



Synthesis of Cellulose Nanofibrils from Banana Residues for the Production of Textile Fibers

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Abstract Agriculture is one of the main sources of solid residues and the banana industry is a good example of this since only the fruit is used. In 2016, 1, 907,822.60 tons of residues were generated in the Mexican Republic. The objective of this work was to evaluate the cellulose nanofibrils obtained from banana rachis residues; and develop from them biomaterials that could substitute synthetic fibers in textiles. This research was divided into two phases: the first phase included waste generation study and sampling at Mexico City's Central Market, according to Mexican Standard NMX-AA-15-1985. The second phase, at laboratory level, was conducted to obtain cellulose nanofibrils from banana rachis residues using the chemical method with sodium hydroxide. The obtained cellulose nanofibrils were first dissolved in 1-ethyl-3-methylimidazolium acetate and N, N-dimethylformamide ionic liquid, then mixed in a solution of PCL with acetone and finally processed in an electrospinning equipment to obtain yarn. The generation study showed that up to 6 tons of banana rachis residues, that can be processed to be used in the textile industry, are generated daily in the Central Market. According to the analyzes of proximate chemical composition and cell walls, banana rachis residues contain 40% of cellulose. The cellulose nanofibrils obtainment process had a yield of 17.75%. As for the making of the yarn through electrospinning, better dissolution results were observed with 16% material by weight. The textile fiber was obtained with a flow of 3.5 to 8 mL / s, a voltage ranging from 15 to 18 Kv and a collector distance from 15 to 20 cm.

Keywords nanofibrils, banana, fibers, textile

Introduction

The agriculture and forestry industries are among the main sources of solid organic waste [1], generating a large number of by-products, only a few of which have a defined use. In the banana industry, a large amount of vegetable waste is produced, since only the fruit is used and once harvested the other parts of the plant such as pseudostem, leaves and rachis (part of the plant that holds the bundles of fruits) have to be discarded [2]. The annual production of bananas in Mexico is about two million tons and since the fruit cluster represents only one-fifth of the weight of the plant, an enormous amount of waste is generated by its production and although some of it is exploited in various ways, the vast majority is dumped [3].

Although this situation represents a challenge not only in Mexico City but also in many areas of the Mexican Republic, it is currently often invisible to many and has thus been overlooked in its social, environmental and legal aspects.



The poor management of these wastes leads to a progressive and cumulative deterioration of the environment that may, in some cases, constitute a public health problem.

Nowadays, the cellulose nanofibrils from the banana rachis are of great interest because of their renewable nature, good mechanical properties and very high length-to-diameter ratio, which leads to a large surface area compared to traditional fibers.

The objective of this research was to make use of plantain (*Musa balbisiana*) waste to obtain cellulose nanofibrils to replace synthetic fibers.

Materials and Methods

This research was divided into two phases: field work and experimentation.

During the field work phase, the necessary samples were taken from rachis or banana trunk residues in Mexico City's Central Market. Residues generated from plantain banana (*Musa balbisiana*) were used since it is the cultivar registering the greatest demand.

The generation study and quarter sampling method were performed according to Mexican Norm NMX-AA-15-1985 - Quarter sampling method [4].

In the second phase, experimentation, laboratory work was conducted in 4 stages. During the first stage, a proximate chemical and cell walls analysis was carried out on the residues to determine the moisture, fiber and cell contents, as well as hemicellulose and cellulose present in the banana rachis [5].

The second experimentation stage was to obtain cellulose nanofibrils from banana rachis residues. The chemical method used consisted of three stages: pre-treatment, bleaching and acid hydrolysis. During pre-treatment, the rachis was reduced to 30 cm sections, washed with water and the fibers were cut at a length of 2 cm. It was then allowed to dry at 105° C during 14 hours. In the bleaching stage, an alkaline treatment was performed with 15% NaOH (w/v) at boiling temperature under continuous stirring during 100 minutes. Most lignin was removed by solubilization through hydrolysis reactions, releasing cellulose fibers. It was washed with distilled water and filtered until neutral pH was reached.

In this stage, cellulose was obtained with residues of lignin and black liquor product of the digestion of different organic compounds, among them lignin, mixed with the consumed reagents. To remove the residual lignin, bleaching was performed with NaClO 0.5% during 1 h at 70 °C. It was filtered and washed with hot distilled water until neutral pH. It was then dried at 80 °C. Finally, in the acid hydrolysis step, it was hydrolyzed in 61% H₂SO₄ (w/v) during 90 minutes at 45° C. The obtained milky colloidal suspension was washed with distilled water using repeated cycles of centrifugation from 10 to 3500 rpm, until a pH ranging from 3 to 5 was reached [6-9].

In the next experimentation stage, the nanofibrils obtained in the prior stage were analyzed by scanning electron microscopy to determine their morphology and sizes.

In the fourth stage, an electrospinning equipment [10, 11], was used to make yarn from the obtained nanofibrils. The material was dissolved in 1-ethyl-3-methylimidazolium acetate and N, N-dimethylformamide ionic liquid [12, 13] at 45 °C during 24 hours. Subsequently, a solution of PCL/Acetone [14], was added, and it was stirred for 3 hours. The resulting solution was placed in a syringe, varying the volume, flow, voltage and manifold distance.

Results and Discussion

The results of the quarter sampling method performed at Mexico City's Central Market are shown in Fig. 1.

Plantain is the only banana cultivar that arrives at Mexico City's Central Market with rachis, the other varieties arriving without it, in clusters and in boxes. They generate more than 6 t/day of residues that are taken to final disposal without being separated from inorganic waste, and much less used.

Rachis contains 40% of usable cellulose; and 16.2% of hemicellulose and 11% of lignin that must be removed during the process of obtaining nanofibrils.

To obtain nanofibrils, 300 grams of banana rachis residues were cut in 30-cm long sections and later in 2-cm long fibers. Rachis was manually defibrated and the fiber obtained was submitted to the drying process in order to carry out the alkaline treatment to obtain cellulose with lignin and black liquor residues (Fig. 2).





Figure 1: Banana rachis residue



Figure 2: Black liquor

Bleaching was used and 3.96 g of cellulose were obtained out of 100 g of residues. Using repeated cycles of centrifugation from 10 to 3500 rpm in an Avanti J-26S XPI refrigerated centrifuge, 0.72 grams of residual lignin free cellulose (Fig. 3) was obtained.



Figure 3: Centrifuged and dried rachis cellulose

In the morphological analysis of the nanofibrils obtained by scanning electron microscopy, 3 banana samples were analyzed; the obtained images are shown in Fig. 4 (a and b).



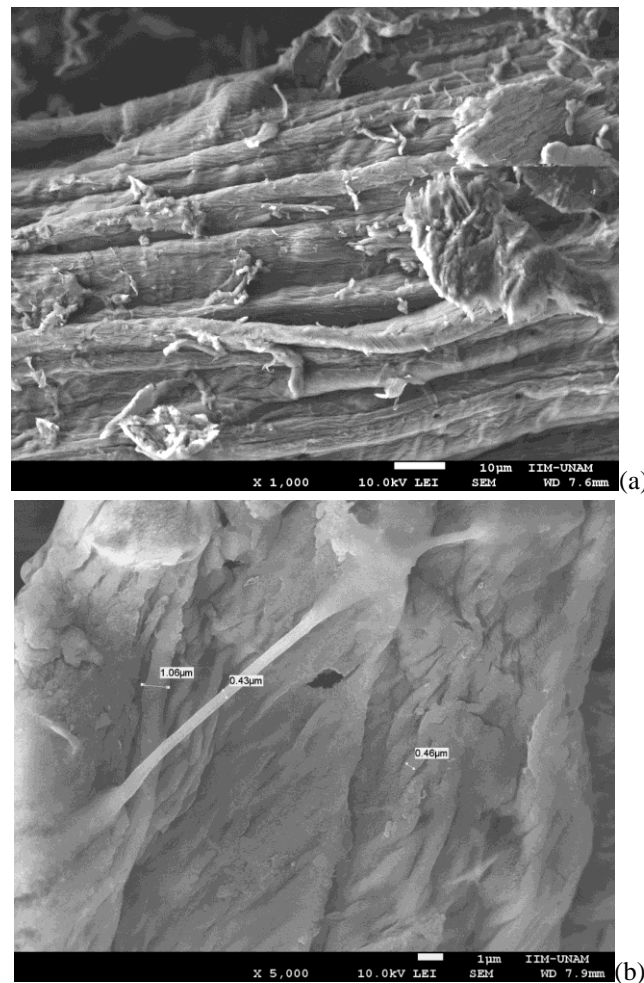


Figure 4: (a) NFC Banana I. (b) NFC Banana II

In Fig. 4 (a), fibers of about 104 microns can be seen, made up of microfibrils of a thickness of 4 microns that are in turn made of more fibers of a thickness of approximately 500 nm. In Fig. 4 (b) a behavior similar to the first banana sample shown in Fig. 4 (a) can be seen.

In the elaboration of the yarn an electrospinning equipment was used, varying the concentration of the material in the 1-ethyl-3-methylimidazolium acetate and N, N-dimethylformamide solution; working with 12% to 16% material by weight, with better dissolution results being observed at 16% material by weight.

With the electrospinning equipment, a yarn was obtained from a flow of 3.5 to 8 mL / s, a voltage between 15 and 18 Kv and manifold distance from 15 to 20 cm. FIG. 5 shows the electrospinning equipment used and the nanofibrils obtained.

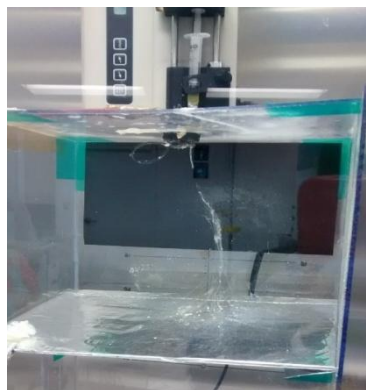


Figure 5: Fiber obtained by electrospinning



Subsequently, the fibers (or yarn) obtained were analyzed by scanning electron microscopy to determine their morphology. FIG. 6 shows the image of the fibers obtained from the 16% banana and 99% PCL/Acetone solution. These fibers have a thickness of approximately 12.14 to 2 μm .

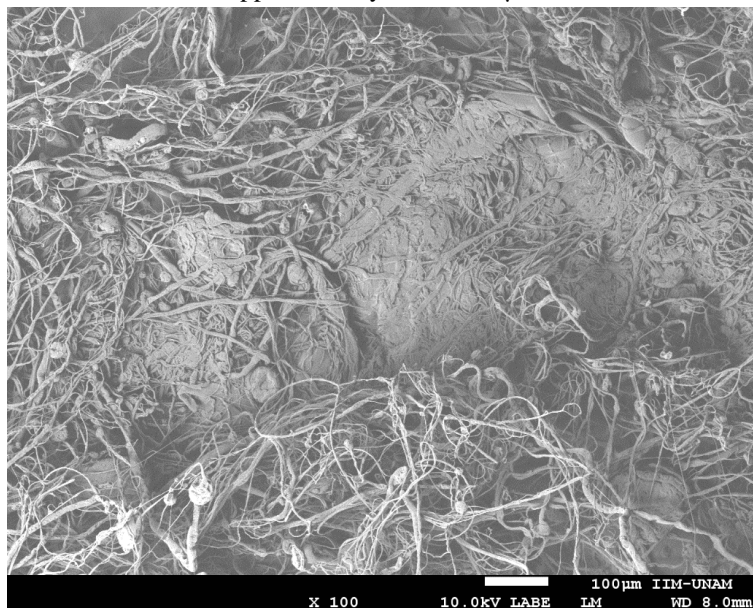


Figure 6: 16% banana and 99% PCL/Acetone solution electrospun fibers

Finally, the thickness and weight of the yarns obtained from the 1% -16% banana were determined. Thicknesses ranged from 0.002 to 0.065 mm, with a weight of 0.12 g. As regards technical feasibility, time and costs involved in the entire process were calculated. The amount of chemical waste generated and the energy used during the process were determined to determine environmental feasibility.

Conclusions

Up to 6 tons per day of plantain rachis residues are generated at Mexico City's Central Market. It is the first time that this generation data is obtained since no previous record was available and said raw material is directly sent to final disposal without any kind of treatment or exploitation. As shown in this study, banana rachis residues can be put to beneficial use, and thus contribute to the decrease of the large amount of waste generated and provide a benefit for the environment.

According to the results obtained from proximate chemistry analysis, banana rachis contains 40% cellulose. It can thus be inferred that banana rachis residues can be used as feed for livestock due to their digestible energy level of 2691.08 kcal/kg in base 90 and 2990.09 kcal / kg in base 100.

Obtaining cellulose nanofibrils from banana residues is a quick and simple process. The procedure had a yield of 17.75%.

The yarn was obtained with the electrospinning technique, using a mixture of two solutions. It was concluded that the solution with the best quality was 16% by dry weight.

As a conclusion, banana rachis residues can be used to obtain cellulose nanofibrils, reducing thus their environmental impact. The process of obtaining cellulose nanofibrils from banana rachis residues is environmentally sound because little amount of chemical reagents and energy is used, and few man-hours are needed, generating therefore less waste. Although the electrospinning process is an innovative and promising yarn manufacturing method, further testing is required.

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