

Transmission electron microscopy of cellulose nanocrystals

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Cellulose nanocrystals (CNC) have recently been the focus of extensive studies due to their unique properties. CNC dimensions and surface properties are key parameters influencing the nanoparticle performance in different applications. Examples include estimation of hydroxyl number available on the CNC surface for optimizing the modification conditions, and determination of their aspect ratio (ratio of the length to the diameter) to calculate the mechanical percolation threshold for CNC nanocomposites. Transmission electron microscopy (TEM) is the mostly used technique for CNC morphological and dimensional characterization. However, the characteristic CNC low contrast in the TEM analysis gives rise to images in which the edge detection of the nanoparticles is difficult. The aim of this work was to develop a sample preparation method protocol for CNC analysis by TEM. Therefore, the CNC suspension concentration, glow discharging, sample volume on the grid, drying and staining (2 wt% aqueous uranyl acetate solution) methods were chosen as the most significant parameters to be evaluated. CNC micrographs were obtained in a JEM 1400 plus transmission electron microscope (JEOL) operating at an 120 kV accelerating voltage. TEM images of the samples, under different preparation conditions, were analyzed using ImageJ software and the mean values of length, width and aspect ratio of the nanoparticles were compared. It was possible to conclude that the CNC observation and analysis were improved by optimization of suspension dilution and application of glow discharging. Statistical analysis of the CNC measurements showed that the developed protocol is suitable for CNC dimensional measurements with high accuracy. As an example, Figure 1 shows a typical CNC TEM micrograph using the most commonly reported sample preparation protocol, while Figure 2 shows a CNC TEM micrograph obtained using the protocol developed herein.

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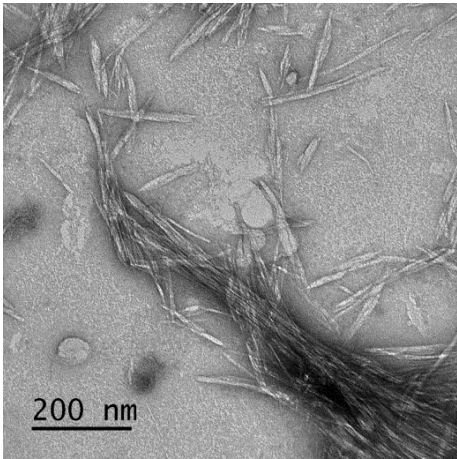


Figure 1. Bright-field TEM micrograph of a commercially available CNC sample prepared by a typical procedure.

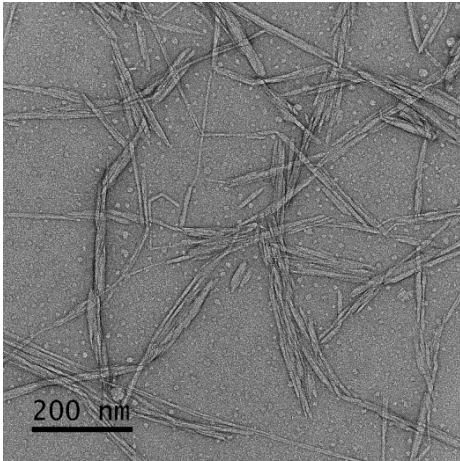


Figure 4. Bright-field TEM micrograph of a commercially available CNC sample prepared using the protocol developed herein.