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Chiral Self-assembly Behavior of Carboxylated Cellulose Nanocrystals Obtained by Ammonium Persulfate (APS) Oxidation

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Abstract

Although the self-assembly of cellulose nanocrystals (CNCs) derived from sulfuric acid hydrolysis has been extensively studied, that of ammonium persulfate (APS)-oxidized CNCs remains comparatively less explored. In this context, carboxylated CNCs were isolated by APS oxidation from filter paper (FP) and molded pulp (MP) to investigate their self-assembly behavior and chiral nematic properties. In this approach, the fibers were directly converted into CNCs without any prior pretreatment. APS oxidation was performed at 70 °C for 16 h under two oxidant concentrations (1 and 1.5 M). A combination of characterization techniques, including conductometric titration, zeta potential, dynamic light scattering (DLS), polarized optical microscopy (POM), atomic force microscopy (AFM), attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR), and X-ray diffraction (XRD) was employed to evaluate surface charge, size distribution, carboxyl content, chiral ordering, morphology, chemical structure, and crystallinity. Carboxylated CNC films were fabricated via an evaporation-induced self-assembly (EISA) process [1]. The preserved chiral nematic ordering of the CNC films was confirmed by POM and field-emission scanning electron microscope (FE-SEM) observations. Clear differences between FP- and MP-derived CNCs were observed in both suspension and film states. Birefringence was detected only in FP-CNC suspensions above the critical concentration, while fingerprint textures were observed exclusively in FP-CNC systems. Structural coloration was presented only in films derived from FP-CNCs, whereas no such effect was observed in MP-derived counterparts. Furthermore, FP-derived films exhibited a pronounced blue shift in color as the APS concentration increased from 1 M to 1.5 M. Cross-sectional SEM images revealed a periodically ordered multilayered structure in FP-CNC films. Chiral CNC films emerge as versatile platforms for advanced photonic, sensing, anti-counterfeiting, and sustainable coating applications, enabled by their tunable chiral nematic architecture and responsive structural coloration [2].

Keywords: Chiral self-assembly, Carboxylated CNCs, Ammonium persulfate oxidation, Birefringence

References

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