Cellulose nanocrystals (CNC) can be obtained by submitting native fibers to acid hydrolysis. These nanostructures can be used to enhance polymer nanocomposite properties due to their high crystallinity and aspect ratio (1-4). However, as a result of the hydrophilic character of CNC, several studies have been performed to promote the CNC surface modification aiming at the improvement of compatibility with hydrophobic polymers. For this purpose, silane coupling agents are widely used (5). Thus, the aim of this work was to characterize the CNC surface modification, which was carried out by using a silane which has isocyanate groups. CNC were obtained by cotton acid hydrolysis, which was carried out by treatment for 2h in a 10 wt% alkali solution, followed by hydrolyzation in a 4M hydrochloric acid at 80 °C for 3 h and 45 min under stirring. The excess acid was removed by repeated cycles of centrifugation. Afterwards, the CNC suspension was dialyzed, lyophilized, dispersed in DMF and kept under an inert atmosphere. Silane was added to the nanocrystal suspension and the reaction was maintained under stirring for 8 h. Catalyst and water were added and the mixture was kept under stirring for 30 min. Lastly, the product was dried and nanocrystal powder was obtained. The morphologies of the samples were investigated in a Carl Zeiss CEM 902 transmission electron microscope (80 kV) equipped with a Castaing-Henry energy filter spectrometer within the column and a Proscan Slow Scan CCD camera. To examine the CNC, a droplet of the diluted suspension was deposited on carbon coated parlodion film supported on a copper grid. Images were acquired using electrons with zero-loss energy and processed using the AnalySis 3.0 software. Fig.1a,b confirm that the hydrolysis conditions gave rise to needle-like structures with a 10 nm approximate diameter and a 166 nm average length. The silylation step did not cause any change in the CNC morphology. Fig. 2 presents ESI-TEM images of the modified nanocrystals and the silicon maps, before and after solvent extraction. Fig.2a,c show regions with high CNC concentration, while Fig.2b,d show the silicon mapping images in the same areas, where the bright regions correspond to silicon-rich domains. These images clearly show that there is a higher silicon concentration around the vicinity of the nanocrystals. Furthermore, a strong contrast can be observed between adjacent nanocrystals, suggesting the formation of a rather uniform polysilsesquioxane layer wrapping of the nanocrystals.

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**Fig. 1:** TEM micrographs of cotton nanocrystals: (a) before and (b) after silylation reaction.

**Fig. 2:** ESI-TEM images of the modified nanocrystals: (a) bright field and (b) silicon map, obtained before solvent extraction, (c) bright field and (d) silicon map, obtained after solvent extraction.