CHALLENGES IN PRECISE MICROSTRUCTURAL CHARACTERIZATION OF WOOD-DERIVATIVE POLYMERS

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Cellulose ethers derived from pulp paper cellulose as raw material are non-toxic white powders, odorless, and tasteless that swell into a clear or slightly turbid colloidal solution. They present great interest for food, pharmaceutical or building industries as a food additive, emulsifier, thickening or suspending agent, excipient, plastering mortars, panel joint fillers, tile adhesives or water-based coatings. Important molecular variables that control efficiency of cellulose ethers are the nature of the monomers and monomer linkers, monomer sequence distribution along chains, average molecular weight and molecular weight distribution and molecular architecture. Due to the broad source of cellulose in nature, their properties are highly impacted by the molecular structure. The most impacting microstructural parameter of is the density of grafting groups on polymer chains, namely the degrees of substitution of the polymer chains. The complexity of cellulose ethers structure and resulting properties is due to the presence of two grafting groups, the methoxy group (OCH_3) and the hydroxy-propyl group (OC_3H_6OH). The goal of this work is to present an original way to characterize the structure of hydroxypropyl methylcellulose (HPMC) class of cellulose ethers on the basis of ^{13}C Nuclear Magnetic Resonance (NMR). In order to propose a precise characterization, preliminary analyses were done using model polymers intervening in the chemical process of HPMC synthesis, like cellulose, methyl cellulose and hydroxypropyl cellulose. These three polymers have an increasing complexity in term of lateral substituents. These substitutions are described by the degree of substitution (DS), i.e. the number of methoxy groups attached on a glycosidic unit, and the molar substitution (MS) i.e. the number of moles of hydroxypropyl group per mole of anhydroglucose in the chain. On the basis of ^{13}C NMR associated to Cross-Polarization Magic Angle Spinning (CP-MAS) technique, a quantitative determination of the degrees of substitution for anhydroglucose in positions 2, 3 and 6 as well as total substitution and molar substitution is achieved. Moreover the weight percent of substituents and reactivities of the substituted carbons are also determined, indicating that C2 and C6 are the most reactive positions.