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Title:Development of dnp-enhanced high-resolution solid-state nmr system for the characterization of the surface structure of polymer materials
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Abstract:A dynamic nuclear polarization (DNP)-enhanced cross-polarization/magic- angle spinning (DNP/CP/MAS) NMR system has been developed by combining a 200 MHz Chemagnetics CMX-200 spectrometer operating at 4.7 T with a high-power 131.5 GHz Gyrotron FU CW IV. The 30 W sub-THz wave generated in a long pulse TE o1+ 41 mode with a frequency of 5 Hz was successfully transmitted to the modified Doty Scientific lowtemperature CP/MAS probe through copper smooth-wall circular waveguides. Since serious RF noises on NMR signals by arcing in the electric circuit of the probe and undesired sample heating were induced by the continuous sub-THz wave pulse irradiation with higher powers, the on-off sub-THz wave pulse irradiation synchronized with the NMR detection was developed and the appropriate setting of the irradiation time and the cooling time corresponding to the non-irradiation time was found to be very effective for the suppression of the arcing and the sample heating. The attainable maximum DNP enhancement was more than 30 folds for C1 13C-enriched D-glucose dissolved in the frozen medium containing mono-radical 4-amino-TEMPO. The first DNP/CP/MAS 13C NMR spectra of poly(methyl methacrylate) (PMMA) sub-micron particles were obtained at the dispersed state in the same frozen medium, indicating that DNP-enhanced 1H spins effectively diffuse from the medium to the PMMA particles through their surface and are detected as high-resolution 13C spectra in the surficial region to which the 1H spins reach. On the basis of these results, the possibility of the DNP/CP/MAS NMR characterization of the surface structure of nanomaterials including polymer materials was discussed. © Springer Science+Business Media, LLC 2012.
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Controlled terms:Esters - Glucose - Gyrotrons - Irradiation - Nuclear magnetic resonance spectroscopy - Polymers - Probes - Surface structure
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