Removing aromatic and oxygenated VOCs from polluted air stream using Pt–carbon aerogels: Assessment of their performance as adsorbents and combustion catalysts

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ABSTRACT

Two series of Pt-catalysts were prepared by impregnation or doping of carbon aerogels and different porous textures and Pt-dispersion were obtained. The performance of the samples in the elimination of organic compounds (VOCs) by adsorption and catalytic combustion was studied and compared with the characteristics of both the VOCs and the catalysts and the interactions between them. Toluene, xylenes and acetone were selected as representative aromatic or oxygenated VOCs. The adsorption of VOCs is favoured at room temperature in the case of meso/microporous materials, but at the higher catalytic reaction temperature, the micropores volume is more important. Adsorption and catalytic combustion occur simultaneously, and are both dependent on temperature, albeit in opposite directions. The combustion of aromatic compounds takes place at a lower temperature than that required for acetone combustion, so favouring the accumulation of adsorbed VOC, something that should be avoided to minimize risks. Catalytic performance improves with the contact time and is independent of oxygen content above 5% v/v, but declines significantly below this limit.

1. Introduction

The control of the emission of volatile organic compounds (VOCs) in gaseous effluents is essential if we want to preserve air quality, including that of indoor air. Two different approaches to the problem have been developed, the first based on VOC recovery techniques (absorption, adsorption, condensation, membrane separation), and the second on destruction techniques (incineration or catalytic combustion) as described in a previous review of this subject [1]. The development of progressively more efficient materials to be used in the different technologies available is therefore an important and stimulating research field. Catalytic combustion and adsorption of VOCs are both promising technologies, and interesting hybrid processes combining adsorption and incineration in a single fixed bed have also been developed [2–4]. The use of combustion catalysts significantly decreases the working temperature compared to more traditional incinerators, so preventing the formation of NO_x and saving energy.

In previous works we used metal-oxide [5] or noble-metal (Pt, Pd) carbon-supported catalysts in BTX combustion [6–10], using carbon supports in different formats, from bulk carbon particles to carbon coatings deposited on ceramic monoliths. The use of carbon supports enabled us to reduce the reaction temperature significantly because their hydrophobic behavior prevents the chemisorption of water produced by combustion [9]. Moreover, materials such as carbon aerogels offer the advantage of controllable porosity and purity, because they are synthesized from pure organic reactants, and therefore do not contain inorganic materials that could contribute to or affect catalyst behavior, as occurs with classical activated carbons. The control of carbon porosity clearly affects the performance of the catalyst, because it influences parameters such as the adsorption of reactants or active phase dispersion, so producing materials with high catalytic capacities [11].

Of the different methods for depositing the noble metal on carbon aerogels (impregnation, doping, equilibrium adsorption, CVD, etc.), doping the initial organic solutions proved to be highly efficient, as it prevented sintering and the loss of active phase by leaching when used on the liquid phase [12]. Nevertheless, the presence of a metal precursor in the starting solution strongly modifies the textural, mechanical and chemical characteristics of carbon aerogels. In addition, some of the metal particles may be surrounded by the carbon matrix and are therefore rendered catalytically inactive [6]. The porosity of carbon aerogels should be optimized in such a way that the metal particles can be active. The

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Table 1
Textural characteristics of carbon aerogels.

Sample	V_2 (cm^3/g)	V_3 (cm^3/g)	W_0 (cm^3/g)	L_0 (nm)	S_{mi} (m^2/g)	S_{BET} (m^2/g)
A500	0.66	0.00	0.23	0.61	760	655
A1000	0.62	0.00	0.25	0.58	857	530
AHPt-500	0.00	0.92	0.20	0.57	689	496
AHPt-1000	0.00	0.91	0.24	0.59	814	483

V_2, pore volume corresponding to pores with a diameter of between 3.7 and 50 nm; V_3, pore volume of pores with a diameter higher than 50 nm; W_0, micropore volume; L_0, mean micropore width; S_{mi}, microporous surface area; S_{BET}, BET surface area.

porosity of carbon aerogels depends on the synthesis process and is influenced by the gelation, carbonization and activation conditions [13].

In this work we prepared two series of Pt-based carbon aerogels. Platinum was deposited by impregnation or doping and carbonized at different temperatures. We then studied the behavior of these samples as VOC adsorbents or VOC combustion catalysts and analyzed the influence of textural parameters on these processes and the relations between them in different experimental conditions. Toluene, xylenes and acetone were selected as target VOC molecules, as they are representative examples of aromatic and oxygenated organic compounds.

2. Experimental

Two series of organic aerogels were prepared by the sol–gel procedure from the polymerisation reaction of resorcinol (R) and formaldehyde (F) in water (W). All the samples were prepared using a molar ratio R/F = 1/2 and R/W = 1/8. In the first sample series (A series) Na$_2$CO$_3$ in a concentration of 1.4×10^{-4} M was used as a polymerization catalyst. In the second case (AHPt series) a Pt-doped aerogel was prepared by replacing Na$_2$CO$_3$ by [Pt(NH$_3$)$_4$]Cl$_2$ and fitting the Pt concentration to the 1 wt% on the final carbon materials. The mixtures were stirred to obtain homogeneous solutions that were cast into glass moulds (75 cm length × 0.5 cm internal diameter) and cured. After the cure cycle, the gel rods were cut into 5 mm pellets, supercritically dried with carbon dioxide to form the corresponding aerogels and finally carbonized.

Pyrolysis of the monolithic organic aerogels to obtain the corresponding monolithic carbon aerogels was carried out in N$_2$ flow at 100 cm^3 min^{-1} and by heating to either 500 or 1000 °C at a heating rate of 1.5 °C/min and a soaking time at constant temperature of 5 h. These carbon aerogels will be referred to in the text as A or AHPt and adding the carbonisation temperature (500 or 1000 °C). A-Pt catalysts series were prepared by impregnation (1 wt%) of the A – carbon aerogels using [Pt(NH$_3$)$_4$]Cl$_2$ as a precursor, while the doped AHPt carbon aerogels were used directly as catalysts. Catalysts were pretreated at 300 °C in H$_2$ for 3 h, prior to their characterization or catalytic test.

Characterization of samples was carried out by N$_2$ and CO$_2$ adsorption at −196 and 0 °C, respectively. Adsorption isotherms were measured using a volumetric device (Autosorbe-1 Quantachrome). The apparent BET surface areas were calculated from the nitrogen adsorption isotherms. The micropore volume, W_0 and the characteristic adsorption energy, E_0 were obtained by the application of the Dubininin–Radushkevich (DR) equation to both CO$_2$ and N$_2$ adsorption isotherms [14]. Then, the mean micropore width, L_0, was obtained by applying the Stoeckli equation [15]. Mercury porosimetry was carried out up to a pressure of 4200 kg/cm^2 using Quantachrome Autoscan 60 equipment. Pt dispersion was studied by high-resolution transmission electron microscopy (HRTEM) and XRD.

Static adsorption measurements of toluene were carried out by exposing 0.2 g of each sample, previously dried at 120 °C, in a desiccator containing toluene or acetone at 25 °C ($P_0 = 26.7$ or 240.4 Torr, respectively). In these experiments, samples were in contact with a saturated atmosphere of the organic vapour until equilibrium was reached, when the total adsorption capacity of the samples was determined. Samples were weighed every day until no increase in weight was observed. Dynamic VOC adsorption from a VOC/air mixture was measured at different temperatures using a thermobalance. Prior to the adsorption experiments, samples were pretreated at 250 °C in air flow (60 cm^3 min^{-1}). All samples showed good stability in this temperature range. They were then cooled down to the adsorption temperature and after stabilization the air flow was replaced by a similar air flow saturated with the corresponding VOCs, recording the weight gained as a function of time. Some regeneration experiments were also carried out by heating in pure air after saturation.

VOC catalytic combustion was conducted in a glass, U-shaped reactor operating in continuous mode at atmospheric pressure. In all cases, 0.10 g of catalyst in powder form was used. The reactant gas consisted of an air flow saturated with VOCs by bubbling through a saturator filled with liquid VOCs cooled to obtain a toluene concentration of 0.15 vol% (1500 ppm). The concentration was always confirmed by gas chromatography before the experiment. The total flow of the reactant mixture was controlled by a flow controller at 3.6 L/h. The reaction was performed at temperatures ranging from 130 to 230 °C, and always from high to low temperature. Analysis of the reaction products was carried out by online gas chromatography using a Perkin Elmer gas chromatograph, model 8500, with a thermal conductivity detector and Paraplot Q capillary column. The only primary products found were CO$_2$ and H$_2$O. Other products were not detected under the experimental conditions used. In all cases, conversion was obtained on the basis of both toluene consumption and CO$_2$ formation.

3. Results and discussion

The textural properties of the samples are summarized in Table 1. Mesoporous supports were obtained in the A-series, while the Pt-doped carbon aerogels are macroporous materials. It is noteworthy that both APtH-500 and APtH-1000 present a similar pore size distribution (PSD) with a mean macropore size of 100 nm. In the A-series the mesopore volume slowly decreases as the carbonization temperature increases and the PSD also became monomodal around pores of about 14 nm.

As regards microporosity, it is well known that this porosity range is produced during the carbonization process, as a consequence of the release of pyrolysis gases [16]. Thus, macro-mesoporosity is influenced by the R–F polymerization conditions and microporosity by carbonization. It is therefore observed that when the carbonization temperature increases from 500 to 1000 °C, there is an increase in the micropore surface (S_{mi}) in both catalyst series as a consequence of the greater micropore volume (W_0) produced by a higher degree of pyrolysis. It is also noteworthy that the S_{N2} is always smaller than S_{mi}, indicating the diffusion restrictions of N$_2$ at 77 K to be adsorbed in the narrowest micropores [17,18]. These diffusion restrictions increase as the carbonization
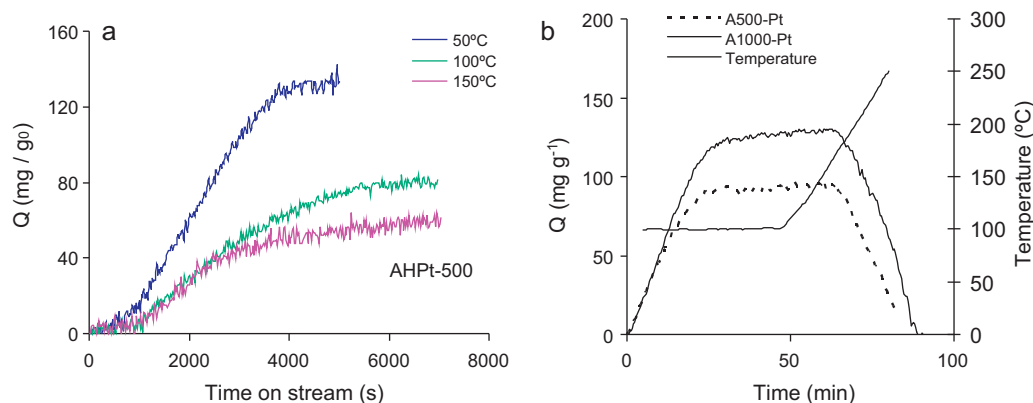
Fig. 1. Dynamic toluene adsorption: (a) effect of the adsorption temperature, (b) effect of the carbonization temperature.

temperature increases, so increasing the differences between S_{mi} and S_{BET}.

The VOC adsorption capacity (Q) of the samples was studied in static or dynamic conditions. The total toluene and acetone adsorption capacities at room temperature were determined in static conditions and the results are summarized in Table 2. Various authors point out that the VOC adsorptive behavior of carbon materials is related to the micropore volume, and in particular to the narrowest micropores less than 0.7 nm wide [19,20]. However, in this series of samples the microporosity characteristics only change slowly (Table 1), while the VOC adsorption capacity differs strongly between meso/microporous and macro/microporous carbon aerogels, a fact which demonstrates the contribution of mesopores to the adsorption capacity at room temperature.

In the case of the AHPt-series, the high macropore volume facilitates access to the microporosity, but does not contribute to the adsorption capacity, which is therefore only determined by the micropore characteristics. In this series, as the carbonization temperature increased, the micropore volume and mean micropore size also increased. This led to a significant increase in the toluene adsorption capacity. The acetone adsorption capacity on the contrary decreased after carbonization at 1000 °C. In the A-series, the toluene adsorption capacity always exceeded that of acetone, and both decreased as carbonization temperature increased. This behavior is the result of textural and chemical aspects. The acetone molecule measures 6.3 Å, while toluene is 3.7 Å width × 7.0 Å length. The flat shape of the toluene molecule makes it easier to adsorb into the narrowest, slit-shaped micropores than acetone. In the case of the A-series moreover, the adsorption capacity decrease for both VOCs should be related to the decrease of the mesoporosity and the narrow microporosity of sample A1000.

Textural characteristics have a clear influence on the adsorption process, which is also affected by the interaction of the VOCs with the carbon surface, which depends on the chemical nature of the adsorbent–adsorptive. The presence of oxygenated surface groups on the carbon surface favours hydrophilic interactions with water or other polar solvents. The dipolar moment of Toluene is 0.4D while for acetone it is 2.91D. This means that while acetone adsorption is favoured in the samples obtained at low carboniza-

tion temperature, for toluene adsorption, the opposite is true. Thus, toluene adsorption is enhanced with increasing carbonization temperature, first by the progressive hydrophobic character of the carbon surface, which favours chemical interactions, and secondly, from a textural point of view, because toluene can access the greater volume of albeit narrower microporosity, due to the flat shape of the toluene molecule. However, acetone adsorption is favoured in large micropores with hydrophilic surfaces obtained at low carbonization temperature.

Toluene adsorption was studied in dynamic conditions. As expected, the adsorption capacity, and indeed the adsorption rate, decrease as temperature increases (Fig. 1a). With increasing temperature, adsorption only occurs in progressively narrower pores, thereby decreasing the adsorption capacity. If we compare the adsorption/desorption cycles of samples at constant temperature (Fig. 1b), we observe that the toluene adsorption rate is quite similar for all the carbonized aerogels, and is independent of the catalysts or carbonization temperature because the L_0 values are always around 0.57 ± 3 nm, while the adsorption capacity is related to the micropore volume. As in previous results [16] toluene adsorption is reversible by heating in this kind of sample (Fig. 1b).

Fig. 2 shows the evolution of the amount of toluene adsorbed in dynamic conditions as a function of temperature in both Pt-doped carbon aerogels. In both cases, the mass gain during the experiments in the thermobalance is zero from 200 °C. In the case of AHPt-500, the adsorption capacity decreased linearly over the entire temperature range (between 25 and 200 °C) indicating a continuous evolution of the micropore PSD, however, in the case of AHPt-1000, as commented, the adsorption capacity was higher and also decreased linearly between 25 and 150 °C. However, above this temperature there was however a strong decline in adsorp-
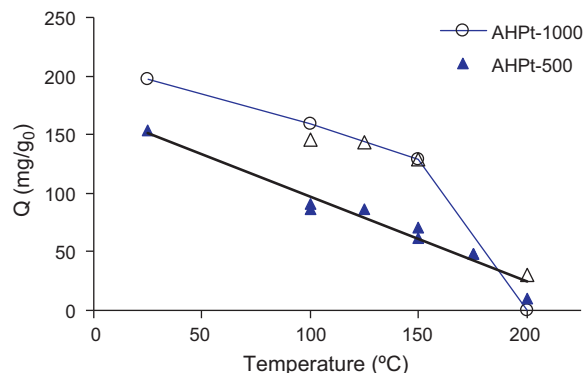
Table 2
Toluene and acetone adsorption capacities at saturation under static conditions at 25 °C.

Sample	Toluene Q (mg/g)	Acetone Q (mg/g)
A-500	715	500
A-1000	529	475
AHPt-500	154	194
AHPt-1000	197	174

Fig. 2. Evolution of toluene adsorption capacity as a function of temperature on Pt-doped carbon aerogels.
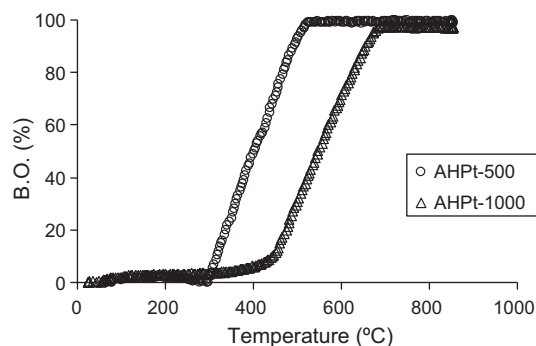
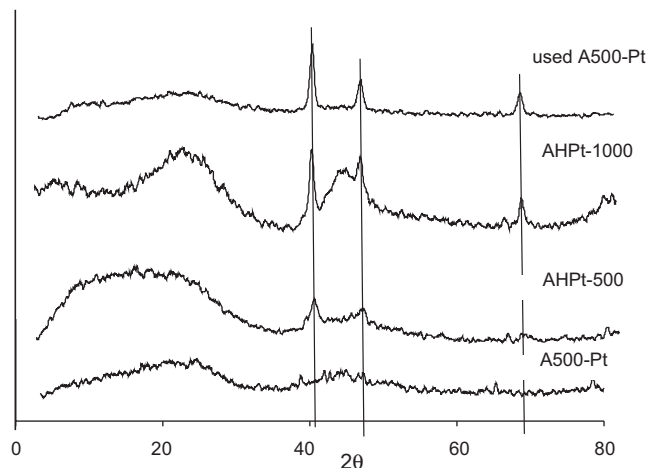**Fig. 3.** Thermal stability of Pt-doped carbon aerogels in air.

Fig. 4. XRD patterns of fresh and used catalysts.

tion capacity. Due to the presence of active combustion metal (Pt) together with VOC adsorption, VOC combustion occurs simultaneously on the carbon aerogel surface and the dependence on temperature of both processes works in clearly opposing directions.

In order to study the possibility of using these materials as adsorbents/oxidation catalysts, we must first investigate their stability in air as a function of temperature. Fig. 3 shows how the thermal stability of the samples increases with increasing carbonization temperature, because in this direction, the pyrolysis percentage was also higher, the weaker chemical groups evolved as gases during carbonization and the solid C–C structure was reinforced [16,21]. In this way we observed that samples obtained at 500 °C are stable in air up to 300 °C, while those obtained at 1000 °C can be used at around 400 °C in air.
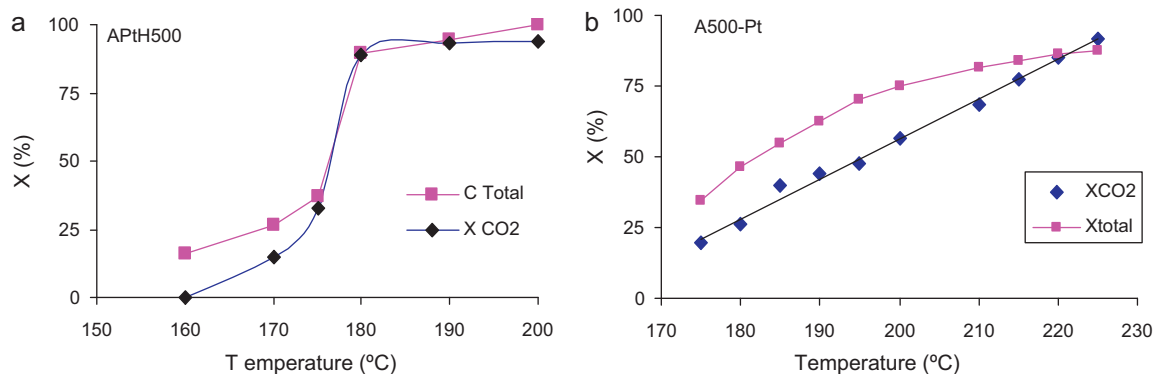
The performance of Pt/C catalysts is mainly controlled by Pt-dispersion. In previous research [7] we showed that combustion activity is favoured by Pt-particle size. After pretreatment, the supported catalysts show a better Pt-dispersion than the doped carbon aerogels, because sintering is favoured in the latter during carbonization at high temperature. Fig. 4 shows that XRD peaks corresponding to Pt were not observed for the supported catalyst after pretreatment at 300 °C, and that Pt-crystal size increased with increasing carbonization temperature in the AHPt series. Nevertheless, the XRD patterns for the samples showed that a strong sintering occurs during reaction mainly on the supported catalyst, because the mobility of Pt-particles is greater than in the case of doped carbon aerogels, where Pt-particles are surrounded or trapped by the carbon matrix.

Fig. 5 shows some typical light-off curves for doped and supported Pt-catalysts. Conversion was calculated on the basis of VOC disappearance (total conversion X_T) and CO_2 formation (X_{CO2}) obviously taking into account the carbon number of each VOC molecule. It should be mentioned that, as in previous works [5–9], this catalytic C/Pt system produces VOC combustion selectively to $CO_2 + H_2O$, i.e. no CO or intermediate VOCs were detected by GC or MS independently of the target VOC. At low reaction temperature and independently of the VOC target, $X_T > X_{CO2}$ as a consequence of VOC adsorption on all the porous carbon aerogels. The temperature range where this occurs depends on the nature of the VOC and the porous texture of carbon aerogels, i.e. on the interactions between both phases. Fig. 5 shows that the ability of these catalysts for acetone combustion is less than that for aromatic (xylenes) compounds. The combustion of p-xylene is complete at 180 °C while in the same experimental conditions acetone combustion only reached around 25%. Similar results were found in the bibliography. O'Malley [22] for example described that the reactivity pattern varies on the different Pt–inorganic supports as: alcohols > aromatics > ketones > carboxylic acids > alkanes, according to the weakness of C–H bonds.

To highlight the equilibrium between both adsorption and catalytic processes, we performed experiments in which the weight of the catalyst was changed so as to vary the contact time. The results obtained in the m-xylene combustion at 190 °C are shown in Fig. 6. At the beginning of the reaction, 100% of the m-xylene was removed, but the X_{CO2} was zero. This clearly indicates that the pollutant is adsorbed initially. The adsorption capacity progressively declines and thus after 2 h on stream, X_T and X_{CO2} presented very low values. After that, both parameters increased simultaneously, reaching total m-xylene combustion after around 17 h of reaction. The increasing combustion activity was favoured by Pt-sintering during reaction as was previously described [6] and demonstrated by the XRD results set out above. By increasing the contact time (using 0.3 g of catalyst), $X_T = 100\%$ after 2 h, because the adsorp-
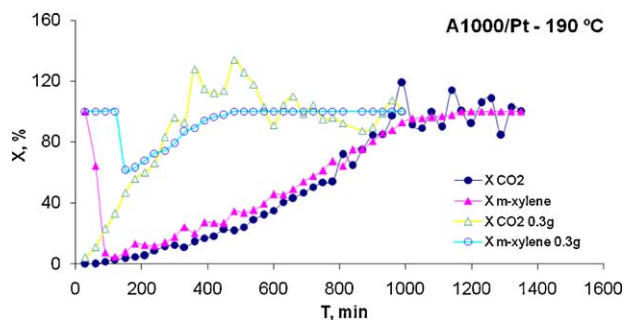

Fig. 5. Light-off curves for the combustion of (a) p-xylene and (b) acetone. 0.1 g catalyst, $F = 60 \, cm^3 \, min^{-1}$, [VOC] = 1500 ppm.

Fig. 6. Influence of contact time on the development of m-xylene combustion.

Fig. 8. TG of A1000-Pt catalyst during the xylene combustion reaction.

tion capacity is still not saturated. The fact that a larger amount of catalyst is used also explains why the combustion process on the catalyst is more developed and thus X_T decay is smaller than in the previous case (in which a smaller amount of catalyst was used), because of the contribution made by catalytic combustion as showed by the evolution X_{CO2}. After that, both X_T and X_{CO2} again increased simultaneously, reaching total m-xylene conversion after 7 h of reaction. These results showed that adsorption is initially faster than the reaction and that combustion needs the pre-concentration of the VOC on the catalyst surface. Even after reaching total VOC combustion, oscillations in both X_T and X_{CO2} indicate that there is a continuous equilibrium between the adsorption/reaction process, and the appearance of X_{CO2} values of more than 100% demonstrated the combustion of previously adsorbed compounds.

Differences between both X_T and X_{CO2} depend on the particular nature of the catalyst and the VOC, but decreases, as pointed out by the light-off curves, and oscillations become shorter with increasing reaction temperature and reaction time, i.e. after reaching the stationary state at high conversion values. Thus, Fig. 7a shows the catalytic performance of A500-Pt on the p-xylene combustion at 170 °C. At this temperature, and after the stabilization period shown in Fig. 6, this catalyst is able to mineralize the xylene completely. Nevertheless, the X_{CO2} curve shows small oscillations above and below 100% conversion values. When the same catalyst is used in acetone combustion at 210 °C (Fig. 7b), the X_T is around 80%, and in these experimental conditions both X_T and X_{CO2} show large oscillations, more marked in the case of X_{CO2}. Previous research describes this oscillatory behavior as the result of coke deposition and coke burning [23] when Pt is deposited on inorganic supports such as zeolites or alumina. Nevertheless, we pointed out [16] the complete reversibility of the toluene adsorption process on carbon aerogels with different porous textures; therefore, the VOC interaction with the basic carbon surface is weaker than with the acidic surface of inorganic supports and VOCs are adsorbed but not transformed on stable coke or coke precursor.
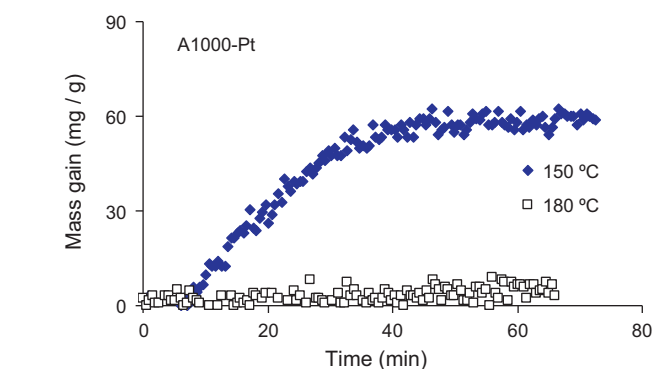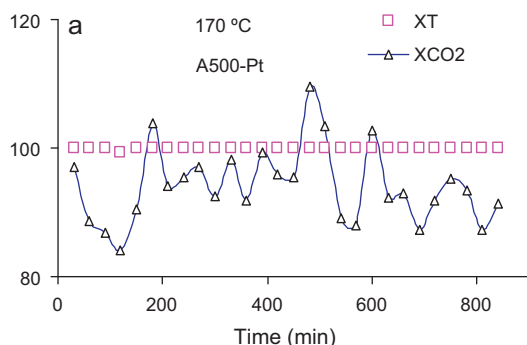
This is an important operational parameter to consider when using carbon materials, because the accumulation of VOC on the catalyst surface and its subsequent combustion can lead to a local increase in temperature, which can cause the carbon to burn with evident safety risks and financial consequences. In order to save energy, it is therefore necessary to determine previously the minimum temperature needed to obtain complete VOC combustion (determination of the light-off curves). The reactor should then be stabilized at this temperature prior to the introduction of the VOC in order to avoid VOC adsorption at lower temperatures. For aromatic compounds which are burned at around 170–180 °C, the use of a slightly higher reaction temperature (at around 200 °C) should be sufficient to minimize the combustion risk. Although the experimental conditions are different, the TG showed in Fig. 8 that the mass gain at 180 °C in xylene-saturated air is negligible, indicating that the adsorbed VOC molecules are quickly combusted.

Finally, and taking into account that exhaust gases can have a lower oxygen content, the efficiency of the catalyst in different oxidant environments was also studied. To this end, synthetic air flows were prepared by mixtures of N_2 and O_2 at different ratios. The results obtained for m-xylene combustion are shown in Fig. 9. It is noteworthy that the oxygen content does not affect xylene adsorption, and that the initial X_T decay (during ≈2 h on stream) is similar in all cases. Both VOC and oxygen are therefore adsorbed on different sites. As regards the combustion process, we observed a similar catalytic behavior when the oxygen content was cut by half (10% v/v), and catalytic performance was not enhanced by air enrichment to 30% O_2 either. However, there is a significant loss of efficiency at low oxygen content (5% v/v).

Alternatively, different mixtures of metal oxides show a good performance as combustion catalysts. The transition metal oxide complex V_2O_5–WO_3/TiO_2 was suggested as the most suitable for the VOC oxidation [24,25]. These authors also demonstrated that the conversion of the VOC is independent of the oxygen concen-
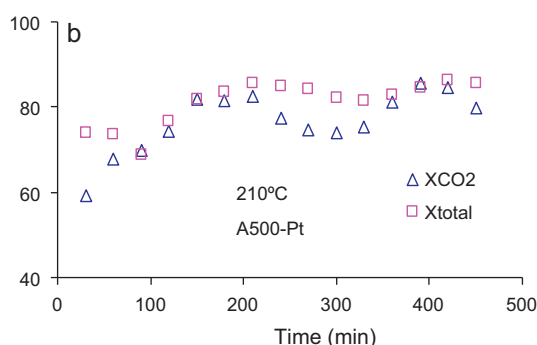

Fig. 7. Catalytic stability of A500-Pt on the combustion of (a) p-xylene and (b) acetone. 0.1 g catalyst, $F = 60$ cm^3 min^{-1}, [VOC] = 1500 ppm.
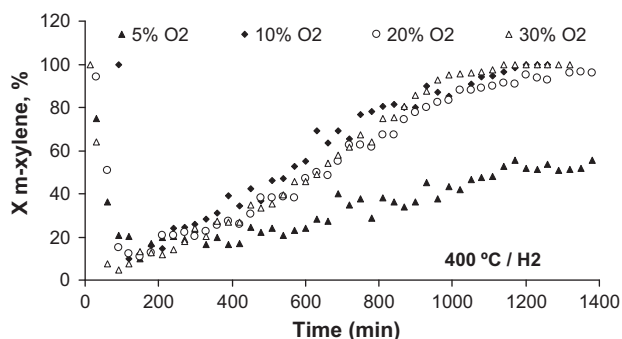
Fig. 9. Influence of oxygen content on catalytic performance.

tration, corresponding to a zero-order dependence of the reaction rate, which is in agreement to our results after 10% O_2. The catalytic activity and stability of nanocrystalline Cu $_{0.13}$Ce $_{0.87}O_y$ catalyst for acetone combustion were improved by using a co-precipitation method combined with a supercritical drying technique and calcination steps [26]. The best catalyst prepared in these conditions reached the total acetone conversion at 200 °C (1000 ppm acetone, 0.2 g catalyst). This catalytic system showed a good stability although at short reaction time undergoes certain deactivation due to the loss of surface area and the chemisorptions of water on the active sites. More recently [27] the same authors pointed out the participation of the oxygen surface groups on the catalytic performance by pulse reaction of pure acetone in the absence of O_2 over the Cu $_{0.13}$Ce $_{0.87}O_y$ concluding that deactivation of the catalysts in this conditions is due to the formation of bulk CuO. Reaction mechanism can be described by a Langmuir–Hinshelwood (LH) equation assuming reaction between adsorbed toluene and adsorbed oxygen on different adsorption sites [28]. These catalytic results are close to those previously presented for our catalysts.

As commented in the introduction section, we have developed combustion catalyst based either in metal oxides (WO$_3$) [5] or noble metals (Pt, Pd) [6–10]. In the first case, the active phase content reached 20 wt%, while using noble metals (Pt, Pd) the metal loading is always smaller than 1 wt%. The selectivity towards CO_2 is in general higher when use noble metals. The beneficial role of the hydrophobicity of carbon supports regarding the hydrophilic inorganic supports was also pointed out [9]. The use of carbon supports also facilitate that the active phase could be easily recovered for recycling only by burning the supports of spent catalysts. Textural and chemical characteristics of carbon aerogels can be fitted during the synthesis, by doping or chemical treatments. Therefore, in our opinion, the combination of porous hydrophobic supports and highly actives and selective phases can be the key for the development of future combustion catalysts. Different aspects regarding the control of the physic-chemical characteristics or thermal stability of catalysts for treatments of recalcitrant pollutants should be still optimized and the sol–gel synthetic route used in this manuscript clearly can be an useful tool.

4. Conclusions

The adsorption of aromatic compounds on carbon aerogels is favoured by the increasing carbonization temperature, because this increases the micropore volume and the carbon surface becomes more hydrophobic. Oxygenated compounds however are preferably adsorbed on carbon aerogels obtained at low carbonization temperature. The adsorption capacity is favoured at room temperature by the presence of mesopores while the macroporosity only contributes to micropore feeding. In dynamic conditions, the adsorption rate is quite similar in all cases because the microp-

ore size is also similar, as adsorption capacity is determined by the micropore volume as a function of the adsorption temperature.

With increasing temperature, adsorption is progressively restricted while catalytic activity is favoured. However even at high conversion values (high temperatures) oscillations in the conversion were observed as a consequence of the accumulation of adsorbed VOCs, something that should be avoided. However, VOC combustion is selective to CO_2, because we did not detect stable coke, CO or intermediate oxidized organic compounds. The efficiency of the catalysts is proved for long reaction periods and is independent of the oxygen content of the stream above 5% v/v of O_2.

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