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A comprehensive approach to investigate the structural and surface properties of activated carbons and related Pd-based catalysts†

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Activated carbons are widely used as supports for industrial catalysts based on metal nanoparticles. The catalytic performance of carbon-supported catalysts is strongly influenced by the carbon activation method. Notwithstanding this important role, the effect induced by different activation methods has been rarely investigated in detail. This work deals with two carbons of wood origin, activated either by steam or by phosphoric acid, and the corresponding catalysts based on supported Pd nanoparticles. We demonstrate that the catalysts perform in a different way in hydrogenation reactions depending on the nature of the carbon used as a support, being the palladium dispersion the same. We propose a multi-technique approach to fully characterize both carbons and catalysts at the micro- and nanoscale. In particular, we investigate how the activation procedure influences the texture (by N₂ physisorption), the morphology (by Scanning Electron Microscopy), the structure (by Solid State Nuclear Magnetic Resonance, Raman spectroscopy and X-ray Diffraction) and the surface properties (by X-ray Photoelectron Spectroscopy, Diffuse Reflectance Infrared Spectroscopy and Inelastic Neutron Scattering) of carbons and of the related catalysts. The comprehensive characterization approach proposed in this work allows the rationalization, at least in part, of the role of activated carbons in enhancing the performance of a hydrogenation catalyst.

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1. Introduction

Activated carbons are important modifications of carbon that find a wide use in the field of catalysis, where they are usually employed as supports for noble metal nanoparticles. They consist of carbonized bio-polymeric materials activated in a second step of the synthesis process.^{1–3} A large number of patents describe numerous ways to activate carbon from sev-

eral natural sources, such as wood, peat or coconut shells.^{1–7} Activation is usually performed in the presence of steam or by adding phosphoric acid to the raw product, resulting in activated carbons having different properties in terms of porosity, structure at a micro- and nanoscale, and surface chemistry. All these three factors play a fundamental role in catalysis. The support may have a direct influence on the catalytic reaction because its surface is often active toward reactants and reaction products.⁸ In addition, the support may exhibit an indirect influence because its physical-chemical properties may affect the properties of the deposited metal nanoparticles (such as shape, size distribution and dispersion), their tendency to aggregate under catalytic conditions (*i.e.* resistance to sintering), and the accessibility of active sites to reactants.^{1–3,9–13}

Pore size and pore volume are important factors for physical adsorption. Many works in the literature report detailed analysis of the porosity of carbons activated following different routes.^{5,7,14} It is now well established that in all cases a high specific surface area is obtained (up to 1500 m² g⁻¹) due to the oxidative generation of micropores of variable size and shape distribution. In the presence of phosphoric acid, a fraction of the additive is incorporated during carbonization into the carbon body and is subsequently removed by

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† Electronic supplementary information (ESI) available: The chemical composition of the two carbons determined by EDX analysis, ¹H CPMAS NMR spectra of the two carbons. See DOI: 10.1039/c6cy00159a

