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ORIGINAL PAPER

Synthesis of cellulose triacetate-I from microfibrillated date seeds cellulose (*Phoenix dactylifera L.*)

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Abstract Cellulose triacetate (CTA) has successfully been synthesized from microfibrillated date seeds cellulose. The cellulosic material under study constituted 84.9% amorphous phase with a degree of polymerization of 950. Acetylation was conducted at 50 °C under optimized heterogeneous conditions by acetic anhydride as acetyl donor, acetic acid as solvent and sulfuric acid as catalyst. In this process, cellulose was acetylated without dissolving the material throughout. The acetylated cellulose chains on the surface were dissolved gradually in acetic acid, which created new accessible zones. The yield of cellulose triacetate was studied varying acetic acid, acetic anhydride and catalyst concentrations, as well as reaction times. The ratio between the intensity of the acetyl C=O stretching band at around 1740 cm^{-1} and the intensity of C–O stretching vibration of the cellulose backbone at 1020–1040 cm^{-1} was used to optimize the reaction time. The optimal reaction conditions of 8% concentration of sulfuric acid, acetic anhydride/cellulose weight ratio of 3:1, acetic acid/cellulose weight ratio of 7:1, reaction time of 3 h and reaction temperature of 50 °C have given highest yield of cellulose triacetate, of about 79%. The obtained date seeds-based cellulose triacetate was characterized thoroughly by Fourier transform infrared (FTIR), X-ray diffraction (XRD), nuclear magnetic resonance spectroscopy (NMR), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The synthesized product was identified as cellulose triacetate-I (CTA-I) characterized by a melting temperature of 217 °C and a decomposition temperature of 372 °C. These results demonstrated that date seeds can be used as potential source of microfibrillated cellulose which can be easily functionalized.

Keywords Date seeds cellulose · Cellulose triacetate · Optimization · Crystalline allomorph-I · Degree of acetyl substitution

Introduction

After chemical modifications, microfibrillated cellulose can provide several biodegradable cellulose derivatives, including carboxymethyl cellulose (CMC), hydroxypropyl cellulose (HPC), hydroxyethyl cellulose (HEC) and cellulose acetates [1]. Cellulose triacetate (CTA) having an average degree of substitution (DS) above 2.8 is a triacetic ester of cellulose used in industrial, chemical and pharmaceutical fields. For example, CTA is used to elaborate high-performance membranes characterized by well-defined pores [2–4] and it has been recognized as a powerful chiral polymeric sorbent for chromatographic separation of enantiomers [5]. Cellulose triacetate (CTA) has two crystalline allomorphs CTA-I and CTA-II in report with the cellobiose chain polarity. CTA-I is produced only by heterogeneous acetylation from cellulose I, while CTA-II is obtained from heterogeneous acetylation of cellulose II and homogeneous acetylation of cellulose followed by the crystallization of the formed CTA [6]. The crystal structures of CTA-I and CTA-II have been investigated by X-ray diffraction (XRD), nuclear magnetic resonance (NMR) and Fourier transform infrared spectroscopy (FTIR) techniques. Computational

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