H₂ storage in carbon materials

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Abstract In this work a series of commercial carbons with different structural and textural properties were characterised and evaluated for their application in hydrogen storage. The results showed that temperature has a greater influence on the storage capacity of carbons than pressure. The highest H₂ storage capacity at 298 K and 90 bar was 0.5 wt%, while at 77 K and atmospheric pressure it was 2.9 wt%. It is also showed that, in order to predict the hydrogen storage capacity of carbon material both at cryogenic and ambient temperature, the only use of BET surface area or total micropore volume obtained from N₂ adsorption isotherm may be insufficient, the characterization of the narrow microporosity is needed due to its high contribution to hydrogen adsorption capacity. The process involved in hydrogen storage in pure carbon materials seems to be physisorption. Morphological or structural characteristics have no influence, at least on gravimetric storage capacity.

Keywords Adsorbents · Carbon materials · Energy · Environmental systems

1 Introduction

Carbon is well known as one of the best adsorbents for gases. This property is due to the ability of this material to exhibit very different morphologies with a tailored and highly porous structure and the existence of particular interactions between carbon atoms and gas molecules. The goal

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of using carbons for hydrogen storage, as a durable, safe an efficient storage technology, can be achieved by packing hydrogen as close as possible, i.e. to reach the highest volumetric density by using as little additional material as possible. The second important criterion for a storage system is the reversibility of uptake and release.

During the last decade, different carbon materials have been studied as hydrogen adsorbents (i.e., nanotubes, nanofibers, activated carbons, etc.) (Viswanathan et al. 2003; Zhou et al. 2004; Schimmel et al. 2004; Parra et al. 2004; Buczek et al. 2005; Bénard and Chachine 2007; Zielinski et al. 2007). However, there are some discrepancies in the values obtained from different research studies (Dillon and Heben 2001; Tagaki et al. 2004; Strobel et al. 1999; Texier-Mandoki et al. 2004). The reasons for these discrepancies may be due to the difficulty of evaluating hydrogen storage capacity, the use of different techniques for making this evaluation, and the interpretation of the experimental data. Additionally, the processes involved in hydrogen adsorption are not yet very clear. Therefore, it is very difficult to compare samples and results in order to reach a general conclusion about the behaviour of the wide range of available carbon materials.

Furthermore, critical factors in a hydrogen storage system are temperature and pressure operating values. Most of the studies have accepted that at cryogenic temperatures carbon materials with a high micropore volume are the most appropriate for hydrogen adsorption (Jordá-Beneyto et al. 2007). However, from a practical point of view, materials that could operate at moderate temperatures would be more effective. Thus, a study of the temperature and pressure dependence of H₂ adsorption, in different carbon materials, is important in order to select the most adequate operating conditions and then evaluate the viability of using carbon materials as an alternative storage system.

Moreover, in order to optimise the best carbon adsorbent for hydrogen storage and to understand the $\rm H_2/carbon$ interaction mechanism, the relationship between hydrogen storage capacity, textural (surface area, total pore volume, micropore volume, pore size distribution) and morphological properties need to be established for a wide variety of carbon materials.

Several works have been published relating the textural properties of carbon materials with the hydrogen storage capacity. Most of them have concluded that BET surface area and total micropore volume obtained from N2 adsorption isotherm are the most important parameters influencing hydrogen storage (Tagaki et al. 2004; Panella et al. 2005; Zütel 2003; Bénard and Chachine 2007; Strobel et al. 1999; Nijkamp et al. 2001; Vasiliev et al. 2007). However, few authors have mentioned that textural characterisation using only N2 adsorption is insufficient to understand the behaviour of carbon materials as hydrogen adsorbent. Thus, the narrower microporosity evaluated by CO₂ adsorption isotherms may have a great contribution during hydrogen adsorption process. These studies have been performed for a particular carbon material at particular conditions (in most cases only at cryogenic conditions) (Texier-Mandoki et al. 2004; Gadiou et al. 2005; Thomas 2007). So in order to really understand the influence of the narrower microporosity during hydrogen adsorption, there is a need to study this fact on a wide variety of carbon materials and at different conditions.

In addition, carbon nanostructures received a big interest as hydrogen storage material due to the high capacities obtained in some laboratories (Dillon et al. 1997). During these last years, several works has been published claiming nanostructured carbons as good candidates for H₂ storage (Hou et al. 2003; Li et al. 2004; Furuya et al. 2004; Lee and Lee 2000; Ye et al. 1999; Liu et al. 1999; Chen and Huang 2007). However the values in the literature vary form 0.03 to 8%wt, maybe because the different type of determination, device, method of evaluation used, etc. Therefore, there is a need to evaluate the real hydrogen storage capacity of these nanoestructured carbons, at different temperatures and pressures, and then compare them with other carbon materials, in order to evaluate if the carbon morphology has an influence on H₂ storage.

Thus, the aim of this work is to analyze the $\rm H_2$ adsorption capacity of a wide series of commercial carbons with very different morphologies, under different operating conditions (i.e., temperatures and pressures), and to study the relationship of these adsorption capacities with their different textural parameters (obtained form $\rm N_2$ and $\rm CO_2$ adsorption isotherms) and morphological properties. The use of commercially available carbons for this study would facilitate the comparison of samples between different laboratories.



2.1 Carbon materials

A wide variety of carbon materials were used in the present work:

- (i) commercially available activated carbon, Maxsorb, from Kansai Coke and Chemicals;
- (ii) commercially available activated carbons from Norit: DLC Super 50 (referred to as PER50) and DLC Supra 50 (referred to as PRA50);
- (iii) a commercial activated carbon from Pica, PICATIF SC, here referred to as PICA;
- (iv) a commercially available carbon cloth from Actitex, WKL20;
- (v) a series of commercially available carbon cloths from Kynol Europa GmbH, ACC-507-20, ACC-507-15, ACC-507-10, referred to as ACC20, ACC15 and ACC10, respectively;
- (vi) commercially available high grade single-wall carbon nanotubes, SWNT, and multi-wall carbon nanotubes, MWNT, from Nanoledge Europe; and
- (vii) multi-wall carbon nanotubes synthetised by CRMD (Delpeux et al. 2002), here referred to as MWNT2.

2.2 Sample characterisation

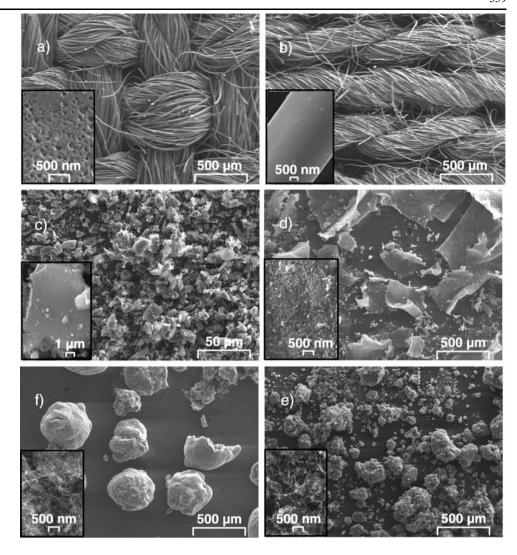
Textural characterisation of the pull of 11 samples was performed by the physical adsorption of N_2 at 77 K and CO_2 at 273 K. The micropore volume was determined by the Dubinin-Radushkevich equation (Dubinin 1960), taking these micropore volume and the mean pore size (L) from the equation of Stoeckli and Ballerini (1991) the micropore surface can be calculated (S mic = 2V mic /L). The micropore size distribution of the samples was obtained by means of the DRS method (Bansal et al. 1988; Dubinin and Stoeckli 1980) applied to the CO_2 adsorption isotherms.

2.3 Hydrogen adsorption measurements

Hydrogen adsorption measurements were carried out at 77 K and room temperature. Hydrogen isotherms were carried out at low pressures and at 77 K, in an automatic volumetric apparatus, whilst isotherms at room temperature were performed in a high pressure gravimetric analyzer. In the latter case, the manifold volume and buoyancy effect were corrected by carrying out helium isotherms. Leaks and background noise were also evaluated. Before performing the isotherms, in the volumetric or gravimetric apparatus, the samples were degassed in situ at 523 K under vacuum.



Fig. 1 SEM photographs of carbons with different morphologies: (a) carbon cloth ACC20, (b) carbon cloth WKL20, (c) active carbon PICA, (d) SWNT, (e) MWNT and (f) MWNT2



3 Results

Figure 1 shows the SEM photographs of a selection of the carbons studied. Samples ACC10 and ACC15 are very similar to ACC20 and so they were not included in this figure. Similarly, the photographs of the activated carbons PER50, PRA50 and Maxsorb did not provide any additional information to PICA photograph, and so they were excluded too. It is clear that the morphology of the samples in Fig. 1 are very different, there being a marked difference not only between the carbon cloths and the powdered activated carbons, but also between the two carbon cloths ACC20 and WKL20. The fibers that compose both cloths present very different surface textures, as can be seen in the magnified images shown in the bottom left-hand corner of the photograph. Furthermore, the morphologies of the nanotubes, both the single and the multiwalled nanotubes are quite different.

The N₂ adsorption isotherms for all the samples studied are shown in Fig. 2. It can be seen that the materials selected for this study cover a wide range of carbon-

based materials, not only concerning their morphologies (Fig. 1) but also from a textural point of view. The different shapes of the isotherms, according to the BDDT classification (Brunauer et al. 1940) provide useful qualitative information on the porous features of the selected materials. For instance, the carbon cloths (ACC10, ACC15, ACC25 and WKL20) present a type I isotherm, which corresponds to materials with a large contribution of micropores. Moreover, the abrupt knee of the isotherms at low relative pressures indicates that microporosity of these samples is mainly composed of pores of small diameter, in a narrow distribution of pore sizes. This is more remarkable for ACC10 and WKL20.

However, Maxsorb and PICA carbons possess a type I with somewhat type IV character isotherm, pointing to microporous carbons with a considerable development of mesoporosity. These activated carbons clearly present a high amount of N_2 adsorbed, along with an opening of the knee of the isotherms with a more gradual approximation to the plateau. This is usually associated with a widening of mi-



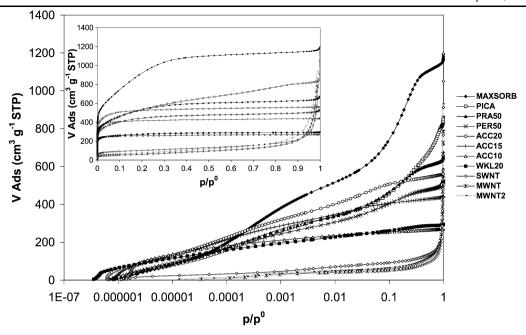


Fig. 2 N_2 adsorption isotherms of the carbons studied at 77 K

croporosity. Moreover, the isotherms of PICA and PRA50 carbons superimpose at relative pressures below 0.3, indicating likely similar pore size distributions in the micropore range. In contrast, at higher relative pressures the PRA50 carbon presents an almost plateau (isotherm parallel to abscises) whereas the amount of nitrogen gradually increases in the case of PICA, resulting in its hybrid I–IV character. This points out the presence of a higher volume of mesopores of larger sizes in the latter carbon.

Furthermore, the nanostructured materials (nanotubes) present the lowest micropore development, with N_2 adsorption isotherms belonging to the type II of the abovementioned classification. The upward increase at relative pressure above 0.8 indicates the presence of complementary mesopores coming from interparticle voids or the empty space between the bunches of nanotubes.

As was mentioned above, microporous carbon materials seem to be the most appropriate for hydrogen adsorption but theorical and experimental studies have shown that the diameter of the micropore also affects hydrogen storage capacity (Gadiou et al. 2005; Rezpka et al. 1998). For this reason, it is very important to obtain as much as information as possible about the full microporosity range, including the narrow micropores. Thus, the textural characterisation of these materials was complemented with the adsorption of CO₂ at 273 K (Fig. 3). The use of CO₂ adsorption at 273 K for a proper textural characterisation of the narrow micropores, has been established as an effective procedure (Jagiello and Thommes 2004; Lozano-Castello et al. 2004). Thus, the combination of N₂ and CO₂ adsorption isotherms data provides complementary and relevant information about the mi-

croporosity of the samples. As can be seen from Fig. 3, except for carbon nanostructures that present a very low CO_2 adsorption, all the carbon materials exhibit narrow microporosity. However, in this case, carbon cloths show a higher CO_2 adsorption than activated carbons such as Maxsorb and PICA, at both low and high relative pressure. This indicates the presence of a larger amount of narrow micropores in this type of carbon materials. Detailed characteristics of their corresponding pore structures are presented in Table 1. The total micropore volume, V_{N2} , and the narrow micropore volume, V_{CO2} , were obtained by applying the Dubinin-Radushkevich equation to the N_2 and CO_2 adsorption isotherms, respectively. For comparison purposes, Table 1 includes the BET surface area evaluated as described elsewhere (Parra et al. 1995).

The carbon materials studied in this work present very different porosities: samples with a very low development of porosity such as the nanotubes (with BET surface areas of less than $340 \text{ m}^2 \text{ g}^{-1}$), and samples with a very well developed porosity such as the activated carbons with BET surface areas up to 2558 m² g⁻¹ for Maxsorb. Moreover, the 11 samples studied also present different micropore size distributions. There are samples with similar micropore volumes calculated from the CO₂ and N₂ isotherms (i.e. ACC10), indicating a narrow micropore size distribution. On the other hand, there are other samples with higher V_{N2} than V_{CO2} , indicating a wider micropore size distribution, the widest being the one corresponding to Maxsorb activated carbon. This is also corroborated by the micropore size distribution, obtained from applying the DRS method to the CO₂ adsorption isotherms, presented in Fig. 4.



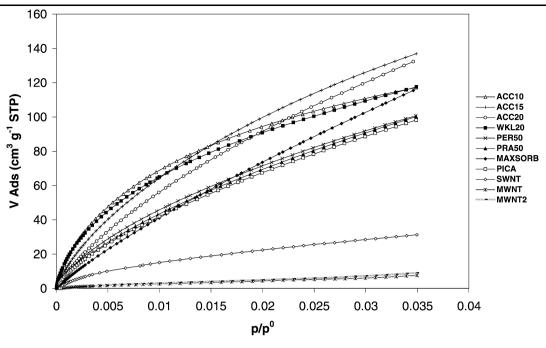


Fig. 3 CO₂ adsorption isotherms of the carbons studied at 273 K

Table 1 Textural properties of the samples studied

Sample	N ₂ adsorption, 77 K			CO ₂ adsorption, 273 K	
	S_{BET} $(m^2 g^{-1})$	V_{N2}^{a} (cm ³ g ⁻¹)	S_{micN2}^{b} $(m^2 g^{-1})$	$ \overline{V_{\text{CO2}}^{\text{a}}} \\ \text{(cm}^{3} \text{g}^{-1}) $	S_{micCO2}^{b} $(m^2 g^{-1})$
ACC10	1052	0.40	1230	0.40	1189
ACC15	1639	0.67	1067	0.44	1139
ACC20	1670	0.79	1112	0.50	1122
WKL20	1049	0.39	957	0.36	956
PER50	1501	0.62	758	0.29	784
PRA50	1730	0.72	747	0.27	725
MAXSORB	2558	1.35	688	0.29	682
PICA	1271	0.75	1428	0.27	720
SWNT	340	0.12	147	0.06	268
MWNT	260	0.08	94	0.02	48
MWNT2	246	0.10	104	0.03	62

^aCalculated from the Dubinin-Radushkevich equation ^bCalculated from combining the Dubinin-Radushkevich and Stoeckli equations

The $\rm H_2$ adsorption capacity of the series of carbons was evaluated at two different conditions: 77 K up to atmospheric pressure, and at 298 K up to 90 bar. The aim was to evaluate the effect of temperature and pressure on $\rm H_2$ adsorption capacity for the different carbon materials selected.

Figure 5 presents the H_2 adsorption isotherms at 77 K of the 11 samples studied. It can be observed that the nanotube samples (i.e., SWNT, MWNT and MWNT2) present the lowest hydrogen adsorption capacities. In general it can be said that the higher the BET surface area, the higher the H_2 adsorption capacity at 77 K and atmospheric pressure. The highest BET value and therefore the highest H_2 adsorp-

tion capacity corresponds to Maxsorb, followed by samples ACC20 and ACC15. However, this is only a general rule, and there are other characteristics (i.e., micropore size distribution) that have a great influence on the H_2 adsorption capacity. Thus, sample PRA50 has a BET surface area ca. $500~\text{m}^2~\text{g}^{-1}$ higher than PICA (i.e., 1730 and 1271 $\text{m}^2~\text{g}^{-1}$, respectively) and both samples present similar H_2 adsorption capacities. This could be due to the strong influence of the narrow microporosity on H_2 adsorption; as can be seen from Table 1, both samples present a similar V_{CO2} , narrow micropore surface area, S_{micCO2} , and pore size distribution (Fig. 4). Moreover, sample PER50 has a higher BET surface



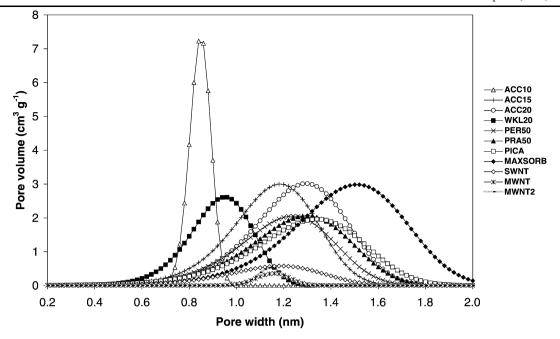


Fig. 4 Pore size distribution obtained by application of the DRS equation to the CO₂ adsorption isotherms of the 11 samples studied

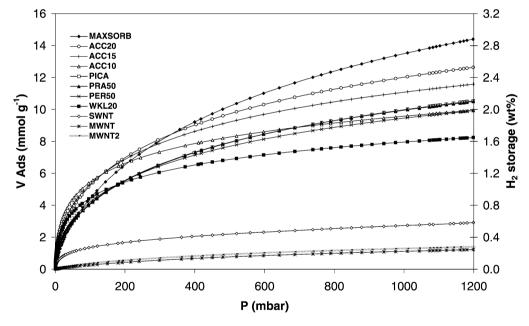


Fig. 5 H₂ adsorption of the carbons studied at 77 K

area than ACC10 (i.e., 1501 and $1052 \text{ m}^2 \text{ g}^{-1}$, respectively) but the latter has a narrow micropore size distribution, as mentioned before, and this plays an important role in H_2 adsorption, as can be seen in Fig. 5 where higher amount of H_2 is adsorbed in ACC10 at low pressures and similar value (i.e., 2 wt%) at high pressure.

According to these results, it can be said that the narrow microporosity makes the main contribution to hydrogen adsorption capacity in carbon materials, but as wide microporosity also plays a role, the general rule is that the higher the surface area, the higher the hydrogen adsorption capacity. However, in this work it is shown that in order to predict the hydrogen storage capacity, independently of the type of carbon material used, it is crucial to take into account the presence of the narrow microporosity, not determined by N_2 adsorption (and thus not evaluated in BET surface area) but totally accessible to H_2 and therefore contributing to the storage capacity of the carbon material.



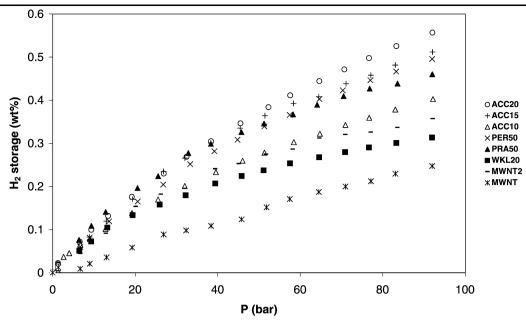


Fig. 6 H₂ adsorption of the carbons studied at 298 K

Figure 6 shows the H₂ adsorption isotherms at room temperature and at high pressure, carried out in the gravimetric analyzer. It can be seen that at room temperature the H₂ adsorption capacity is very low, even at pressures of 90 bars. This is due to the weak interaction between the hydrogen and the carbon surface, which is based on van der Waals forces, and becomes stronger at low temperatures. Thus, temperature seems to be the most important factor, or at least a factor with greater influence than pressure, for H₂ storage in pure carbon materials, where physisorption is the main mechanism of uptake. The highest H2 storage capacity at 298 K and 90 bar was 0.5 wt%, while at 77 K and atmospheric pressure it was 2.9 wt%. By combining temperature and pressure and other factors such as the surface chemistry in order to changing the nature of the H₂/carbon interaction, the adsorption capacity of the carbon samples would probably be improved, but this optimization was not the aim of this work.

From the shape of the isotherms presented in Figs. 5 and 6 it can be said that, most of the samples do not present a saturated isotherm, especially the isotherms at high pressure, compared to some of the data already reported (Wong-Foy et al. 2006). As it was mentioned before, the contribution of the narrow microporosity was shown in previous work for some carbon materials at cryogenic temperatures (Texier-Mandoki et al. 2004). In the present work, the influence of the narrow microporosity was also studied at different temperatures and pressures for the wide variety of carbons studied. In Table 1, it can be seen that activated carbon PRA50 has higher BET surface area than the activated carbon cloth ACC15; however the hydrogen storage capacity at ambient

temperature and high pressure is higher for the latter one (Fig. 6). This could be explained by the different narrow microporosity content: $V_{\rm CO2}$ value for ACC15 nearly double the one belonging to PRA50. This indicates that the narrow microporosity contributes strongly to hydrogen adsorption at ambient temperature, as it happens at cryogenic conditions (Texier-Mandoki et al. 2004).

In order to see more clearly the variation of hydrogen adsorption with the textural properties of carbons, Fig. 7 shows the relationship of the amount of hydrogen adsorbed (weight percent) versus the BET surface area. The small effect of surface area on H₂ storage capacity at 298 K and at high pressure, can be due to the low adsorption capacities obtained at these conditions. When the H₂ adsorption is performed at low temperature (i.e., 77 K), even at atmospheric pressure the adsorption capacity is notably higher and the effect of BET surface area is noticeable. The values of the adsorption capacity range from 0.2 to 2.9 wt% at 77 K/1 bar, whilst the values range between 0.2 and 0.5 at 298 K/90 bar.

Figure 7 shows some scattering. Thus rather than a linear relationship there is a tendency for the storage capacity to increase with the increase in BET surface area. Although it should be noted that this is only a general tendency, as can be seen that 4 samples, ACC10, PICA, PER50 and PRA50 with different BET surface areas of 1052, 1271, 1501 and 1730 m² g⁻¹, present nearly the same H₂ storage capacity (2.0, 2.1, 1.98 and 2.1 wt%, respectively). Furthermore, it can be seen that some samples with a similar BET surface area present some different hydrogen adsorption capacities; all of which indicates that although the BET surface area has



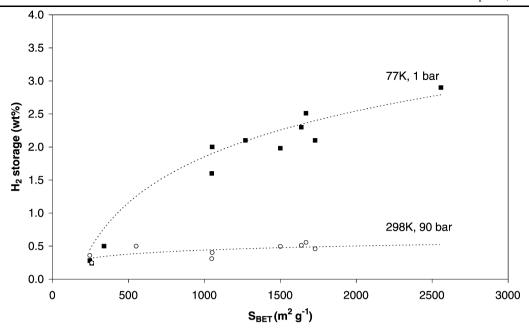


Fig. 7 Amount of hydrogen adsorbed at 77 K/1 bar, and 298 K/90 bar versus the BET surface area of the samples studied

an influence on the H_2 storage capacity, it is clearly not the only influencing factor.

In order to illustrate the relationship between the H_2 adsorption capacity and the presence of micropores, Fig. 8 shows the relationship between the weight percent of hydrogen storage, at the two different operating conditions studied, and the total micropore volume (V_{N2}) , Fig. 8a, and the so called narrow micropore volume (V_{CO2}) , Fig. 8b.

Similar conclusions can be drawn from Figs. 7 and 8a. There is a general tendency for the H₂ adsorption capacity to increase, especially at 77 K, with the increase in micropore volume V_{N2}. However, there are samples with similar micropore volumes and different H₂ adsorption capacities. Figure 8b again shows an increasing tendency, with only one sample exhibiting a very clear deviation, which is the Maxsorb carbon. The reason for this deviation could be attributed to the broad micropore size distribution of this sample. This has already been mentioned above as inferred from the data of Table 1, where it can be seen the relatively low volume of narrow micropores (i.e., V_{CO2}) and high volume of total micropores (i.e., V_{N2}). Moreover, Maxsorb also present a wide narrow micropore size distribution (see Fig. 4). Taking into account these textural properties, Maxsorb should present lower H₂ storage capacity than it does. The high adsorption capacity is probably due to its large specific surface area. Thus, both surface area and pore size distribution have a role in the H₂ storage capacity at 77 K and 1 bar. Moreover, this deviation of Maxsorb from the general trend does not appear at 298 K and 90 bars, probably because at these operating conditions the pore size distribution is the main factor influencing on the storage capacity (Jordá-Beneyto et

al. 2007) and so the values obtained for Maxsorb follow the general trend.

Another important observation that can be made from this study is that the morphology of the carbon material does not seem to be an important factor for the gravimetric hydrogen storage capacity at any of the operating conditions studied in this work. It has been reported elsewhere (Cheng et al. 2001) that besides textural properties, the structural properties of certain carbon materials could enhance the H_2 /carbon interaction and thus increase storage capacity, the nanostructured carbons (i.e., nanotubes) being one of the most effective enhancers. However, according to the results obtained in this work, neither the nanostructures, nor the fibrous nature has a great influence on gravimetric storage capacity.

4 Conclusions

This work has studied the hydrogen storage capacity of various carbon materials of different nature, morphology and textural properties at two extreme conditions: low temperature (77 K) and atmospheric pressure and room temperature (298 K) and high pressure (90 bar). The results showed that temperature has a greater influence on the H₂ storage capacity of carbons than pressure. Furthermore, it is demonstrated that in order to predict the hydrogen storage capacity of certain carbon material, both at cryogenic and ambient temperatures, the only use of BET surface area or total micropore volume obtained from N₂ adsorption isotherm may be insufficient. The characterization of the narrow microporosity (micropore volume and micropore size distribu-



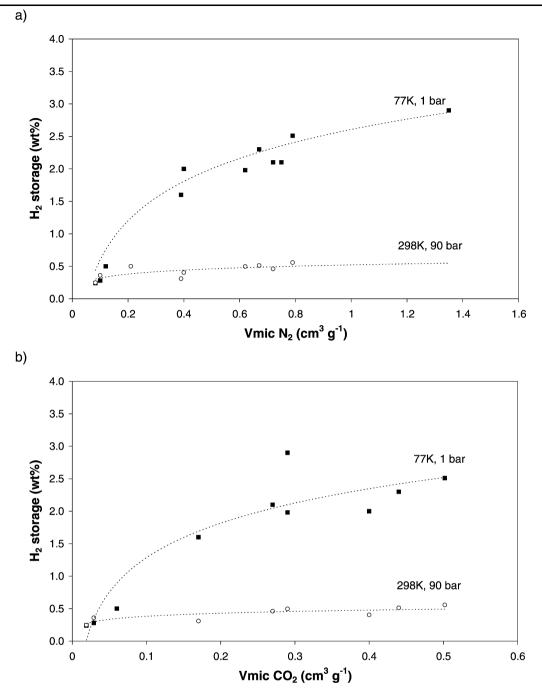


Fig. 8 Amount of hydrogen adsorbed at 77 K/1 bar, and 298 K/90 bar versus (a) the total micropore volume and (b) the narrow micropore volume of the samples studied

tion) is needed due to its high contribution to hydrogen adsorption. According to the results obtained, the gravimetric H₂ storage capacity of pure carbons depends on their textural properties and not on their morphology. A combination of low temperatures, high pressure, adequate textural properties, besides proper chemical surface, seem to be necessary to attain the ambitious targets set by the DOE and EU.

Nomenclature

 V_{N2} , total micropore volume (cm³ g⁻¹), obtained from the N_2 adsorption isotherm

 V_{CO2} , narrow micropore volume (cm³ g⁻¹), obtained from the CO_2 adsorption isotherm

 S_{micN2} , total micropore surface area (m² g⁻¹), obtained from the N_2 adsorption isotherm



- S_{micCO2} , narrow micropore surface area (m² g⁻¹), obtained from the CO_2 adsorption isotherm
 - S_{BET} , BET surface area (m² g⁻¹), obtained from the N_2 adsorption isotherm
 - V Ads, Volume adsorbed
 - STP, Standard Temperature and Pressure, 273 K and 1 bar
 - DRS, Dubinin-Radushkevich-Stoeckli method

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