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Editors:

Caue Ribeiro
Odílio Benedito Garrido de Assis
Luiz Henrique Capparelli Mattoso
Sergio Mascarenhas

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Influence of NaOH addition to accelerate the neutralization process of the brown cotton cellulose nanofibers on their crystallinity and thermal stability

E. de M. Teixeira^{1*}, A. C. Corrêa¹, K. B. R. Teodoro^{1,2}, J. M. Marconcini¹ and L. H. C. Mattoso¹

e-mail: eliangelat@yahoo.com.br

Abstract – Suspension of brown cotton nanofiber was obtained by the acid hydrolysis and its neutralization process was accelerated by the use of NaOH (1M) solution. The effect on crystallinity and thermal stability of resulting nanofibers were investigated by X-ray diffraction (XRD) and thermogravimetry analysis (TGA) and compared to the properties of the nanofibers neutralized by conventional dialysis method. The results indicated that the use of NaOH solution to neutralize nanofibers suspensions resulted in nanofibers with similar crystallinity and more thermally stable than nanofibers neutralized by dialysis method. However there was the formation of sodium sulfate thus contaminating the nanofibers suspensions.

Cellulose nanofibers can be obtained by hydrolysis of amorphous phase of cellulose using strong acids such as sulfuric acid $(H_2SO_4)^{[1]}$. After this, the resulting nanofibers suspensions are highly acid and, in general, are subjected to the process of dialysis in water. This process requires a long time to neutralize the suspension. For example, using 5 g of fiber to hydrolysis, this period of neutralization varies from 4 to 5 days while neutralization with NaOH solutions takes only few minutes. The dialysis promotes not only the discharge of a high volume of water but also the dumping of acidic water to the environment. In this study we propose the use of a basic solution (NaOH) to accelerate the neutralization process and the influence on the crystallinity and thermal stability were investigate and compared to the nanofibers neutralized by conventional dialysis process. The nanofibers suspensions were obtained from brown cotton fibers using H_2SO_4 (6M) at $45^{\circ}C$ and 75 min. The sulfuric acid was partially removed by centrifugation at 10,000 rpm for 10 minutes and about 500 mL of water were added. This suspension was submitted to dialysis in current water or neutralized with NaOH (1M) solution. After that, the suspensions were dried at $35^{\circ}C$ for 12 h in an air-circulating oven.

The DRX analysis presented similar crystallinity for both samples (around 90%) but sodium sulfate was formed from neutralization with NaOH solution, as a show in Figure 1(B). But the use of NaOH solution presented nanofibers with better thermal stability than those dialyzed (Figure 1(C)). It caused an increase of around 80°C on thermal stability. Both dried nanofibers were resuspended in water (1% solution) resulting in stable suspension again.

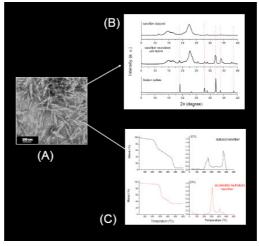


Figure 1: TEM image, (B) XRD pattern and (C) TG/DTG curves of nanofibers.

Reference

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¹ National Nanotechnology Laboratory for Agriculture (LNNA), Embrapa Agricultural Instrumentation, P. O Box 741, CEP: 13560-970, São Carlos - SP, Brazil.

² Chemical Department, Federal University of São Carlos (UFSCar), via Washington Luiz, KM 235, Monjolinho, P. O. Box: 676, CEP: 13565-905, São Carlos, SP - Brazil * Corresponding author.