Characterization of Native and Plasticized Starch Using Solid State NMR

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Abstract. The crystallinity of native starches influences numerous properties of starch-based materials and for this reason, its assessment is of great importance. It is known that the phase composition of native starch can be obtained by the decomposition of its ^{13}C NMR spectrum into the sub-spectra of the ordered (crystalline and interfacial) and amorphous phases. The percentage of amorphous phase can be obtained from the intensity of C_4 carbon resonance. The deconvolution of the C_1 carbon resonance in the sub-spectrum of the ordered phase gives information on the ratio of crystalline and interfacial phases and the starch crystalline type, which is inferred from the presence of triplet (A-type) or doublet (B-type) of even intensities. Four native starches - soltex (wheat, A-type), amylex (potato, B-type) meritena and maisita (both corn A-type) were studied and as their amorphous counterparts the starches plasticized with urea were used. The ^{13}C NMR spectra of the studied samples were recorded using the cross polarization (CP) and the magic angle spinning (MAS) techniques and using the decomposition of the spectra the phase compositions of native starches were obtained. In order to evaluate the mobility of different phases in native as well as plasticized samples the measurements of carbon spin-lattice relaxation times $T_1(^{13}C)$ in laboratory frame were carried out. The $T_1(^{13}C)$ values obtained fitting the bi-exponential function to the C_1 carbon intensity decay and taking into account the calculated composition of the samples (ordered and disordered phases) show that A-type starches have close $T_1(^{13}C)$ values which are shorter (16.6 – 20.8 s) for disordered phase and longer (53.9 – 80.1 s) for ordered phase proving different mobility of these phases. The B-type sample has distinctly lower $T_1(^{13}C)$ values (1.7 s, 37.0 s) which are in line with less spatial constraints in its structure. The mono-exponential functions fitted to the data obtained for plasticized samples provided $T_1(^{13}C)$ values in the interval (43.9–79.9 s) which could be explained by different molecular structure of native samples.