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Experimental evidence of molecular relaxation processes in cellulose-amine physical complexes.

Cellulose complexes with a series of amines of various sizes and chemical structures were studied in this work. These complexes were obtained by putting microfibrillated cellulose films or Flax fibers in contact with a solution of ethylenediamine (EDA) and a target mono- or diamine under an inert atmosphere while raising the temperature. Excess of EDA could be eliminated under vacuum overnight while maintaining the complex with larger amines. Wide Angle X-ray Scattering (WAXS) characterization showed that amine intercalated in cellulose III-like structure with the d(010) spacing linearly increasing with the molar volume of the amine. Modulated Differential Scanning Calorimetry (MDSC), Dynamic Mechanical Analysis (DMA), and Broadband Dielectric Spectroscopy measurements showed the presence of two glass transition-like processes (Tg) in all studied complexes. The first transition occurred at ca. 0℃ and its value is independent from the amine size. The second transition appeared at between 80 and 110℃ and depends on the size of the amine, i.e. when the amine molecular size increases, the Tg decreases. Solid-State 13C NMR CP-MAS and 2D WISE measurements at increasing temperatures showed that, when approaching the second Tg, a fraction of the amines' peaks became closer to the chemical displacements at liquid state. The fraction of such amine increased with temperature. Moreover NMR T1 relaxation time measurements showed that the complexed amines possessed two to three relaxation domains and their corresponding relaxation times became shorter with increasing temperature. With these results we have attributed the first Tg to molecular motions of the complexed amines within the cellulose structure while the second high-temperature Tg would correspond to the molecular relaxation of the cellulose-amine complexes. Mechanical characterization of complexed films at various temperatures will be carried out to relate the complexes' mechanical properties with their molecular mobility.

Preferred topic

Biopolymers

Primary authors: RIOS DE ANDA, Agustin (CERMAV & ICMPE); HEUX, Laurent (CERMAV); VERGELATI, Caroll (Solvay); MAZEAU, Karim (CERMAV); NISHIYAMA, Yoshiharu (CERMAV-CNRS)

Presenter: NISHIYAMA, Yoshiharu (CERMAV-CNRS)