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Obtaining and Study of Cellulose Microcrystals from Agave Lechugilla

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Abstract – *Cellulose is an abundant material that finds in nature and it is possible to obtain from any vegetal organism. This biopolymer shows extraordinary properties at nanometric level that can use to create materials with unique characteristics. Recent investigations demonstrate that when utilizing natural fibers of vegetal origin as reinforcements of polymeric materials, it has achieved very fair results regarding the improvement of some mechanical properties displacing synthetic fibers like glass fiber or carbon fiber.*

In this work, it isolated cellulose microcrystals from agave lechugilla, employing physical and chemical procedures as acid hydrolysis, alkaline extraction, whitening and sonification. Optical Microscope (OM), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and Thermal Gravimetric Analysis (TGA) characterized the final product. The results obtained serve like basis for microcellulose obtaining.

Key Words: Cellulose, microcrystals, nanocrystals, acid hydrolysis.

1. INTRODUCTION

Along history, natural polymers have been so important, outstanding the use of linen, wool, cotton and especially cellulose. Cellulose finds forming part of wood, which constitutes the log of trees and plants, finding a percentage of 40 – 50% depending on the species. It is a biodegradable material present in different fibers, especially in Mexico. It exists a big number of potential uses for microcrystalline cellulose in different areas. Its properties at nanoscale allow the employing of many applications such as affinity membrane reinforcement to composite materials among others [1, 2]. In addition, cellulose nanocrystals have endless applications.

Cellulose is a long polymeric chain of variable molecular weight formed by glucose ring molecules ($C_6H_{10}O_5$) that contain between 43.6 and 45% of carbon, 6 and 6.5 % of hydrogen and the rest is oxygen [3], Glucose ring bind through a covalent bond of oxygen to C_1 of a glucose ring and to C_4 of adjacent ring [4].

The hydroxyl groups that are part of the cellulose macromolecule cause the variety of hydrogen bonds inter and intra-molecular that origin several ordered crystalline arrangements [5, 6].

Although, cellulose encompasses approximately 33% of the most of vegetal cells, the rest is a set of lipids and proteins that have to eliminate before extracting the crystal.

In order to achieve this, investigators have established the methods that involve the use of mechanical milling techniques to grind, crush and then to give a treatment with alkyl hydroxide and peroxide [7]. The obtaining of cellulose nanocrystals implicate an additional chemical procedure. The cellulose nanocrystals NCC produce when breaking the cellulose fibres and isolating crystalline regions.

The strong acid hydrolysis utilizes to isolate successfully cellulose nanocrystals. Strong acids as sulfuric, nitric and hydrochloric acid use to degrade cellulose fibres. Sulfuric acid seems to be the most effective. The accepted explanation currently represent this process of acid hydrolysis as a heterogeneous process that involves the acid diffusion in the cellulose fibres.

The acid interacts mainly in the amorphous regions of the cellulose, since they are easy to get because they have major surface. Therefore, the amorphous regions degrade first by the strong acid followed by the regions of greater crystallinity. Thus, a controlled hydrolysis can extract regions of a specific crystallinity from a cellulose sample. The amorphous regions in cellulose chains are more susceptible to acid hydrolysis, so that in this sections facilitate the breaking of glycosidic bonds realising the single crystals, as it observes in figure 1.

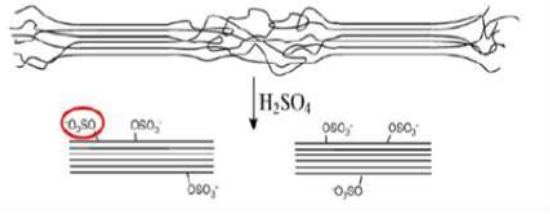


Figure 1. Acid hydrolysis. Cellulose hydrolysis reaction to produce NCC.

Among all of the investigations for the obtaining of nanocellulose, it realizes that if temperature and time increase, crystals reduce length and the performance decreases. The exposition prolonged to acids results in very

low performances, so that; it attributes to the acids the decomposition of entire cellulose fibres including crystalline regions [8].

In recent years, it has been important to the use of vegetal fibres utilizing nanotechnology, and so replacing synthetic fibres in some industrial applications, that in many cases are pollutant. This investigation work proposes an alternative in the use of sustainable lignocellulosic fibers as providers of cellulose for the development of materials with more value added.

Micro-cellulose production is significant, mainly because comes from a sustainable source of low environment risk. The study case is the lechugilla fiber (agave lechugilla) [9].

This is a species of plant of the Agavaceae family (figure 2)

Figure 2. Agave lechugilla plant

It has significant physical-mechanical properties and tension resistance. It is an excellent source of hard fibre of high strength and durability; it is also highly resistant to chemical solvents, heat, acids and abrasive products like diluted and concentrated acids, alcohols, and petroleum distillate, resistant in water at high temperatures.

Agave lechugilla fiber has an extraordinary resistance and high potential as reinforcement component in nanometric size in composite materials.

2. Description of the method

It employs biological material that collects and dries to the sun, the fibers pass through a sieve to diminish the variability of size.

The reagents that use for the extraction of cellulose micro-crystals are sulfuric acid H_2SO_4 at 98% (J.T. Baker) sodium hydroxide Na OH at 99% (Mercker) and sodium hypochlorite NaClO at 13%.

2.1 Micro-cellulose extraction

It adds NaClO at 13% to 3.0 g of agave lechugilla fibers shaking continuously during fifteen minutes. Later, it washes with distilled water and sonification during five minutes. After this treatment, it filters and extracts the fat content by means of alkaline treatment with 100 mL of NaOH at 10% solution in continuous shaking for one hour to isolate the cellulose from hemicellulose.

Consecutively, it washes, filters and adds 100 mL of distilled water to the solution with fiber, adding slowly 100 mL of sulfuric acid at 98% by shaking during two hours at constant temperature (50°C). Later, it adds 100 mL of distilled water to such suspension to stop the hydrolysis.

The samples neutralize with NaOH. It keeps at 3°C during 12 hours and filter, it washes with abundant distilled water, it adds NaClO to whiten the product obtained and washes with distilled water until obtaining a colorless filtering.

2.2 Characterization methods

Optical Microscope: It characterized the cellulose microcrystals prepared in solution with distilled water and later dried before observing them in a microscope Olympu model AX70.

Scanning Electron Microscope: The resulting cellulose microcrystals of the fibers diluted in distilled water and placed in the sample holder to analyze them in the microscope JEOL model JSM5800LV.

Transmission Electron Microscope of Field Emission: It allowed carrying out a whole analysis of the sample, accomplishing to study the morphology and its composition.

Thermal Gravimetric Analysis: The cellulose obtained characterized by means of thermal gravimetric analysis from 25°C to 800°C at a rate of 10°C/min under nitrogen atmosphere to prevent thermal-oxidative reactions, using simultaneous analyzer TGA-DSC model Q600.

3. Results.

When adding sodium hydroxide to the fiber, previously sieved, it observed a little precipitation of white color cellulose. When reacting the fibers with sulfuric acid, it formed a black suspension. The suspension caused by degradation of amorphous structures to the fiber structure. The suspension after filtering and washing, it brought to seven pH and immediately it carried out the drying to obtain the cellulose microcrystals (figure 3).

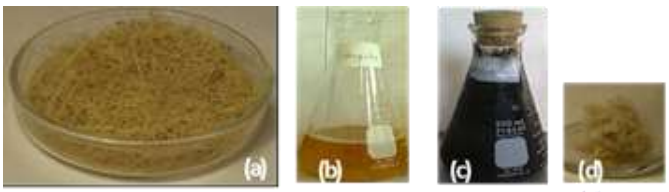


Figure 3. Lechugilla fiber, a) Without treatment, b) With NaOH, c) With H_2SO_4, d) Cellulose microcrystals.

Optical Microscope: It observe the fibers with previous treatment and in hydrolysis (NaOH and H_2SO_4). It identified that fibers with treatment with NaOH are porous and after the treatment with sulfuric acid have a non-variable structure with a naked eye. In the microscope took a magnification of 20 μm (figure 4).

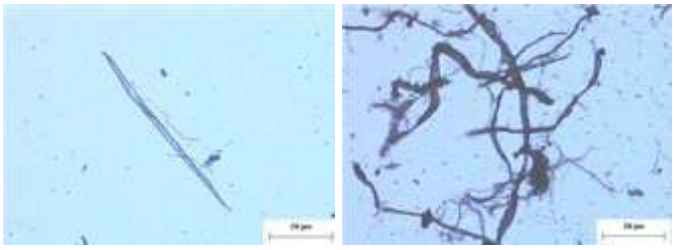


Figure 4. Lechugilla micrographics with treatment in NaOH and after sulfuric acid treatment.

Scanning Electron Microscope: The crystals micrographics of lechugilla cellulose crystals are very fibrous. It observes fibers with well-defined with magnifications of 50 and 20 μm (figure 5).

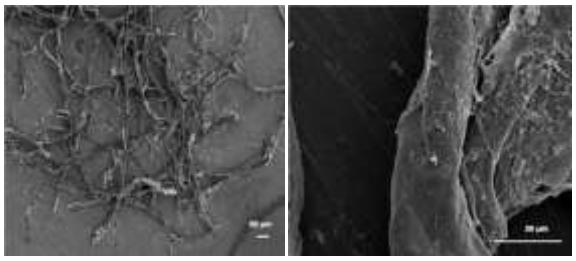
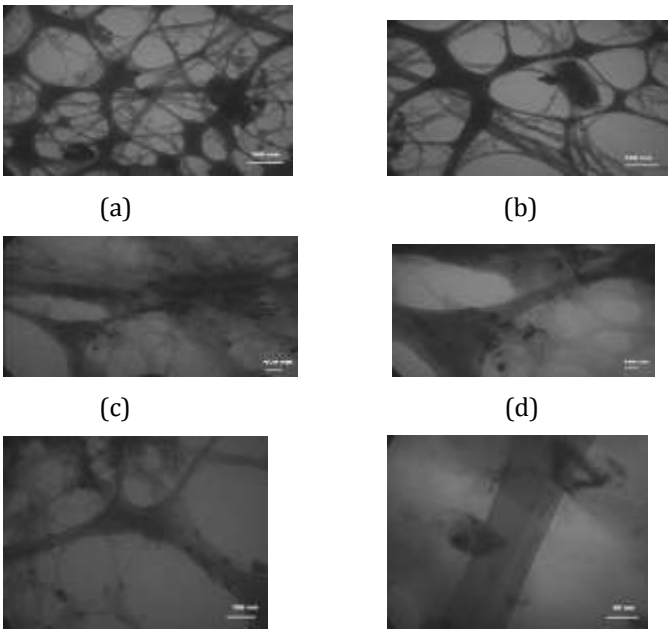


Figure 5. Lechugilla cellulose micrographics observed in Scanning Electron Microscope.

Transmission Electron Microscope: The image show the wall and cell membrane of the fibers. The micrographics obtained from Transmission Electron Microscope exhibit the fiber net that shape the cellulose structure. The lechugilla fiber is surrounded of cell membrane. The magnifications are of 100nm (figure 6).



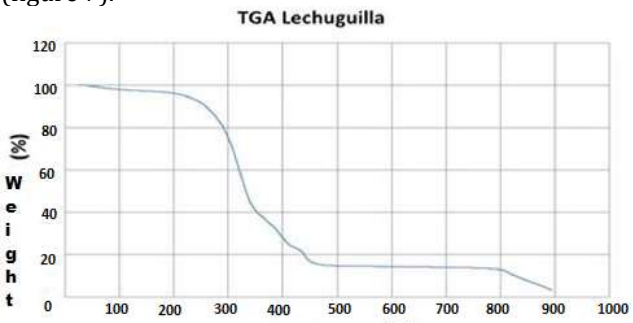
(a) (b)

(c) (d)

(e) (f)

Figure 6. (a) and (b) Lechugilla fiber, (c) and (d) Cell membrane, (e) and (f) Lechugilla cell wall

Thermal Gravimetric Analysis (TGA): The TGA of the lechugilla presented moisture evaporation at 98.4°C, losing 2.06% of its original weight, the lignin and hemicellulose decomposition at 207.26°C resulting in a loss of weight of 4.3%. A cellulose decomposition displacement of 417.57°C with decreasing of weight of 76.22% considering that the temperature of degradation of lignin and hemicellulose is between 180 – 340°C [10]. The cellulose at 600°C loses 82.93% of its weight, by referencing the commercial cellulose that presents degradation from 400 to 600°C (figure 7).



	Moisture (Water)	Lignin and hemicellulose decomposition	Beginning of cellulose decomposition	Cellulose degradation
T °C	98.42	207.26	417.57	600
% Loss Weight	2.06	4.3	76.22	82.93

Figure 7. Thermal Gravimetric Analysis of lechugilla cellulose microcrystals.

6. CONCLUSIONS

It obtained microcrystals from agave lechugilla observable in SEM microscope and analyzed the morphology in TEM at 100 nm. The fiber presents thermal stability, beginning with substantial loss from 400 to 600°C. The study of this work serves like the basis to obtain cellulose microcrystals. The features of the obtained product represent a promissory source for the production on nanocellulose and applicable to reinforcement materials.

To improve the lechugilla cellulose microcrystals performance, it has to evaluate several times of reaction, vary acid concentrations and decrease the time of reaction with NaOH, only to mention some variables, which can serve for future investigations.

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