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Research Article

Isolation and Characterization of Nanofibrillar Cellulose from Agave tequilana Weber Bagasse

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1. Introduction

The agroindustrial waste that is available worldwide has become a topic of interest, given its potential as a raw material for the generation of diverse products [1, 2]. There is, in fact, a need to add value to biomass that has been otherwise reduced to “waste” or residue from different agroindustrial processes [3–6]. This effort considers the use of agave residues for potential uses in the production of cellulose nanofibers, which can then be used in the manufacture of advanced materials.

Lignocellulosic biomass is generated as a residue from the agricultural, forestry, and related industrial sectors. Agroforest waste is generated in large volumes, mainly in the form of woody and plant residues. It not only has an economic impact as far as the handling of the solids but also causes serious environmental challenges during their disposal [7]. Among the different raw materials of lignocellulosic origin, agave bagasse is an abundant source of biomass, most relevant to the state of Jalisco, Mexico. The continuous and growing demand for tequila leads to the production of millions of litres of this beverage; therefore, there is a major concern with the generation and handling of the large volume of the associated agroindustrial waste, agave bagasse [4, 8].

Agave bagasse is a residual fibrous material that is left after the extraction of the fermentable juice from the pineapple of Agave tequilana Weber. Around 40% of the total weight of the consumed agave corresponds to residual bagasse [9]. In 2016, the tequila industry alone generated...
approximately 941 thousand tons of such lignocellulosic waste [4, 10]. Due to its high availability, bagasse residues have caused serious problems to the tequila industry, given that their disposal involves high environmental, capital, and operational costs [11, 12]. Therefore, finding routes for the utilization of this resource has become a necessity, and consequently, the generation of high-value products is extremely appealing [13, 14].

Agave bagasse is rich in cellulose, hemicellulose, and lignin. This composition makes this material suitable to different areas of applications. Reported uses include harvesting biopolymers, enzymes such as laccases, and the production of compost [11], hydrogen [9], and nanomaterials [3, 12]. The use of agave residuals is still very marginal. Since most of this material ends up as waste in clandestine landfills, environmental and economic problems are thriving in the region. The inappropriate disposal of agave bagasse causes negative effects on the fertility of farmland, the generation of contaminating leachates and phytosanitary risks, mainly due to the inadequate incorporation of this material into the soil [4].

In previous work of our group, we have investigated the use of agave bagasse to obtain cellulose nanocrystals using the organosolv process to extract cellulose followed by acid hydrolysis with sulfuric acid [3]. The organosolv method allowed an almost complete delignification of the cellulose and high solubilization of the lignin produced [3, 6]. The organic solvents used, mainly ethanol (but also including methanol and acetone), are relatively inexpensive and have low toxicity [15].

The difference between cellulose nanocrystals and cellulose nanofibers is in the length, the cellulose nanocrystals are in the order of 50 to 100 nm, and the nanofibers can have a micrometer length [14]. The inherent properties of cellulose nanofibers make them competitive if compared to those of the precursor material. The greater surface area per unit weight, generated by reducing the fiber size to the nanometre scale, makes the obtained material unique as far as properties such as mechanical strength. They allow for applications in different areas such as lightweight composite materials and systems for water treatment, food, and medicine [13, 14].

In the present study, agave bagasse was processed following a simple and low-cost method, namely, the organosolv process that extracted the cellulosic component. Following microfluidization, cellulose nanofibrils (CNFs) were produced. The microfluidization is effective in defibrillating or separating the fibers at room temperature with the application of high shearing [16]. The resulting CNF can be applied in different areas, for example, in tissue engineering [17] and in the biomedical field [18] as reinforcement material in biodegradable films and composites [19, 20].

2. Materials and Methods

Agave (Agave tequilana Weber) bagasse was collected as residues from a tequila factory (distillery Cava de Oro) in the village of Arenal Jalisco, Mexico, and was used as the raw material in this work. The bagasse was washed, dried, and cut in a hammer mill (PC408-75), then the particles were sieved, and those corresponding to 40 mesh (425 μm) were separated for further processing.

2.1. Chemical Composition of the Agave Bagasse. The chemical composition of the agave bagasse was determined following the standards of the Technical Association of the Pulp and Paper Industry [21]. The cellulose content was determined using the T 203 cm−99 standard, and acid-soluble lignin content was determined using T 222 om-02. The extractable compounds in acetone were determined according to T 207 cm−99 and ash content with T211 om-02. The reagents used to determine the chemical composition were purchased from Sigma-Aldrich, Mexico: sodium hydroxide (NaOH) 97% w/v, potassium hydroxide (KOH) 90% w/v, sodium chloride (NaClO3) 80% w/v, boric acid (H3BO3) 99.5% w/v, acetic acid (CH3COOH) 99% w/v, and nitric acid (HNO3) 65% w/v. All the experiments were carried out in triplicate.

2.2. Organosolv Treatment for Cellulose Fiber from Agave Bagasse. The organosolv treatment used organic solvents to remove lignin from the lignocellulosic material. These treatments were carried out according to previous research [3, 8]: 400 g (on dry basis) of agave bagasse was treated for 2.5 h, in a high-pressure digester at 175°C with 2 L of deionized water, 2 L of ethanol, and 12 mL of acetic acid. Once the container had cooled down, the resulting black liquor (lignin-rich solution) was extracted, and the solid phase (fibers) obtained was washed with water. Subsequently, the fibers were bleached with NaClO3 (80% w/v) and CH3COOH (99% w/v) for a total of 4 h, with 1 h between the addition of each of the reactants. The hemicellulose was then extracted via KOH treatment (24% w/v) for 12 h. The cellulose was extracted with NaOH (17.5% w/v) and H3BO3 (4% w/v). Finally, the material was washed several times with deionized water until reaching neutral pH, and the residual acid was removed.

2.3. Cellulose Nanofibers (CNFs). The agave cellulose was processed as an aqueous dispersion at 1.5% solid content using Microfluidizer M-110P. A mass of 7.5 g of the lignin-free (bleached) agave cellulose fibers were dispersed in 500 mL of deionized water, and the suspension was kept under magnetic stirring for 12 h. Thereafter, the material was passed several times through the microfluidizer using the 400 and 200 μm chambers arranged in series. Subsequently, the material was passed 6 more times using the 200 and 100 μm chambers, yielding cellulose nanofibrils, according to previous research [22]. The equipment used an intensifying pump that increased the pressure in the chamber, which favoured the interaction between the fibers and the shearing forces and impact against the current of fibrillation [23].
2.4. Characterization of Cellulose and CNFs. The structure and morphology of the extracted agave bagasse fibers were analysed by imaging using a scanning electron microscope (FE SEM Tescan MIRA3 LMU). The material was also characterized by infrared spectrometry (FTIR, PerkinElmer Spectrum GX) using solid samples (0.20 g); 16 scans were acquired in the range of 400–4400 cm⁻¹ and a resolution of 4 cm⁻¹. This analysis was also performed on the starting material to compare the differences due to the organosolv treatment of the fibers.

Atomic force microscopy (AFM, Nanoscope III from Digital Instruments MultiMode) was used for imaging. The CNF samples were prepared by spin coating a suspension of 0.01% w/w nanofibers on a mica substrate and dried at room temperature. The images were acquired under contact mode and analysed using ImageJ 1.45 software. The apparent zeta potential of the nanofibers was determined using a Zetasizer NanoZS90, which used 1 mL of the cellulose extracted by the organosolv method.

Using the following equation:
\[
\text{IC} = \frac{I_{002} - I_{AM}}{I_{002}} \times 100,
\]
where \(I_{002}\) is the maximum intensity of the refractive peak (002), while \(I_{AM}\) is the intensity corresponding to the amorphous component. The analysis was also carried out for cellulose extracted by the organosolv method.

The structure of the agave bagasse was determined by X-ray diffraction (XRD, Empyream). The degree of crystallinity of the cellulose was determined using XRD (Empyream). The degree of crystallinity of the cellulose nanofibers was determined by X-ray diffraction (XRD, Empyream). The degree of crystallinity of the cellulose nanofibers was determined by X-ray diffraction (XRD, Empyream).

The zeta potential of the nanofibers was determined using a Zetasizer Nano ZS90, which used 1 mL of the cellulose nanofiber suspension at 0.35% solids. In addition, the cellulose was also measured using a Zetasizer Nano ZS90, which used 1 mL of the cellulose nanofiber suspension at 0.35% solids. The measurement of the zeta potential was also performed on the starting material to compare the differences due to the organosolv treatment of the fibers.

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The XRD spectrum and crystallinity index are shown in Figure 4 and Table 2, respectively. The crystallinity index increased with the removal of the noncrystalline components. As expected, the crystallinity index is lower compared to that of cellulose nanocrystals, about 80% [3]. In the XRD spectra, it was observed that cellulose in the native state has a semicrystalline structure with characteristic crystalline domains of cellulose I, with the maximum 2θ peak between 19° and 22° (101) [31]. Once the cellulose was subjected to the pretreatment using the organosolv method and was bleached, a transformation to cellulose II took place, with three peaks recorded in the diffractogram (Figure 4(b)). The main diffraction peak (002) was between approximately 25° and 28°, while the intensity of the amorphous part was recorded between 12° and 14°, which were the characteristics of cellulose II [30]. The change in the crystallinity of cellulose I to cellulose II is attributed to amorphous cellulose I which dissolved during acid hydrolysis [26]. However, Figure 4(b) also shows the signal (101) indicating that cellulose I was also present in the treated fiber.

The TG curves for organosolv cellulose and cellulose nanofibers (Figure 5) in nitrogen atmosphere show an initial small drop between 50°C and 100°C, which corresponds to a mass loss of approximately 5% due to the moisture absorbed. Then, there is a weight loss event approximately between 313 and 388°C, which begins at 313°C for the cellulose and 343°C for the NFC; the weight loss for this process was 62% for the cellulose and 78% for the NFC. The DTG curves showed corresponding peaks at 349°C and 378°C, respectively.
This step can be attributed to cellulose depolymerisation. The thermal degradation of nanofibers proceeded at higher temperatures than their respective raw organosolv cellulose. The thermal stability of nanofibers is shown in Figure 5. This is an interesting characteristic for future applications of nanofibrillar cellulose in processing polymeric nanocomposites that involve exposure time at higher temperatures.

4. Conclusions

In this study, it was possible to verify that from agro-industrial wastes such as agave bagasse, and cellulose nanofibers can be present, which had an uniform length and an average diameter of 75 nm. The material obtained showed a good percentage of crystallinity and a high surface charge, which means that the nanofibers are stable and do not agglomerate, giving them desirable properties for future applications in various fields, for example, food, biomedical, in the elaboration of composite materials, membranes for water treatment, and the textile industry, among others. Also, it is important to point out that, to obtain cellulose and cellulose nanofibers, environmentally friendly methods (organosolv and mechanical defibrillation) were used, respectively. These processes have a significant impact on the size distribution of nanofibers because it was only necessary

Table 2: Crystallinity index, z potential, and fiber size of the cellulose and nanocellulose.

| Samples                          | Crystallinity index (%) | Zeta potential (mV) | Fiber size |   |
|----------------------------------|-------------------------|---------------------|------------|---|
| Agave bagasse                    | 60.5                    | —                   | 144 mm     | 500 μm |
| Organosolv cellulose after bleaching | 63.5              | −30                 | 0.5 mm     | 25 μm  |
| Cellulose nanofibrils            | 68.5                    | −42                 | 500 nm     | 75 nm  |

Figure 4: XRD spectra of agave bagasse fiber, organosolv cellulose, and nanofibrillar cellulose.

Figure 5: TG curves of organosolv cellulose and nanofibrillar cellulose.
to perform 6 cycles in the microfluidizer without the need for previous treatment (chemical or enzymatic), decreasing energy consumption.

**Data Availability**
The data used to supporting of the study are included in the supplementary file.

**Conflicts of Interest**
The authors declare that they have no conflicts of interest.

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**Supplementary Materials**
The supplementary materials file is the data obtained for X-ray diffraction, IR spectroscopy, and thermogravimetric analysis (TGA) of agave bagasse, organosolv cellulose, and nanofibers cellulose. (Supplementary Materials)

**References**

[1] M. Bilal, M. Asgher, H. M. N. Iqbal, H. Hu, and X. Zhang, "Biotransformation of lignocellulosic materials into value-added products—a review," *International Journal of Biological Macromolecules*, vol. 98, pp. 447–458, 2017.

[2] J. I. Morán, V. A. Alvarez, V. P. Cýras, and A. Vazaquez, "Extraction of cellulose and preparation of nanocellulose from sisal fibers," *Cellulose*, vol. 15, no. 1, pp. 149–159, 2007.

[3] J. A. Hernández, V. H. Romero, A. Escalante et al., "Agave tequilana bagasse as source of cellulose nanocrystals via organosolv treatment," *BioResources*, vol. 13, no. 2, 2018.

[4] G. S. Aleman-Nava, I. A. Gatti, R. Parra-Saldívar, J.-F. Dallemand, B. E. Rittmann, and H. M. N. Iqbal, "Biotic-technological valorization of Tequila waste and by-product streams for cleaner production—a review from bio-refinery perspective," *Journal of Cleaner Production*, vol. 172, pp. 3713–3720, 2018.

[5] A. Arevalo-Gallegos, Z. Ahmad, M. Asgher, R. Parra-Saldívar, and H. M. N. Iqbal, "Lignocellulose: a sustainable material to produce value-added products with a zero waste approach—a review," *International Journal of Biological Macromolecules*, vol. 99, pp. 308–318, 2017.

[6] M. Bilal, H. M. N. Iqbal, H. Hu, W. Wang, and X. Zhang, "Metabolic engineering and enzyme-mediated processing: a biotechnological venture towards biofuel production—a review," *Renewable and Sustainable Energy Reviews*, vol. 82, pp. 436–447, 2018.

[7] M. Bilal, M. Z. Nawaz, H. M. N. Iqbal et al., "Engineering ligninolytic consortia for bioconversion of lignocellulosics to ethanol and chemicals," *Protein & Peptide Letters*, vol. 25, no. 2, pp. 108–119, 2018.

[8] J. M. Gutiérrez-Hernández, A. Escalante, R. N. Murillo-Vázquez, E. Delgado, F. J. González, and G. Toriz, "Use of agave tequilana-lignin and zinc oxide nanoparticles for skin photoprotection," *Journal of Photochemistry and Photobiology B: Biology*, vol. 163, pp. 156–161, 2016.

[9] J. Arreola-Vargas, A. Flores-Larios, V. González-Alvarez, R. I. Corona-González, and H. O. Méndez-Acosta, "Single and two-stage anaerobic digestion for hydrogen and methane production from acid and enzymatic hydrolysates of Agave tequilana bagasse," *International Journal of Hydrogen Energy*, vol. 41, no. 2, pp. 897–904, 2016.

[10] National Regulator Council for Tequila Industry, 2018, https://www.crt.org.mx/.2018.

[11] G. Íñiguez, A. Valadez, R. Manríquez, and M. V. Moreno, "Utilization of by-products from the tequila industry. Part 10: characterization of different decomposition stages of agave tequilana webber bagasse using FTIR spectroscopy, thermogravimetric analysis and scanning electron microscopy," *Revista Internacional de Contaminación Ambiental*, vol. 27, no. 1, pp. 61–74, 2011.

[12] M. Á. Robles-García, C. L. Del-Toro-Sánchez, E. Márquez-Ríos et al., "Nanofibers of cellulose bagasse from Agave tequilana Weber var. azul by electrospinning: preparation and characterization," *Carbohydrate Polymers*, vol. 192, pp. 69–74, 2018.

[13] P. Phanthong, P. Reubroycharoen, X. Hao et al., "Nanocellulose: extraction and application," *Carbon Resources Conversion*, vol. 1, no. 1, pp. 32–43, 2018.

[14] C. Salas, T. Nypelö, C. Rodriguez-Abreu, C. Carrillo, and O. J. Rojas, "Nanocellulose properties and applications in colloids and interfaces," *Current Opinion in Colloid & Interface Science*, vol. 19, no. 5, pp. 383–396, 2014.

[15] P. Nantharpithom, W. Kraithong, N. Viriya-empikul, and A. Eiad-Ua, "Organosolv pretreatment transformation process of bagasse to porous carbon material," *Materials Today: Proceedings*, vol. 4, no. 5, pp. 6261–6266, 2017.

[16] A. Ferrer, I. Filipponen, A. Rodríguez, J. Laine, and O. J. Rojas, "Valorization of residual empty palm fruit bunch fibers (EPFBB) by microfluidization: production of nanofibrillated cellulose and EPFBB nanopaper," *Bioresource Technology*, vol. 125, pp. 249–255, 2012.

[17] B. Nasiri-Nasrabadi, M. Mehrasa, M. Rafienia et al., "Porous starch/cellulose nanofibers composite prepared by salt leaching technique for tissue engineering," *Carbohydrate Polymers*, vol. 108, pp. 232–238, 2014.

[18] L. M. M. Costa, G. M. de Olyveira, B. M. Cherian et al., "Bionanocomposites from electrosyn PVA/pineapple nanofibers/Stryphnodendron adstringens bark extract for medical applications," *Industrial Crops and Products*, vol. 41, pp. 198–202, 2013.

[19] A. Ghanbari, T. Tabarsa, A. Ashori, A. Shakeri, and M. Mashkour, "Preparation and characterization of thermoplastic starch and cellulose nanofibers as green nanocomposites: extrusion processing," *International Journal of Biological Macromolecules*, vol. 112, pp. 442–447, 2018.

[20] E. Robles, I. Urruzola, J. Labidi, and L. Serrano, "Surface-modified nano-cellulose as reinforcement in polylactic acid to conform new composites," *Industrial Crops and Products*, vol. 71, pp. 44–53, 2015.

[21] TAPPI, Technical Association of the Pulp Paper, *TAPPI Test Methods*, TAPPI, Atlanta, GA, USA, 1988.

[22] C. A. Carrillo, T. E. Nypelö, and O. J. Rojas, "Cellulose nanofibrils for one-step stabilization of multiple emulsions (W/O/W) based on soybean oil," *Journal of Colloid and Interface Science*, vol. 445, pp. 166–173, 2015.
[23] J. Velásquez-Cock, A. Serpa, L. Vélez et al., “Influence of cellulose nanofibrils on the structural elements of ice cream,” Food Hydrocolloids, vol. 87, pp. 204–213, 2019.

[24] L. Segal, J. Creely, A. Martin, and C. Conrad, “An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer,” Textile Research Journal, vol. 29, no. 10, pp. 786–794, 1959.

[25] S. Kestur, T. H. Flores-Sahagun, L. P. Dos Santos et al., “Characterization of blue agave bagasse fibers of Mexico,” Composites Part A: Applied Science and Manufacturing, vol. 45, pp. 153–161, 2013.

[26] E. Robles, J. Fernández-Rodríguez, A. M. Barbosa et al., “Production of cellulose nanoparticles from blue agave waste treated with environmentally friendly processes,” Carbohydrate Polymers, vol. 183, pp. 294–302, 2018.

[27] A. Dufresne, D. Dupeyre, and M. R. Vignon, “Cellulose microfibrils from potato tuber cells: processing and characterization of starch–cellulose microfibril composites,” Journal of Applied Polymer Science, vol. 76, no. 14, pp. 2080–2092, 2000.

[28] N. Lin and A. Dufresne, “Surface chemistry, morphological analysis and properties of cellulose nanocrystals with gradient sulfation degrees,” Nanoscale, vol. 6, no. 10, pp. 5384–5393, 2014.

[29] F. M. Pelissari, P. J. D. A. Sobral, and F. C. Menegalli, “Isolation and characterization of cellulose nanofibers from banana peels,” Cellulose, vol. 21, no. 1, pp. 417–432, 2014.

[30] S. Elanthikkal, U. Gopalakrishnapanicker, S. Varghese, and J. T. Guthrie, “Cellulose microfibres produced from banana plant wastes: isolation and characterization,” Carbohydrate Polymers, vol. 80, no. 3, pp. 852–859, 2010.

[31] S. Kumar, R. Gupta, Y. Y. Lee, and R. B. Gupta, “Cellulose pretreatment in subcritical water: effect of temperature on molecular structure and enzymatic reactivity,” Bioresource Technology, vol. 101, no. 4, pp. 1337–1347, 2010.
