# MORPHOLOGY OF CELLULOSE ACETATE/FUNCTIONALIZED CELLULOSE NANOCRYSTAL NANOCOMPOSITES OBTAINED BY MELT EXTRUSION

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#### **Abstract**

The aim of this study was to investigate the morphology of cellulose acetate nanocomposites containing cellulose nanocrystals, functionalized by acetic acid esterification. Field emission scanning electron microscopy images showed uniform distribution of the nanocrystals, which were visualized as white dots in the fracture sample surface. The results indicated the promising use of cellulose nanocrystals as a cellulose acetate reinforcement to improve the mechanical properties.

Keywords: Functionalized cellulose nanocrystals, Cellulose acetate, Nanocomposites.

### INTRODUCTION

Cellulose nanocrystals (CNC) have attracted great interest in material science due to their interesting characteristics including low cost, renewability, biodegradability and availability. However, one restriction towards the use of CNC as a reinforcement is the poor compatibility between the hydrophilic CNC and the hydrophobic polymer matrices<sup>1</sup>. In addition, the strong inter-particle hydrogen bonding and high surface area cause nanocrystal aggregation. Surface modification, such as acetylation, is an effective way to overcome this drawback<sup>2</sup>. Among the different methods of nanocomposite preparation, the melt extrusion process presents advantages in terms of industrial application, due to its high productivity and non solvent use<sup>3</sup>. In the present work, cellulose nanocrystals were isolated and functionalized by a single-step procedure using a hydrochloric and acetic acid mixture. The CA/CNC nanocomposite was prepared by extrusion technique and the nanocomposite morphology was investigated.

#### **METHODS**

Isolation and surface functionalization of cellulose nanocrystals were simultaneously carried out using cotton fibers by a single-step method. The reaction occurred in a combined acid system composed of 90 wt% acetic acid and 0.027 M hydrochloric acid aqueous solution. The reaction was performed under constant stirring at 105 °C for 9h. The resulting product was cooled down, centrifuged, dialyzed and lyophilized to obtain CNC. The nanocomposites were prepared by mixing CA with 30 wt% triethyl citrate (TEC) and different CNC contents in a twin-screw Micro compounder – DSM Xplore<sup>™</sup>. Plasticized CA, without the addition of CNC, was also prepared according to the above mentioned process. Nanocrystals and nanocomposite morphologies were examined in a JEOL JSM-6340 field emission scanning electron microscope (FESEM), operating at an accelerating voltage of 5.0 kV. Samples were prepared by dropping a dispersion of CNC in isopropanol on top of a mica sheet mounted on a sample holder. The nanocomposites were cryofractured in liquid nitrogen for cross section examination. All the samples were platinum sputter coated in a Bal-Tec MD 020 instrument (Balzers).

## **RESULTS AND DISCUSSION**

FESEM micrographs of the functionalized cellulose nanocrystals, isolated from cotton fiber, and CA/CNC nanocomposites are presented in Figure 1. The image in Figure 1a confirms that the hydrolysis conditions were able to efficiently break the interfibrilar links of the cotton fiber. CNC showed approximately 30 nm diameter and

300 nm average length needle-like structures. Figures 1b and 1c show the fracture surfaces of CA and CA/CNC 1.5 wt% nanocomposite, respectively. Compared to the smooth fractured surface of CA, white dots are observed in the CA/CNC nanocomposite cross section, which might be assigned to the CNC aggregates. The nanocomposite fracture morphology showed good dispersion and uniform distribution of CNC in the CA matrix, which is evidence of strong hydrogen bonding between CNC and CA acetate groups.

### **ACKNOWLEGMENT**

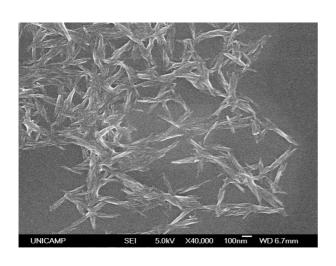
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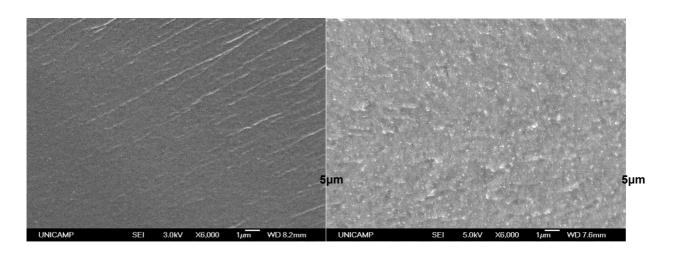


Figure 1. FESEM micrographs of (a) functionalized cellulose nanocrystals and fracture morphologies of (b) CA and (c) CA/CNC 1.5 wt% nanocomposite.