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Cellulose microfibrils and nanocrystals thin films deposited by drying-dewetting

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Abstract – Cellulose microfibrils and nanocrystals produced from eucalyptus cellulose wood pulp where self assembled as semi-continuous and continuous films over mica substrate by drying-deweeting of dilute suspensions of the nanosized materials. Cellulose microfibrils were produced by high power ultrasound treatment of eucalyptus wood pulp suspensions previously dispersed in water to promote water inclusions between cellulose microfibrils. Cellulose nanocrystals were produced by acid hydrolysis of eucalyptus wood pulp using sulfuric acid at 64 % (w/w) and 45 °C. Cellulose microfibrils and nanocrystals were characterized by transmissions electron microscopy (TEM) and by atomic force microscopy (AFM). The films over mica substrate were characterized by AFM.

Nanosized cellulose materials such as microfibrils and nanocrystals or whiskers have been attract great attention due to its increase application in several systems, ranging from nanocomposites to devices in wide areas of technology and science [1]. Nature usually performs the bottom-up process approach to fabricate a product, such as cellulose microfibrils, which range from 5 to 15 nm diameter. These nanofibers are extremely homogeneous and can be used in nano-technology for several applications. In this work we investigate the formation of well organized films by self-assembly nanofibers and nanocrystals of cellulose on mica substrate by drying-dewetting [2]. The film, as we will see can be transferred to other substrates to produce devices or used in nanocomposites as a “nano fiber mat”. Cellulose microfibrils were produced from cellulose eucalyptus wood pulp suspended in water by 48 hours after thermal cycle (0 – 95 °C) by ultrasound treatment in a UP400S model Hielscher ultrasound equipment operated at 24 kHz, 400 W from 5 to 15 min. Cellulose nanocrystals were produced by treating dried cellulose wood pulp with a 65 % (w/w) sulfuric acid solution cooled at 10 °C. The mixture was keep at 45 °C under vigorous stirring for 1 hour. The nanocrystals were isolated by successive centrifugation (3.600 rpm/ 30 min) until the washing water shows pH not below 6. The materials were characterized by electron transmission microscopy operated at 110- 175 kV and by atomic force microscopy in the “tapping mode”. For TEM analysis the samples were prepared on 400 mesh cupper grid after immersion in uranyl acetate. Cellulose microfibrils and nanocrystals suspensions with concentrations ranging from 0,05 to 0,1 % were deposited onto mica substrate in a single drop which was allowed to dried at 40 °C for 1 hour and analyzed by AFM. Figure 1a and 1b shows microfibrils and nanocrystals TEM images, respectively.

Cellulose microfibrils (Fig. 1a) display diameter with a very narrow variation ranging from 5 to 10 nm with several microns in length. Cellulose nanocrystals (Fig 1b) are more regular with diameter of ~15 nm and length of ~200 nm. Figure 2a and 2b shows the film of microfibrils and nanocrystals respectively over mica substrate. Figure 2 shows that smooth thin surfaces can be obtained using the drying-dewetting technique. The roughness of the prepared films taken in a small area (100 x 100 nm) is in the range 50 – 70 nm. The authors acknowledge **FAPESP** and **CNPq** for the financial support and **CNPq** for the grant to Debora P. Magalhães. We also tanks to Prof. R. M. Faria and Dra. D. T. Balogh from IFSC/USP for the collaboration.

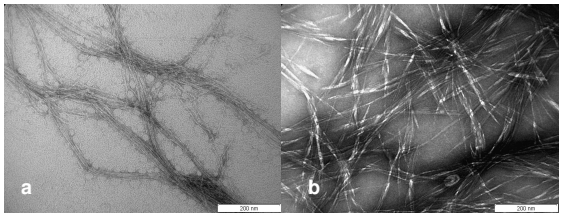

Figure 1: TEM image of a) cellulose microfibrils and b) cellulose nanocrystals.

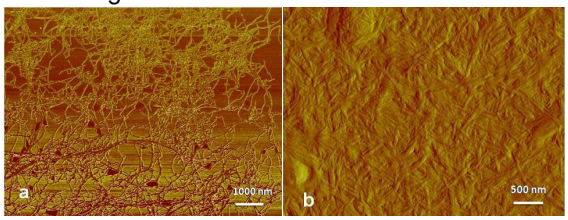

Figure 2: AFM images in phase mode from a) cellulose microfibrils and b) cellulose nanocrystals.

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