High-purity crystalline silicon gels

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In the present investigation, silica gels have been synthesized via sol-gel method under microwave radiation. For that, precursor solutions were prepared using tetraethylorthosilicate (TEOS) as the silica precursor, varying the molar ratios of water and ethanol to it. HCl was added before heating to perform acid gelation, while NH_3 (2 M) was added after gelation to promote polycondensation and Ostwald ripening reactions during aging. The use of microwave radiation under these conditions resulted in a favourable effect on the final structure of the polymeric network [1]. This approach makes it possible to obtain mesoporous silica gels in a short time, but amorphous in all cases (Fig. 1). The XRD pattern displayed the presence of a broad peak at 2θ = 17–29° that corresponds to the formation of amorphous silica according to JCPDS-card 96-900-1582.

The magnesio-thermal reduction process has already been reported as a useful way to convert silica into silicon in the presence of magnesium as a reduction agent [2,3]. Thus, our amorphous silica gels were mixed with Mg in a weight ratio of 1:1 and treated at 750 °C for 12 h under an inert atmosphere (Ar, 300 mL/min). Many phases can be produced from the reduction process of SiO_2 and Mg, such as MgO and Mg_2Si. Thus, the reduced samples were subsequently washed with HCl (1 M) for 4 hours to eliminate the undesired secondary phases. The structural properties of the obtained silicon gels were analysed and measured by X-ray diffraction (XRD), X-ray fluorescence (XRF) and high-resolution transmission electron microscopy (HR-TEM). Fig. 1 shows the XRD data of the final reduced silicon gel, illustrating the complete removal of SiO_2, with only Si peaks remaining in the structure. The major diffraction peaks at 2θ = 28.4°, 47.4° and 56.2° are presented at (111), (202) and (131) planes, respectively, which can be attributed to high-purity silicon gel according to JCPDS-card 96-901-3109. Also, the absence of additional peaks indicates that no impurities are present in the structure.

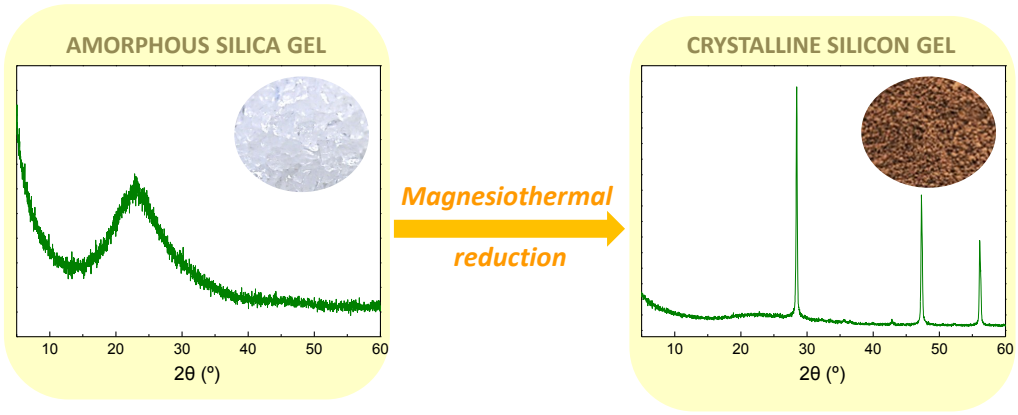

Figure 1. XRD data of a) silica gel before treatment and b) silicon gels after magnesio-thermal reduction.

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