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Standard nitrogen adsorption data for α-alumina and their use for characterization of mesoporous alumina-based materials

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Abstract Nitrogen adsorption isotherm measured at −196 °C for a macroporous α-alumina (α-Al_2O_3) is reported. This isotherm is compared with the previously reported adsorption data measured on LiChrospher 1000 silica and with available reference isotherms measured at moderate and high relative pressures on macroporous aluminas. The isotherm reported in this work for α-Al_2O_3 and that recorded previously on LiChrospher 1000 silica were used as reference data for adsorption characterization of ordered and disordered mesoporous aluminas by α_s-plot analysis and pore size analysis. It is shown that both reference isotherms provide almost identical adsorption characteristics of the aforementioned mesoporous aluminas, indicating that the available reference data for the silica surface are also suitable for adsorption analysis of alumina-based materials.

Keywords Nitrogen adsorption · Mesoporous alumina · Standard adsorption data · α_s-plot analysis · Porosity

1 Introduction

Gas adsorption has been widely used for characterization of surface properties and porosity of various solids (Rudzinski and Everett 1992; Jaroniec and Madey 1988; Sing et al. 1985; Kruk and Jaroniec, 2001). Namely, the specific surface area, pore volume, adsorption energy distributions (AED) and pore size distributions (PSD) are commonly evaluated on the basis of gas adsorption isotherms (Rudzinski and Everett 1992; Jaroniec and Madey 1988; Sing et al. 1985). While adsorption isotherm data at higher relative pressures offer information about formation of multilayer and capillary condensation of adsorbate in mesopores, the low pressure adsorption is used for evaluation of surface heterogeneity and microporosity (Rudzinski and Everett 1992; Jaroniec and Madey 1988; Kruk and Jaroniec 2001). Currently, there are advanced numerical methods for evaluation of the AED and PSD curves from gas adsorption isotherms; all these methods are based on the integral equation of adsorption, the solution of which requires assumptions about adsorption model on an energetically homogeneous surface and topography of adsorption sites (in the case of AED calculation; Rudzinski and Everett 1992; Jaroniec and Madey 1988) and about adsorption model for filling uniform pores of a given geometry (in the case of PSD calculation; Kruk and Jaroniec 2001; Kruk et al. 1998). Although the AED and PSD curves are extremely important for detailed characterization of energetic and structural heterogeneity of porous materials, their evaluation is model-dependent and can be problematic for materials with complicated heterogeneity. Thus, there is a great need for refinement of comparative plots such as α_S-plots and *t*-plots, which are model independent and can be easily used for obtaining basic information about surface properties and porosity of solids (Sing et al. 1985; Jaroniec and Kaneko 1997). For instance, the α_S-plot is obtained by plotting the amount of gas adsorbed on the solid studied against the standard reduced adsorption (the amount adsorbed on a nonporous

This work is dedicated to Professor Wladyslaw Rudzinski on his 70th Birthday.

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reference solid normalized by the amount adsorbed on this solid at the relative pressure of 0.40; Sing et al. 1985; Kruk and Jaroniec 2001). In the case of *t*-plot, the amount adsorbed on the sample studied is plotted against the statistical film thickness (t) obtained for a reference nonporous/macroporous solid (Jaroniec and Kaneko 1997). This type of plot allows one to obtain the total and external surface areas, the micropore volumes and even some information about surface properties of the sample studied (Jaroniec and Kaneko 1997; Kruk et al. 1997a).

If the aforementioned α_S-plot and *t*-plot methods are used for characterization of microporosity of the material studied, it would be better to use a nonporous reference solid of similar surface properties to those of the material under investigation (Jaroniec and Kaneko 1997; Kruk and Jaroniec 2001). For instance, nitrogen adsorption isotherm measured at −196 °C on a macroporous silica LiChrospher 1000 (Jaroniec et al. 1999) has been employed for determination of the statistical film thicknesses of nitrogen on the silica surface and used for the calculation of pore size distributions for ordered mesoporous silicas (OMS) (Kruk et al. 1997b). The micropore volumes for the polymer-templated ordered mesoporous silicas (OMS) have been estimated by α-plot analysis based on the reference adsorption data for the aforementioned LiChrospher 1000 macroporous silica (Kruk et al. 1997b). Analogous adsorption studies (Choma et al. 2002,2008) have been performed for porous carbons by using Cabot BP280 carbon black as a reference amorphous carbon material (Kruk et al. 1999a). Also, reference adsorption data have been published for hydrophobic surfaces obtained by attachment of longer alkyl groups to the silica surface (Kruk et al. 1999b) and for graphitized carbons (Gardner et al. 2001).

There are many other important materials such as mesoporous alumina (MA), which have been widely used as industrial catalysts and catalyst supports (Oberlander 1984; Trueba and Trasatti 2005) and as membranes for separations (Lin et al. 2002; van de Water and Maschmeyer 2004). These applications require accurate evaluation of the adsorption properties of these materials. Furthermore, amorphous (Yang et al. 1998; Niesz et al. 2005) and crystalline (Yuan and Yin 2008) ordered mesoporous alumina (OMA) and ordered alumina-supported metal oxides (Morris et al. 2008) have been prepared in the presence of triblock copolymers as soft templates. These materials exhibit large mesopore volumes and high specific surface areas, which may be attractive for advanced catalytic applications (Jacobs et al. 2002; Kwak et al. 2009). Hence, characterization of the surface properties of mesoporous alumina materials by gas adsorption is of great importance for further development of alumina-based adsorbents and catalysts. Therefore, it is not surprising that some researchers have published nitrogen adsorption isotherms for α-alumina standards (Matejova et al. 2008; Cejka et al. 2004), but in some cases these isotherms were reported at moderate and high relative pressures (Matejova et al. 2008). Also, Cejka et al. (2004) reported comparative adsorption analysis of alumina materials by using α-alumina as a reference solid; however, no comparison was provided to the previously published adsorption data for LiChrospher 1000 silica reference material.

In this work, the standard nitrogen adsorption isotherm measured at −196 °C on ultra-pure α-Al_2O_3 standard is reported and used for the α_S-plot analysis of selected alumina samples. Analogous analysis of these samples is performed by using LiChrospher 1000 silica as a reference solid. It is shown that only a negligible difference is observed at low and high relative pressures for the α_S-plots obtained under assumption of both reference materials, indicating that the previously reported adsorption isotherm on LiChrospher 1000 silica reference solid is suitable for adsorption analysis of porous alumina samples. These results are of great importance not only for the characterization of pure porous alumina materials, but also for silica–alumina composites, which are used for the preparation of porous membranes employed in catalysis and separations (Lin et al. 2002; van de Water and Maschmeyer 2004).

2 Experimental

2.1 Mesoporous alumina materials

A commercial α-alumina sample (denoted as Al_2O_3-R) from Sumitomo Materials (Japan) was selected as a reference nonporous solid for characterization of porous alumina materials. Nitrogen adsorption isotherm was measured on this sample to obtain the standard reduced adsorption on the alumina surface. For the purpose of illustration, these standard adsorption data were used to characterize two mesoporous alumina samples by the α_S-plot analysis. Nitrogen adsorption isotherms for the aforementioned porous alumina samples were reported by Fulvio et al. (2010). One of them is an ordered mesoporous alumina synthesized using a similar procedure to that reported by Yuan and Yin (2008). This sample was obtained by using about 2.0 g of $(EO)_{20}(PO)_{70}(EO)_{20}$ triblock copolymer (Pluronic P123 from BASF, Co.), which was dissolved in 20.0 mL of 99.5 + % anhydrous ethanol (Acrós Organics), and stirred for 4 h. Then, approximately 20 mmol of 98 + % aluminum isopropoxide (Acrós Organics) were added to the polymer solution followed by 3.2 mL of 68–70 wt% nitric acid (Acrós Organics) and 10.0 mL of anhydrous ethanol. The sol was stirred for 5 h at room temperature and gelled by evaporating the solvent

at 60 °C for 48 h in air under static conditions. The resulting polymer-pseudo boehmite composite was calcined at 400 °C in a horizontal quartz tube furnace with a heating rate of 1 °C min^{-1} and held at the final temperature for 4 h. The final material was denoted as OMA sample. Another mesoporous alumina was prepared by direct calcination of boehmite powder (Catapal A from Sasol) using the same heating program and conditions as in the case of the OMA sample. The resulting alumina exhibited disordered mesoporosity and it was labeled as MA sample.

2.2 Characterization

Nitrogen adsorption isotherms were measured at −196 °C using ASAP 2010 and 2020 volumetric adsorption analyzers manufactured by Micromeritics (Norcross, GA). Before adsorption measurements the calcined alumina samples were degassed under vacuum for at least two hours at 200 °C. The specific surface area of these samples was calculated using the Brunauer–Emmett–Teller method (BET) within the relative pressure range of 0.05–0.20 (Kruk and Jaroniec 2001). Pore size distributions were calculated using the BJH method for cylindrical pores improved by Kruk et al. (1997a), known as the KJS method. The statistical film thickness for nitrogen adsorbed on the α-Al_2O_3-R reference solid was obtained by multiplication of the corresponding nitrogen adsorption isotherm by a factor, which assured the best agreement of the resulting curve with the statistical film thickness for nitrogen on the LiChrospher 1000 silica surface within the relative pressure range of 0.20–0.70.

The XRD analysis was conducted for the calcined samples using a PANalytical, Inc. X'Pert Pro (MPD) Multi Purpose Diffractometer with Cu Kα radiation (1.5406 Å), with an operating voltage of 40 kV, 0.020° step size, 3 s step time and $10.0^\circ < 2\theta < 80.0^\circ$ at room temperature. The small angle XRD of the template-free OMA was measured using the same conditions, but 20 s step time and $0.400^\circ < 2\theta < 5.000^\circ$.

3 Results and discussion

3.1 XRD studies of the structural properties of alumina reference, OMA and MA materials

Figure 1a shows the wide-angle powder XRD spectra for alumina samples studied. The spectrum of reference alumina shows intense reflections for α-Al_2O_3 only (JCPDS 14-4268). This figure shows also the XRD spectra of boehmite powder (JCPDS 1-774) and the resulting MA material obtained after calcination of boehmite at 400 °C, which was assigned to γ-Al_2O_3 phase (JCPDS 1-1303). The γ-Al_2O_3 phase is typically stable up to 500 °C and above this temperature other transition phases may occur (Oberlander 1984); however, it can be stable at temperatures as high as 1,000 °C when it is formed from crystalline precursors (Trueba and Trasatti 2005). The OMA material remained amorphous after calcination up to ~800 °C (Yuan and Yin 2008; Morris et al. 2008); however, its small angle XRD pattern (Fig. 1b) exhibits three intense reflections due to the presence of ordered mesopores. These peaks can be assigned according to the *p6m* symmetry, with the most intense reflection being the (100) peak, characteristic for two-dimensional (2D) hexagonal ordered mesostructure.

3.2 Adsorption properties of reference α-Al_2O_3 and mesoporous aluminas

Nitrogen adsorption isotherm measured at −196 °C on α-Al_2O_3-R is provided in Table 1 as the standard reduced

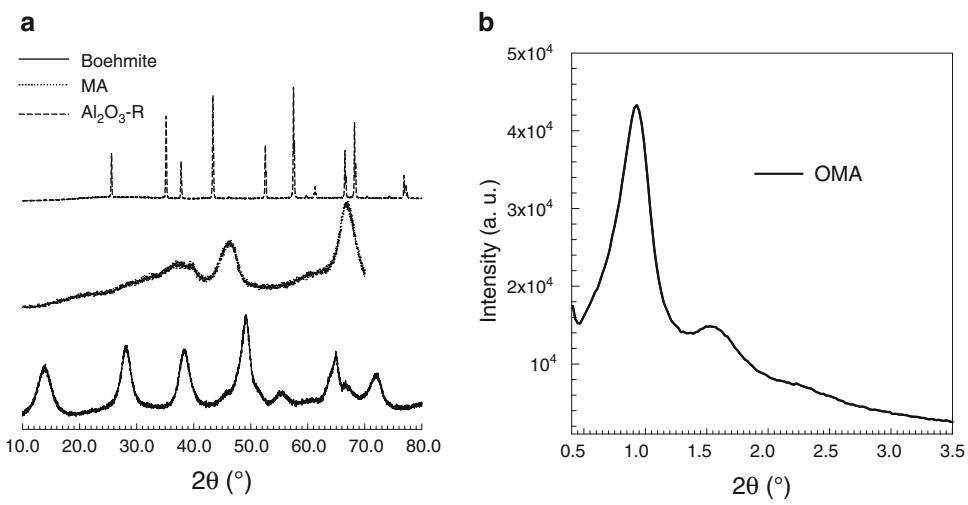

Fig. 1 Powder XRD patterns for reference alumina, boehmite and the resulting MA material obtained after calcination of boehmite at 400 °C (**a**). Small angle powder XRD pattern for the OMA sample calcined at 400 °C (**b**)

Table 1 Standard N_2 at −196 °C reduced adsorption isotherm for α-Al_2O_3-R material

p/p_0	α_S^a	p/p_0	α_S^a	p/p_0	α_S^a	p/p_0	α_S^a
1.8020×10^{-6}	0.0108	1.2351×10^{-3}	0.2563	0.1211	0.6500	0.6598	1.3387
4.1220×10^{-6}	0.0211	1.4000×10^{-3}	0.2645	0.1399	0.6765	0.6799	1.3756
7.3310×10^{-6}	0.0307	1.5729×10^{-3}	0.2720	0.1600	0.7048	0.7007	1.4155
1.1397×10^{-5}	0.0394	1.7312×10^{-3}	0.2783	0.1807	0.7316	0.7200	1.4528
1.5970×10^{-5}	0.0474	1.8943×10^{-3}	0.2841	0.2007	0.7566	0.7398	1.4943
2.3462×10^{-5}	0.0582	2.1997×10^{-3}	0.2940	0.2199	0.7907	0.7606	1.5359
3.2798×10^{-5}	0.0688	2.5156×10^{-3}	0.3030	0.2405	0.8144	0.7805	1.5848
4.4209×10^{-5}	0.0793	3.3018×10^{-3}	0.3213	0.2598	0.8369	0.8011	1.6290
5.7923×10^{-5}	0.0897	4.1309×10^{-3}	0.3366	0.2800	0.8599	0.8199	1.6865
7.4103×10^{-5}	0.0997	4.9855×10^{-3}	0.3497	0.3002	0.8811	0.8397	1.7715
9.3065×10^{-5}	0.1096	5.8571×10^{-3}	0.3609	0.3200	0.9044	0.8603	1.8610
1.1488×10^{-4}	0.1192	6.7447×10^{-3}	0.3709	0.3409	0.9281	0.8805	1.9648
1.3974×10^{-4}	0.1286	9.3171×10^{-3}	0.3949	0.3596	0.9509	0.9003	2.0866
1.6911×10^{-4}	0.1377	0.0115	0.4097	0.3800	0.9760	0.9111	2.1692
2.0037×10^{-4}	0.1466	0.0154	0.4311	0.4006	1.0000	0.9208	2.2528
2.3493×10^{-4}	0.1552	0.0190	0.4480	0.4199	1.0241	0.9303	2.3508
2.7304×10^{-4}	0.1635	0.0207	0.4549	0.4405	1.0477	0.9401	2.4741
3.1458×10^{-4}	0.1714	0.0232	0.4648	0.4605	1.0709	0.9498	2.6243
3.5956×10^{-4}	0.1792	0.0242	0.4691	0.4797	1.0896	0.9553	2.7333
4.4010×10^{-4}	0.1913	0.0262	0.4767	0.4998	1.1157	0.9605	2.8567
5.2871×10^{-4}	0.2024	0.0278	0.4812	0.5203	1.1421	0.9657	3.0054
6.2318×10^{-4}	0.2126	0.0307	0.4906	0.5396	1.1668	0.9706	3.1799
7.2131×10^{-4}	0.2218	0.0397	0.5130	0.5601	1.1937	0.9757	3.4131
8.1740×10^{-4}	0.2297	0.0498	0.5365	0.5801	1.2210	0.9801	3.7010
9.2400×10^{-4}	0.2376	0.0607	0.5581	0.6007	1.2499	0.9848	4.1285
1.0350×10^{-3}	0.2449	0.0808	0.5932	0.6207	1.2827	0.9894	4.8213
1.1306×10^{-3}	0.2506	0.1001	0.6206	0.6409	1.3111	0.9936	6.0318

[a] Standard reduced adsorption obtained using the amount adsorbed, 1.2815 cm^3 STP g^{-1} at p/p_0 of 0.4006; the specific surface area of α-Al_2O_3-R = 3.56 m^2 g^{-1} and the monolayer capacity = 0.8172 cm^3 STP g^{-1} obtained by the BET equation in the relative pressure range of 0.05–0.20

adsorption versus relative pressure. This adsorption isotherm is Type II, typical for nonporous and macroporous solids with inflection point at low relative pressures corresponding to the completion of monolayer (Sing et al. 1985; Kruk and Jaroniec 2001). The specific surface area of this Al_2O_3-R sample was estimated to be 3.56 m^2 g^{-1} with monolayer capacity of 0.8172 cm^3 STP g^{-1} according to the BET equation in the relative pressure range of 0.05–0.20 (Kruk and Jaroniec 2001). The calculated *t*-curve for the reference α-Al_2O_3-R is shown in Fig. 2a, b for high and low relative pressure ranges, respectively. This *t*-curve is plotted in comparison to the t-curves reported for alumina (Matejova et al. 2008), here labeled α-Al_2O_3-S, and LiChrospher 1000 silica (Jaroniec et al. 1999). It is shown that the t-curves for all three reference materials are in a very good agreement demonstrating that nonporous silica and α-Al_2O_3 materials have similar adsorption properties with respect to nitrogen (Kruk and Jaroniec 2001). Also, inset in Fig. 2b shows a good agreement between the t-curves obtained by using the multiplication factors based on the BET monolayer capacity of nitrogen on the two alumina references. While this agreement is very good at low and moderate relative pressures, some deviation can be observed within the multilayer range (not shown) with respect to the silica reference; this is probably due to some differences in macroporosity of the samples and inaccuracy in determining the exact monolayer capacity for different solids by the BET method.

N_2 adsorption isotherm recorded on the OMA material (Fig. 3a) is type IV with H-1 hysteresis loop, which is observed for cylindrical mesopores (Sing et al. 1985; Kruk and Jaroniec 2001). The steep capillary condensation step indicates high uniformity of these mesopores. This sample had the BET specific surface area of 394 m^2 g^{-1} and showed large maximum N_2 uptake corresponding to the total pore volume of 0.90 cm^3 g^{-1}. The MA material (Fig. 3a) showed much lower total pore volume, 0.43 cm^3 g^{-1} as compared to that of OMA and slightly lower surface area, 351 m^2 g^{-1}.

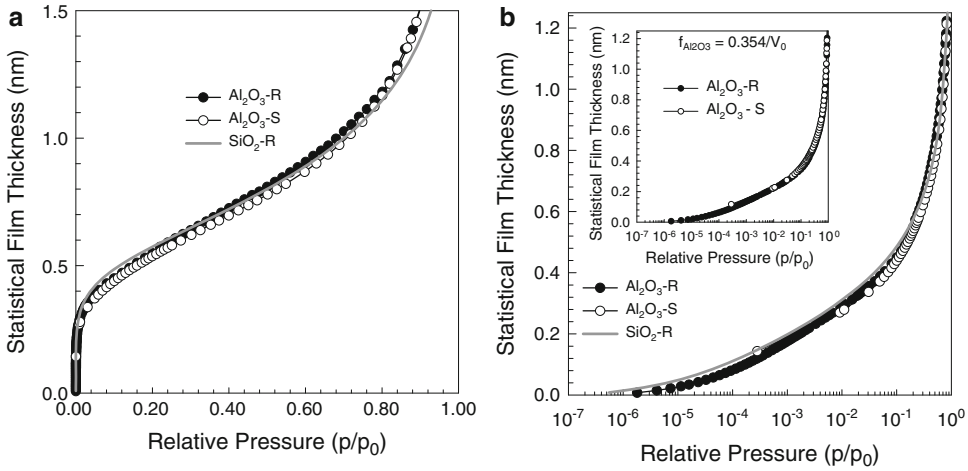

Fig. 2 The *t*-curves for SiO_2-R, Al_2O_3-R and Al_2O_3-S reference solids in the multilayer range (**a**) and at low relative pressures (**b**). *Inset* in (**b**) shows the *t*-curves obtained with conversion factor calculated by using the thickness of monomolecular layer of nitrogen (0.354 nm) and the BET monolayer adsorption capacity (V_0)

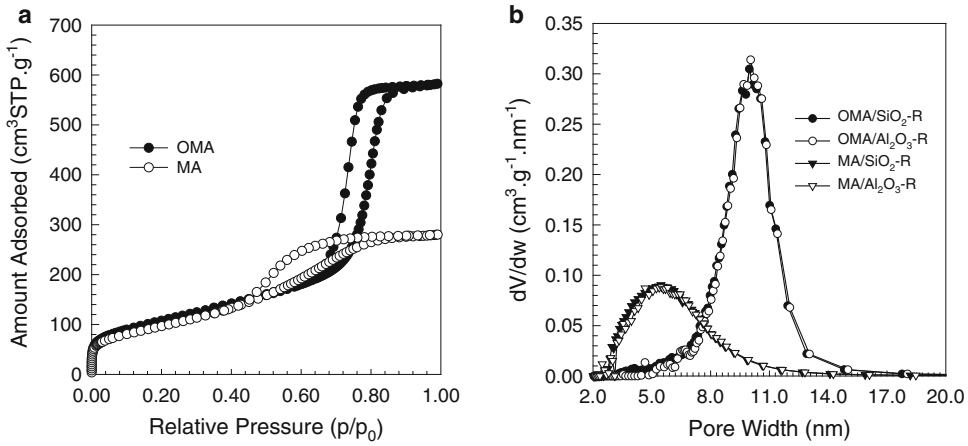

Fig. 3 Nitrogen adsorption isotherms at −196 °C for the OMA and MA materials calcined at 400 °C (**a**) and the corresponding pore size distributions (PSDs) calculated by using SiO_2-R and Al_2O_3-R as reference solids (**b**)

Furthermore, adsorption isotherm recorded on MA exhibited also a broad H-2 hysteresis loop characteristic of materials with constricted mesopores (Sing et al. 1985; Kruk and Jaroniec 2001), which are formed between agglomerates of small alumina nanoparticles in a disordered way.

The pore size distribution (PSD) curves for the OMA and MA materials were obtained from nitrogen adsorption isotherms by using the KJS method (Kruk et al. 1997b) calibrated for pores up to 19 nm (Fig. 3b). These curves were calculated using the nitrogen statistical film thickness derived for both SiO_2-R and Al_2O_3-R reference isotherms. The results obtained for both standard materials are nearly identical for the entire range of mesopores. For instance, both PSD curves for the OMA material show a maximum at the mesopore width of $\sim$10.0 nm and the pore volume of 0.94 $cm^3\ g^{-1}$ in the range of 2–50 nm. In the case of the MA sample, the mesopore width at the maximum of PSD is $\sim$5.5 nm and the corresponding total pore volume 0.45 $cm^3\ g^{-1}$. The latter pore volumes are also in a very good agreement with the single-point pore volumes obtained directly from nitrogen adsorption isotherms for both samples. This good agreement was obtained for both types of alumina materials, which differ in the uniformity of mesopores and crystallinity of the alumina framework.

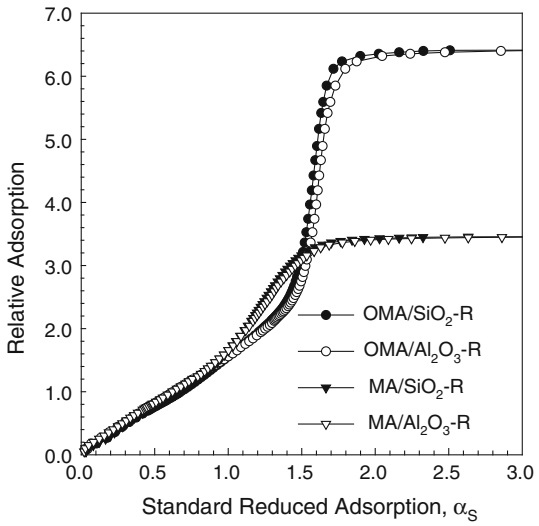

Fig. 4 The α-plots for OMA and MA materials obtained by using SiO_2-R and Al_2O_3-R reference solids

The α-plots for the OMA and MA samples are shown in Fig. 4; these plots were obtained by using both nitrogen adsorption isotherms measured on both SiO_2-R and Al_2O_3-R reference solids. The relative adsorption was calculated by dividing the amount adsorbed at a given relative pressure by the BET monolayer capacity of each sample. As in the case of PSDs, the α-plot curves coincide very well. In addition, both plots are almost linear towards the origin, indicating the lack of micropores in the samples studied (Jaroniec and Kaneko 1997; Kruk and Jaroniec 2001).

4 Conclusions

In summary, nonporous silica and α-alumina reference materials were compared for the characterization of mesoporous alumina materials. Only very small differences are observed in nitrogen adsorption isotherms at the low and high relative pressures indicating that both reference solids can be used for characterization of silica and alumina-based porous materials. Amorphous SiO_2-R has Si atoms in tetrahedral coordination, linked by O atoms forming siloxane bridges (Si–O–Si), in addition to the surface silanols (Si–OH) (Blitz and Augustine 1994). The α-Al_2O_3 has crystalline structure with Al^{3+} occupying octahedral positions in the lattice, with each Al^{3+} linked to others and surface terminated by oxo-bridges Al–O–Al. However, in addition to the oxo-bridges, α-Al_2O_3 has some surface terminated hydroxyl groups (Hass et al. 1998) that makes its interaction with nitrogen molecules similar to those with silica. Thus, both LiChrospher 1000 and α-Al_2O_3 can be used as reference solids for characterization of ordered and disordered porous aluminas with amorphous or nanocrystalline pore walls.

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