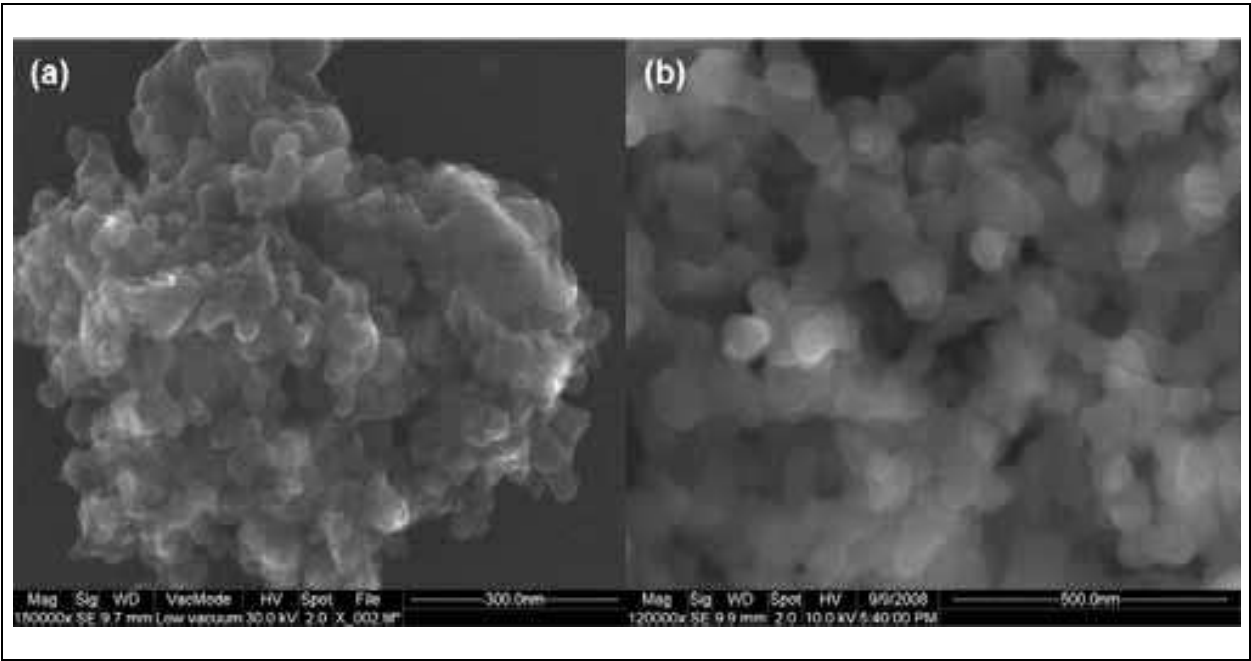


Fig. 10. SEM micrograph of the soot removed of a poisoned catalyst.

samples (ash and soot) collected of various *poisoned* catalysts were analyzed by EDS, and showed great amount of carbon, sulfur, silica, alumina and magnesia, as well as, smaller quantities of phosphorus, iron and nickel (Fig 9c).

Figure 10 shows micrographies of the ash and soot retired of a poisoned catalyst, which show characteristic of nanoparticulate material, with particle sizes about 45 ± 15 nm, forming agglomerates with size in the range from 500 to 2 μ m. This type of material is very active due to its small particle size and, when inhaled, is harmful to the health, causing lung diseases. Actually, the filters used are not capable of retaining this kind of material that goes to atmosphere by the smoke.

2.2.3 Gas adsorption technique

The capacity of adsorption of new and used catalysts has been evaluated. Various types of catalysts have been analyzed and the results have been similar. The specific surface area varies with the type and model of the catalyst, but the decrease in the values is proportional. Table 1 shows the changes in density, specific surface area and total pore volume values of two catalysts that showed the lowest and largest specific surface area, one of a vehicle powered by gasoline and other of a flex-fuel vehicle. The used samples (poisoned catalysts) have been cleaned to eliminate the soot trapped in the beehive of the catalyst, remaining only the soot physically adsorbed in the pores of the catalyst. The density of the used catalyst is larger than the new catalyst, suggesting a densification process due to the high temperature of operation. Consequently, the used catalyst has textural values lower than those of the new catalyst, which causes its deactivation. It is observed increasing of about 85% in the specific surface area and 75% in the porosity.

Sample	Density /g.cm ⁻³	Specific Surface Area /m ² .g ⁻¹	Total Pore Volume / 10 ⁻³ cm ³ .g ⁻¹	Average Pore Size/nm
New (Gasoline)	3.1 ± 0.1	58	141	11
Used (Gasoline)	3.6 ± 0.1	9	36	4
New (Flex)	3.0 ± 0.1	282	100	12
Used (Flex)	3.4 ± 0.1	18	8	6

Table 1. Textural characteristics of new and used catalyst of a vehicles powered by gasoline and flex fuel (gasoline and alcohol).

The adsorption-desorption isotherms (Fig. 11 and Fig. 12) are characteristics of mesoporous materials (isotherm type IV, according to IUPAC classification) and show that the new catalyst sample adsorbs a higher volume of nitrogen when compared to the used catalyst. The catalyst of the vehicle powered by gasoline lost 75% of its adsorptive capacity and the catalyst of the flex-fuel vehicle lost 92%. The changes in the shape of the isotherms of the used catalysts show a large variation in pore shape, that together with the results in Table 1 prove the high variation on the textural characteristics of the used catalyst when compared with a new one.

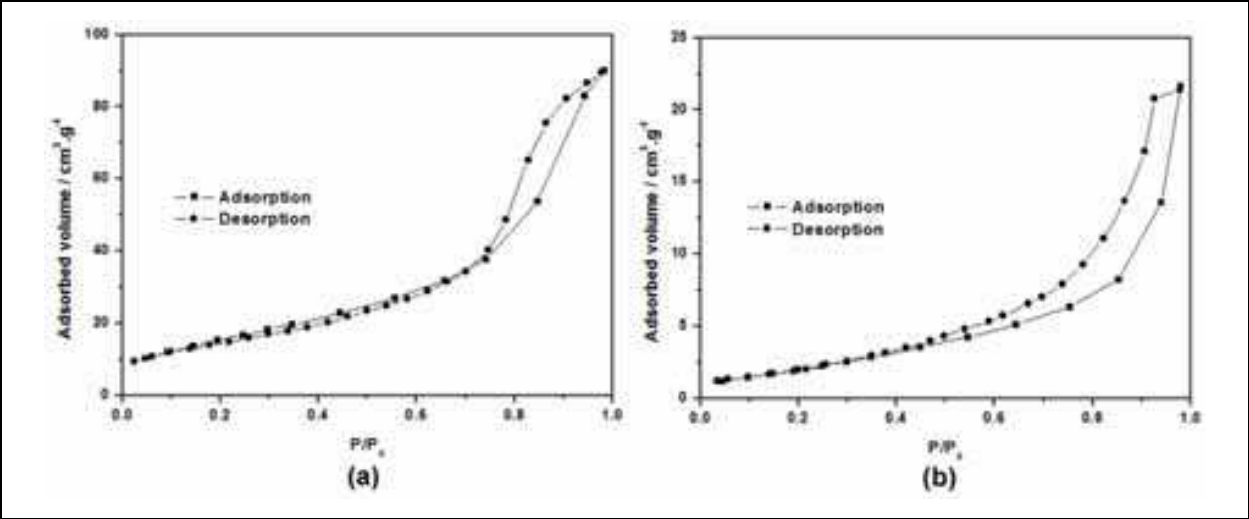


Fig. 11. Adsorption-desorption curves of (a) new and (b) used catalysts of a vehicle powered by gasoline.

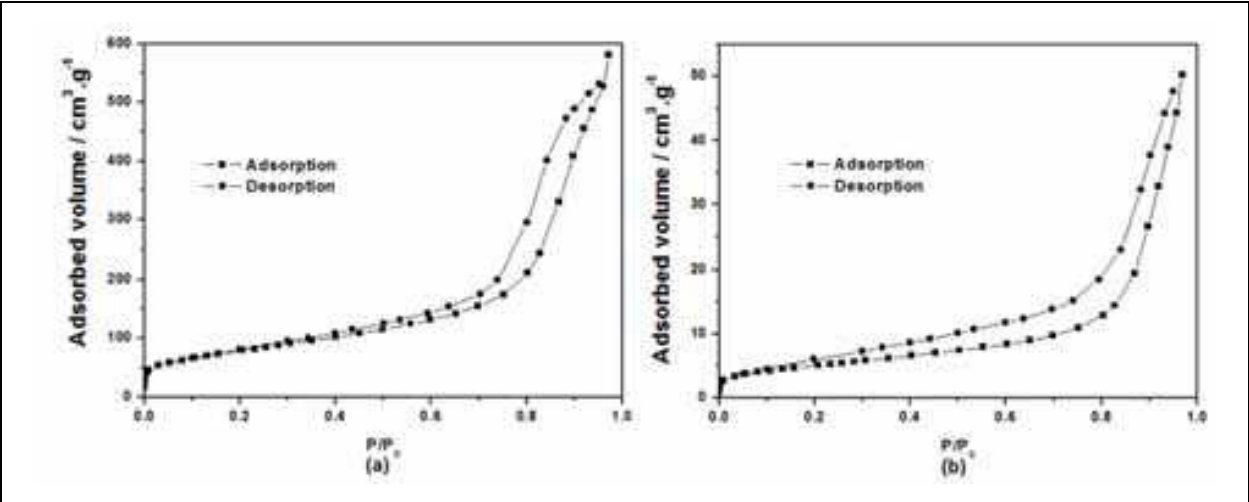


Fig. 12. Adsorption-desorption curves of (a) new and (b) used catalysts of a flex -fuel vehicle.

3. Study of the temperature effect on textural and morphological characteristics of automotive catalysts

3.1 Experimental

Selected new catalysts have been broken in pieces of 2 cm of side and have been thermally treated at 500, 700 and 900 °C during 5 hours to verify the changes in the textural, morphological and structural characteristics as a function of the temperature. Simultaneous thermogravimetric and differential thermal analysis (TG-DTA) measurements have been performed in air and N₂ (TA Instrument SDT 2960). Samples have been heated from room temperature to 1400°C at 10 °C min⁻¹. The variation on the sample morphologies have been observed by scanning electron microscopy in an equipment JEOL JSM, model 840 and in an equipment Quanta 200, FEG-FEI.

Variation in the true density has been evaluated by helium picnometry (Quantachrome) and the textural characteristics have been determined by nitrogen gas adsorption (Autosorb - Quantachrome) at liquid nitrogen temperature. The samples have been outgassed at 200 °C for 12 hours before each analysis.

3.2 Results and discussion

3.2.1 Thermal analysis

The analysis of the TG curves (Figure 13a) shows a significant loss of mass (about 20 %) for new automotive catalysts during the heating between 100 and 700 °C under air atmosphere. This loss corresponds probably to the oxidation, densification and crystallization processes, which is corroborated by exothermic events in the DTA curves in the same region, and by X-ray diffraction results obtained by new and used catalysts. TG and DTA curves (Figure 13b) of samples heated in N₂ atmosphere show a minor loss of mass (about 10 %) and events less exothermic. Considering that the automotive catalyst changes considerably with temperature up to 600 °C, we can conclude that the operational temperature of 900 °C is enough to deactivate partially the catalyst.

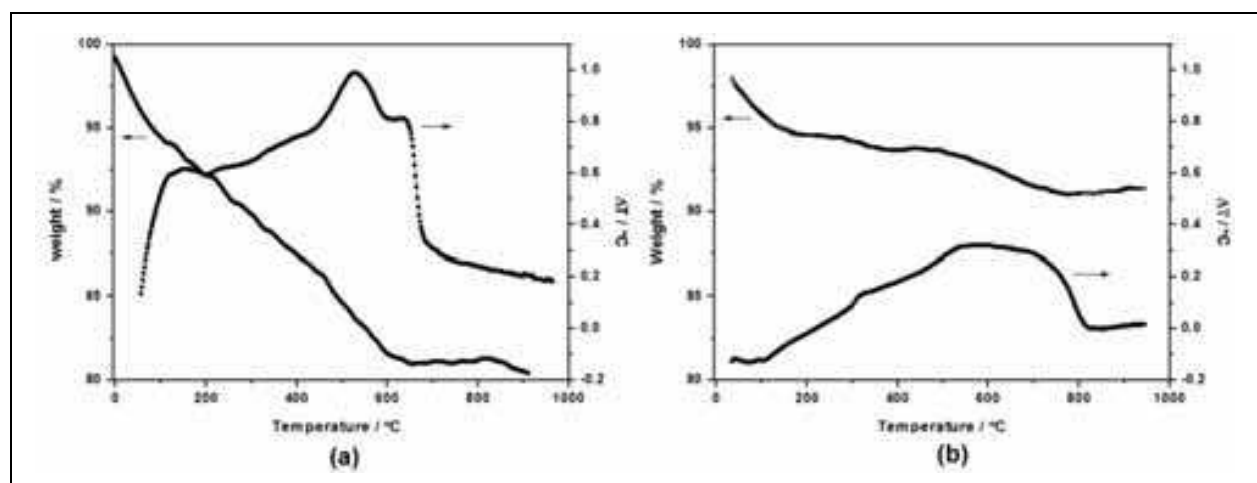


Fig. 13. TG-DTA curves obtained in (a) air and in (b) N₂ atmosphere for new catalyst.

3.2.2 Scanning electron microscopy

Figures 14 and 15 shows micrographies obtained by SEM of catalyst samples without treatment (a), treated at 500 °C (b) and at 1100 °C (c). In the various tests realized, the thickness of the alumina film diminishes with a simple thermal treatment at 500 °C for 5 h (of about 40 %) and with thermal treatment at 1100°C for 5 h diminishes of about 60 %. It is observed shrinkage and the appearance of cracks in the alumina films deposited on the cordierite due to the increasing in the heating temperature.

Figure 16 shows, with more detail, images of the heating effect in catalyst samples without treatment and treated at 500 and 900° C. Figure 16a shows the alumina film of a catalyst without treatment and Figure 16b shows the alumina film treated at 500 °C. It is possible to observe the beginning of densification of the film treated at 500 °C. Figures 16c and 16d show the cordierite without treatment, more porous, and treated at 500°C, respectively. Figures 16e and 16f show the diffusion of the precious metal and the sintering process of the catalyst treated at 900 °C, respectively.

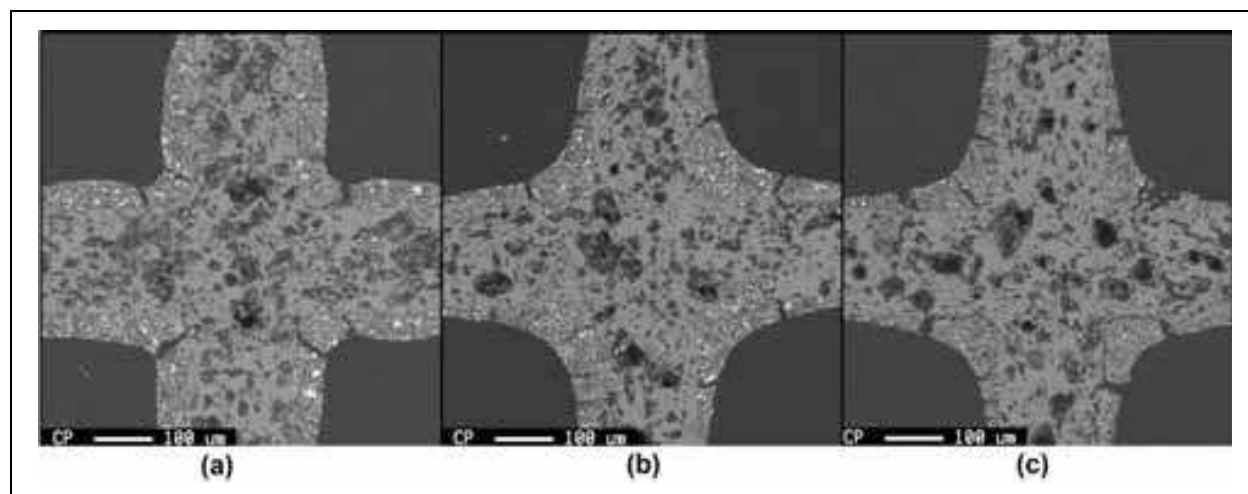


Fig. 14. Backscatter SEM images of (a) new catalyst without heating, (b) heated at 500 °C, and (c) heated at 1100 °C.

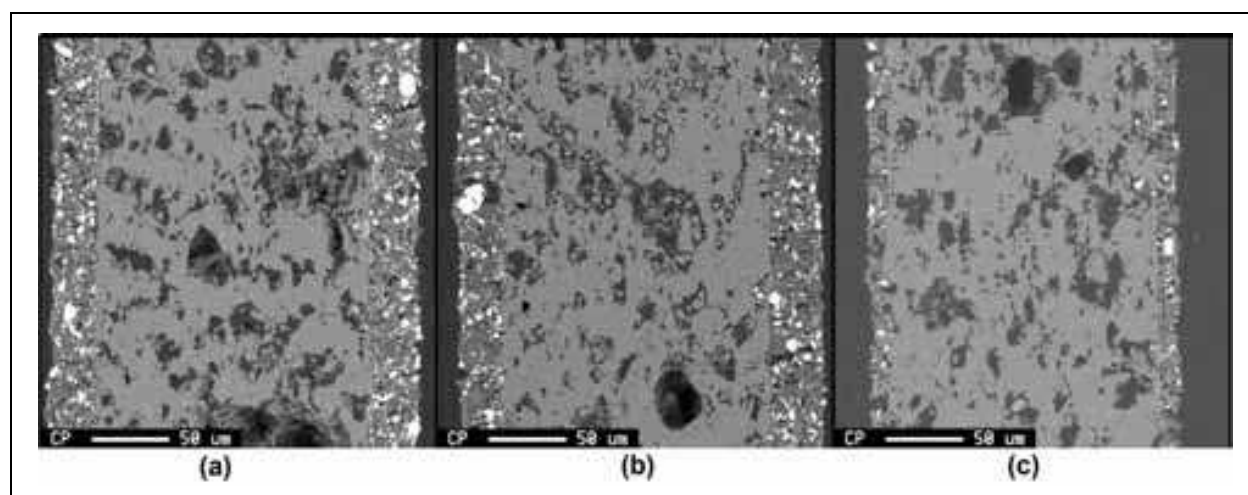


Fig. 15. Backscatter SEM images of (a) new catalyst without heating, (b) heated at 500 °C, and (c) heated at 1100 °C.

3.2.3 Gas adsorption technique

Table 2 shows the changes in density, specific surface area and total pore volume values of a catalyst heated at various temperatures. With the increasing in heating temperature occurred a drastic decreasing in the textural values up to 900 °C. These results suggest a densification process due to increase in the temperature next to the temperature of operation. The value obtained by the catalyst heated at 900 °C is similar to the obtained by the used catalysts.

The adsorption-desorption isotherms of samples treated between 500 and 900 °C (Fig. 17) are also characteristics of mesoporous materials and show a lower adsorptive capacity than the new catalyst without thermal treatment (Fig. 13a). The new catalysts lost 65% of their adsorptive capacity when heated at 500 °C for 5 hours and 72% when heated at 900 °C for 5 hours. The shape of hysteresis remained nearly constant, showing that there was little variation in pore size due to the densification process, while the amount of pores (porosity) decreased.

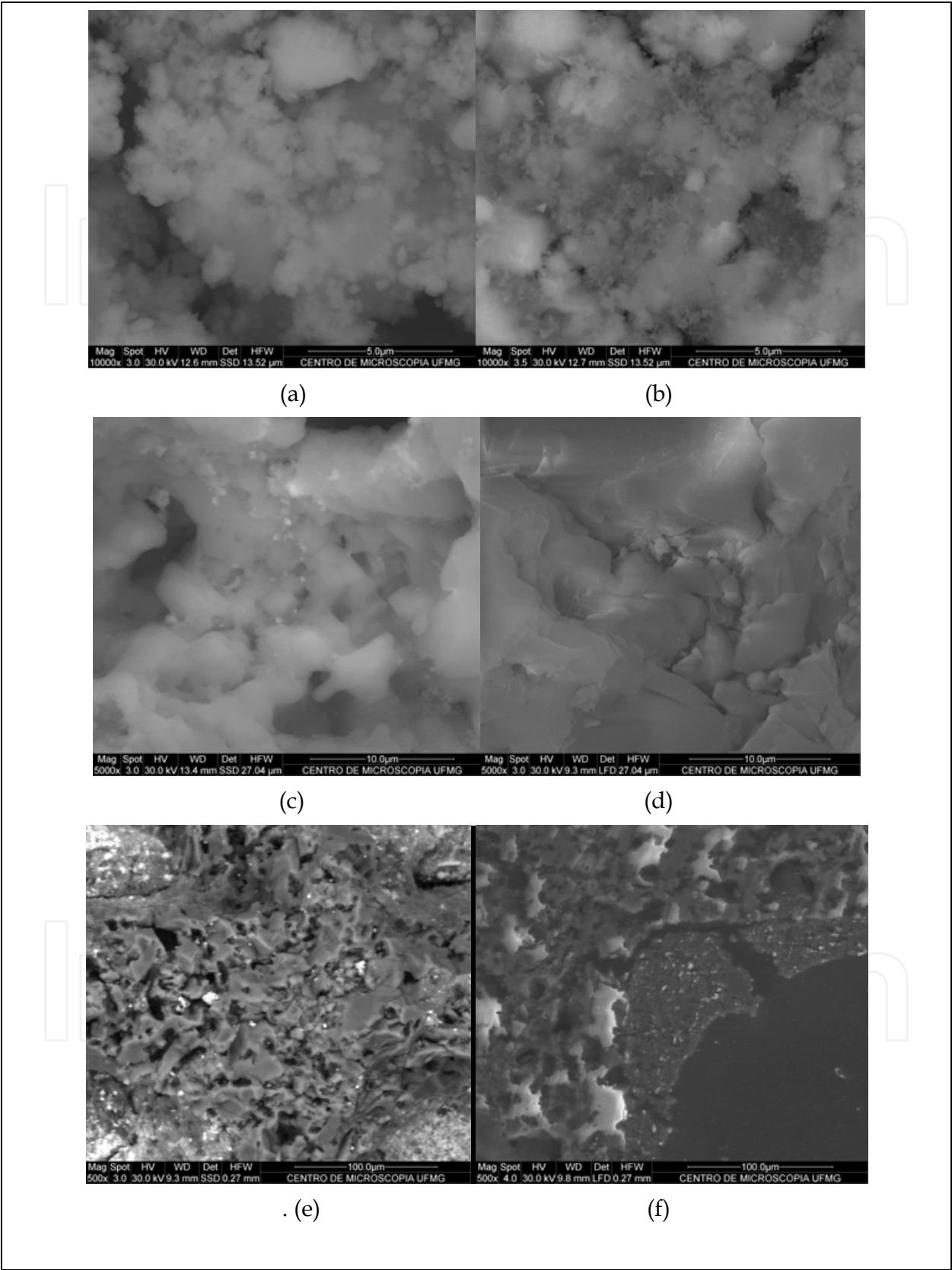


Fig. 16. SEM images of new catalyst (a and c) without heating, (b and d) heated at 500 °C, and (e and f) heated at 900 °C.

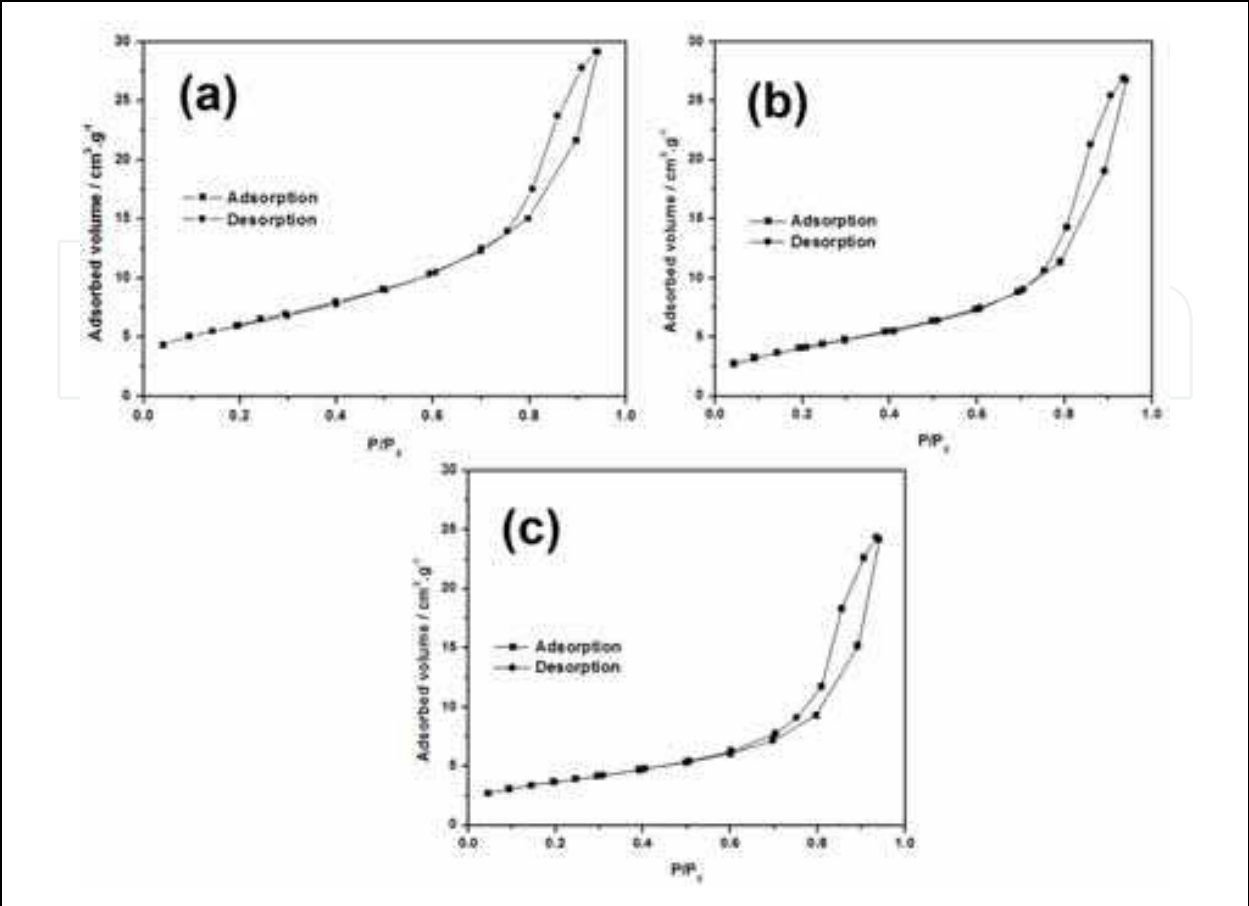


Fig. 17. Adsorption-desorption curves of new catalyst heated at: (a) 500 °C, (b) 700 °C and (c) 900 °C.

Heating Temperature New Catalyst/°C	Density /g.cm ⁻³	Specific Surface Area /m ² .g ⁻¹	Total Pore Volume / 10 ⁻³ cm ³ .g ⁻¹	Average Pore Size /nm
New	3.1 ± 0.1	58	141	110
500	3.1 ± 0.1	30	48	37
700	3.2 ± 0.1	17	30	36
900	3.2 ± 0.1	12	27	30

Table 2. Textural characteristics of a new catalyst of vehicle powered by gasoline heated at various temperatures

The adsorption capacity of a catalyst decreases at high temperatures (operating temperature) due to the physical-chemistry alterations that occur mainly in ceramic catalyst surface, such as specific surface area decrease, changes in the phase of the alumina film and metal diffusion inside the ceramic matrix to generate large agglomerates. Fig. 18 shows a schematic illustration of the catalyst deactivation due to these thermal effects.

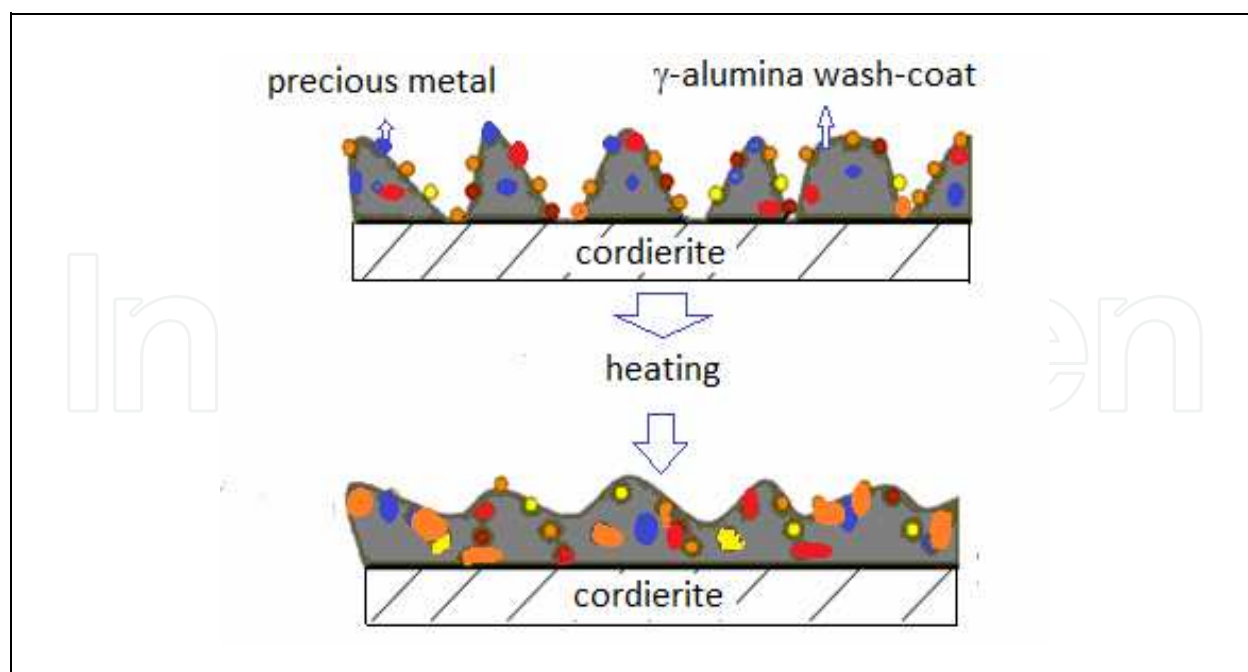


Fig. 18. Schematic illustration of the catalyst deactivation due to the thermal effects.

4. Conclusion

With the current increase in production of vehicles and consequent increase in gas emissions, especially CO₂, various solutions have been sought by scientists and governments around the world. The research on electric cars, already under implementation, the total or partial replacement of petrol by biofuels and gas, and the constant development of new types of automotive catalysts are examples of the attempt to contain the gaseous emissions.

Regarding the catalytic converter, new technologies have been developed but are not employed because the environment laws are not as stringent. With the increase in emissions and consequent global temperature increase, as has been happening during the year 2010, government decisions on the environment should be more stringent.

This work showed problems related to mechanical deactivation of catalysts, due to the soot deposited on their active surface and thermal deactivation involving the reduction of surface area, diffusion and agglomeration of the precious metals, with subsequent decrease of adsorption capacity of catalysts. Some results showed that a catalyst can be deactivated in the first hours of operation, if the temperature reaches between 500 and 900 °C.

These problems can be solved by improving the precious metals distribution on the alumina films by nanotechnology practices, using alumina thin films more stable at high temperatures as sol-gel technology and various other new technologies available in the scientific literature.

5. Acknowledgements

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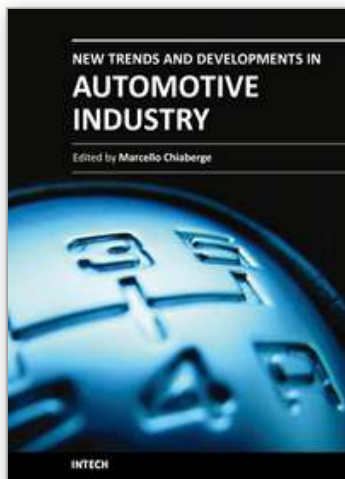
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