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Functionalization of Agave Cellulose Nanoparticles and its Characterization by Microscopy and Spectroscopy Techniques

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Agave is a non-used waste of the Tequila and Mezcal industry in Mexico. It posses a high content of cellulose, hemicellulose and lignins [1]. Cellulose is being widely studied as nanomaterial due to its reinforcement capacity, strenght and thermical expansion. However, metalic nanoparticles are more studied as reinforcers tan thus from organic sources [2].Nowadays, nanoparticles from many lignocellulosic materials are being synthetized, but non conventional materials, such as Agave waste, have not been used in the industry yet. To purify cellulose from agave waste could be the basis to obtain organic nanoparticles that can be functionalized with different compounds to improve their properties. Also, they could be used in many industries. Cellulose has crystalline properties, which make it stable, however, the amorphus part is the most important for the functionalization due to its free functional groups [3].

Thus, the objective of this work is to créate cellulose nanoparticles from agave fibers, functionalize them with an antimicrobial agent and a pH indicator. The nanoparticle synthesis was made using dry Agave fibers [4]which were washed in DI wáter for 24 hours to remove all the remaining sugars. Then, the fibers are pretreated with a NaOH solution and washed to remove the lignin and hemicelluloses and also change the orientation of the crystals from cellulose 1 to cellulose 2. The pretreated fibers are hydrolized with a H_2SO_4 and HCl solution for 3 hours in and ultrasonic bath. Finally, the functionalization was made by adding some drops of Congo Red solution and then 1 ml of silver nitrate 250mM dropping meanwhile heating to 80° for 24 hours.

The characterization of the materials was performed by Scanning Electron Microscopy (SEM), Mapping, Transmision Electron Microscopy (TEM), Confocal Microscopy, FTIR and XRD. SEM micrographs of the cellulose nanoparticles show an average partice size of 25 nm (Figure 1A). However, Figure 1B and 1C show how the addition of silver and Congo red changes the morphology, becoming more spherical and with an smaller size (9nm) to the ones non-functionalized. Figure 2 (A-D) show the confocal image where it can be seen the interaction between the silver and the Congo red with the cellulose and this was corroborated with the FTIR (Figure 2E) showing the characteristic peaks of the cellulose among other caused by the addition of silver nitrate.

TEM images show that the cellulose nanoparticles have a triclinic crystalline structure (Figure 3A) and are surrounded by amorphous zones. Functionalized nanoparticles with silver show a change in this molecular structure compared to the non-functionalized (Figure 3B). Figure 3C is from cellulose nanoparticles with the silver-Congo red complex and is clar that the amorphous zones have nearly dissapear from the edge. XRD was performed to corroborate these results and to probe that the main components and crystallinity change between pure cellulose nanoparticles and those functionalized.
These results demonstrate that it is possible to obtain cellulose nanoparticles from agave waste by a

simple method and that these cellulose can be functionalized to improve its properties that can be used in the biological área because they are more biocompatible tan the metalic nanopaticles, which nanotoxicity has not been described yet. These functionalized nanoparticles can be used in the industry as reinforcements, foams and intelligent packages.

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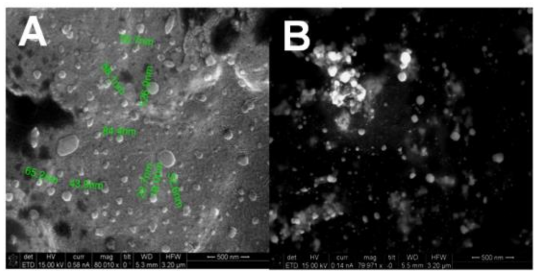

Figure 1. MEB. A) Pure cellulose nanoparticles with an average size of 25 nm. B) Functionalized cellulose nanoparticles with an average particle size of 9 nm.

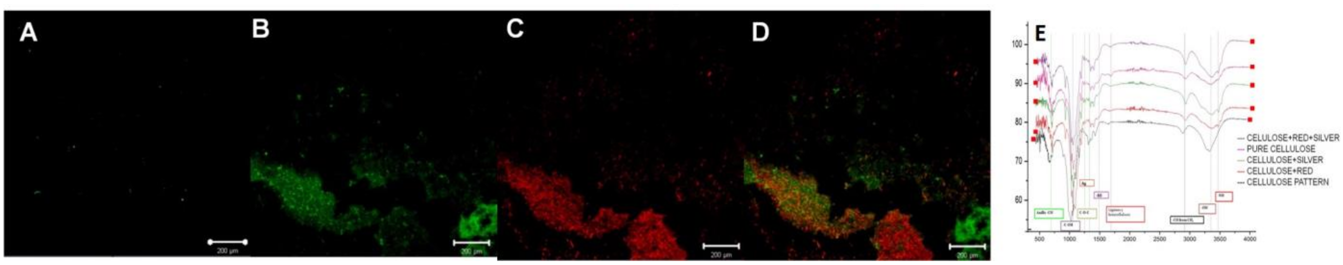

Figure 2. Confocal Micrographs A) Pure cellulose. B) Congo red-cellulose complex. C) Silver-cellulose complex. D) Cellulose-silver-Congo red complex. E) FTIR of the different cellulose nanoparticles obtained.

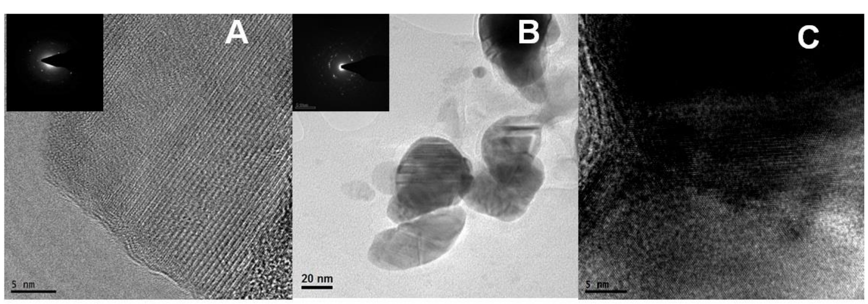

Figure 3. TEM. A) Cellulose nanoparticle micrograph and diffraction pattern on top. B) Silver-cellulose nanoparticle micrograph and diffraction pattern on top. C) Cellulose-silver-Congo red nanoparticle micrograph.

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