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Cellulose nanocrystals from coconut fiber: Preparation and Characterization

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Abstract – Coconut fiber has been used to prepare cellulose nanocrystals (CNCs) by sulfuric acid hydrolysis under different conditions of reaction time. CNCs suspensions were characterized by transmission electron microscopy (TEM), thermogravimetric analysis (TGA) and X-Ray Diffraction. Ultrathin CNCs with diameters as low as 5 nm and aspect ratio of up to 55 were obtained. When comparing the hydrolysis degree as a function of fiber extraction time, it was found that the length of CNCs was greater for shorter extraction time. The results promote the use of coconut fibers as a novel renewable source of CNCs with potential as reinforcing agents in nanocomposites.

The extraction of nanometer-sized single crystals of cellulose, commonly referred to as whiskers or nanowhiskers, has gained more attention in recent years due to the exceptional mechanical properties, large specific surface area, high aspect ratio, environmental benefits and low cost.

Coconut fibers were subjected to a pre-treatment according to the method modified by Malainine et al. [1] and partial delignification (bleaching with $NaClO_2$) according to the adapted method described by Wise et al. [2]. Cellulose nanocrystals were prepared by sulfuric acid hydrolysis (64 wt%) at 45°C for 120, 150 and 180 min [3]. Fiber length, width, aspect ratio (length-to-width) were analyzed in transmission electron microscopy (TEM), crystallinity degree was calculated by X-ray and Thermogravimetric analysis (TGA) was performed to study the thermal degradation of these cellulose nanocrystals.

Aqueous suspensions consisted mostly of individual CNCs (Figure 1) and some aggregates. The dimensions of CNCs are shown in Table 1. Typically, CNCs had a length ranging from 60 to 400 nm and a width of about 5.5 nm. The aspect ratio ranged from about 24 up to 56, with an average value of about 40 ± 16. These results are consistent to the reports where CNCs were taken out from different sources [4]. In general, the aspect ratio depends on the source and CNCs preparation conditions. High aspect ratio CNCs give a good reinforcing effect, resulting in mechanical improvements at low loads.

X-ray diffraction demonstrated that for non-hydrolyzed samples (raw material), there is an increase by about 20% percent in crystallinity with bleaching. For hydrolyzed samples, the hydrolysis time influenced on the degree of crystallinity. Samples hydrolyzed for 120 min had the highest crystallinity, whereas it slightly decreased with increasing time. This might have occurred because prolonged acid treatment not only removes the amorphous portion of cellulose fibers but also partly destroys the crystalline ones.

There were no major differences between the thermal behavior of CNCs obtained from the different hydrolysis times

Table 1 – Dimensions of CNCs prepared by acid hydrolysis at different times (120, 150, 180 min), determined from TEM images.

Hydrolysis time (min)	Length, L (nm)			Width, D (nm) *	Average aspect ratio, (L/D) *
	min	max	average		
120	85	374	194 ± 70	5.5 ± 1.5	39 ± 14
150	58	447	179 ± 59	5.5 ± 1.4	36 ± 12
180	58	322	204 ± 76	5.6 ± 1.3	41 ± 15

* Errors are the standard deviations of the distributions.

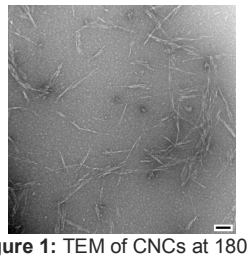

Figure 1: TEM of CNCs at 180 min

This study shows that cellulose nanocrystals can be successfully prepared from coconut fibers by acid hydrolysis, stimulating the use of coconut fibers as a novel renewable source of nanocellulose with potential as reinforcing agents in nanocomposites.

References

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