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^{1}H and ^{31}P NMR spectroscopy of dynamic structures in nano-and mesostructured hydroxyapatites

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The ^{1}H spin-lattice and spin-spin relaxation time as well as high data point density ^{1}H–^{31}P cross-polarization kinetics (static and upon MAS) measurements have been carried out for calcium hydroxyapatite containing amorphous phosphate phase (ACP-CaHA) and nano-structured hydroxyapatite (CaHA). The chosen setting of the sampling frequency of 5×10^{4} s^{-1} allowed to reveal all spin interactions having the dipolar splitting $b\leq25$ kHz that in the case of ^{1}H–^{31}P interaction means the structures with the inter-nuclear distances $r\geq0.125$ nm could be resolved. The advanced processing of CP MAS kinetic data has been developed introducing the variable cut-off distribution of the dipolar coupling. This procedure allows to describe the oscillatory kinetics and CP curves in nano-structured materials over a wide range of contact time and to determine the characteristic size profile and composition of the spin clusters. The characteristic size of ^{31}P–$(^{1}H)_{n}$ spin nano-cluster being within 0.3÷0.5 nm has been determined for nano-structured CaHA. The ^{1}H spin-lattice and spin-spin relaxation time measurements revealed the fast spin motion takes place in ACP-CaHA. The effect of MAS rate on the ^{31}P signal shape confirms that the correlation time of this motion gets into the time scale of microseconds or even nanoseconds. Such fast dynamics can be attributed to the rotational diffusion of adsorbed water molecules. The magnitude of the inhomogeneous anisotropic broadening of 1220±20 Hz determined for nano-structured sample is very close to 1185 Hz that corresponds to the maximum of Gauss distribution of dipolar ^{1}H–^{31}P coupling obtained using ^{31}P–^{1}H CP MAS kinetics. The dynamics of this interactions runs in the time scale of microseconds and it is much slower that in ACP-CaHA.

Biography

Vytautas Balevicius is a Physicist, Doctor of physical sciences and Professor. Currently he runs the Vilnius University Faculty of Physics dean's office.

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