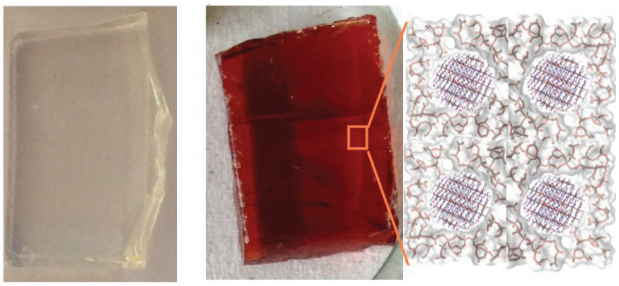

Figure 1. Photographs of transparent empty silica monolith (left) and SNP-silica nanocomposite (right) and schematic representation of encapsulated nanoparticles.

Keywords: total scattering, NMR, nanocrystalline material, molecular compounds

MS23-P15 Tailoring phase composition and microstructural features of $Ba_4Nb_2O_9$ polymorphs *via* thermal decomposition route

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Synthesis of nanoparticles with a controlled size has been found to be crucial for tailoring desired material properties since thermal, electric, optical, catalytic, and magnetic properties are strongly composition-, structure-, but also, size- and shape-dependent.[1,2] It is well known that $Ba_4Nb_2O_9$ exhibits mixed electronic, oxide ion, and proton conductivity, which makes it especially attractive in the field of fuel cells, steam electrolysers, and humidity sensors.[3] Especially, the γ-$Ba_4Nb_2O_9$ phase exhibits several orders higher conductivity than α-$Ba_4Nb_2O_9$ due to a faster protonic and oxide ionic transport.[3,4] Up to now, the γ polymorph was metastable at room temperature (RT) and so could be isolated only by quenching the sample from high temperatures, because slow cooling led to a transformation to α phase.[3] In the present study the mixed Ba^{II}–Nb^{V} containing oxides were prepared using $\{Ba_2(H_2O)_5[NbO(C_2O_4)_3]HC_2O_4\}\cdot H_2O$ as a single-molecular precursor.[5] A systematic study of preparation conditions was carried out, namely, tuning the final structural and microstructural parameters by (*i*) the holding time (2, 1 and 0.5 h) at 1175 °C and (*ii*) the cooling rate (3, 7 and 12 °C min^{-1}) from 1175 °C back to RT. Nanocrystalline products obtained by thermal decomposition of the precursor were investigated by X-ray powder diffraction at RT. Size and strain analysis were obtained in the course of the Rietveld refinement. Since all samples prepared by a cooling rate of 12 °C min^{-1} contained only the γ-$Ba_4Nb_2O_9$ polymorph, it was evident that faster cooling prevented the $\gamma \rightarrow \alpha$ phase transition, which resulted in the retention of the high-temperature γ-$Ba_4Nb_2O_9$ phase at RT. Shortening of the time period for which γ-$Ba_4Nb_2O_9$ containing samples were held at 1175 °C had no impact on their phase composition. All samples were found to be strain free. Crystallite sizes were ~20 nm for γ-$Ba_4Nb_2O_9$ prepared with a holding time of 2 h while it decreased to ~5 nm with shortening of the holding time to 0.5 h.

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Keywords: phase composition, size and strain analysis, Rietveld refinement