

Research Article

Isolation and Characterization of Nanofibrillar Cellulose from *Agave tequilana* Weber Bagasse

Hasbleidy Palacios Hinestroza,¹ Javier A. Hernández Díaz,¹ Marianelly Esquivel Alfaro,² Guillermo Toriz,³ Orlando J. Rojas ,⁴ and Belkis C. Sulbarán-Rangel ¹

¹Department of Water and Energy, University of Guadalajara Campus Tonalá 45425, Mexico

²Polymers Research Laboratory (POLIUNA), Chemistry Department, National University of Costa Rica, Heredia 3000, Costa Rica

³Department of Wood, Cellulose and Paper, University of Guadalajara, Tonalá 450110, Mexico

⁴Department of Bioproducts and Biosystems, School of Chemical Engineering, Aalto University, Espoo 00076, Finland

Correspondence should be addressed to Belkis C. Sulbarán-Rangel; belkis.sulbaran@academicos.udg.mx

Received 13 October 2018; Revised 2 December 2018; Accepted 27 December 2018; Published 12 February 2019

Academic Editor: Zhonghua Yao

Copyright © 2019 Hasbleidy Palacios Hinestroza et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

The bagasse of *Agave tequilana* Weber is one of the most abundant agroindustrial wastes in the state of Jalisco. However, at the present time, there is no technical use for this waste, and its high availability makes it an environmental problem. The objective of this research was to take advantage of this waste and give it an added value to be used in the elaboration of advanced materials. In this sense, the agave bagasse cellulose was obtained using an organosolv method. To obtain the nanofibrils, the cellulose was passed through 6 cycles of a microfluidizer. The material was classified by FTIR, confirming the presence of the functional groups (O-H, C-H, C-C, and C-O-C), characteristics of cellulose, and the elimination of hemicellulose and lignin present in agave bagasse without treatment. The X-ray diffraction technique allowed the determination of the degree of crystallinity of the cellulose nanofibers, which was 68.5%, with a negative zeta potential of -42 mV. The images from the atomic force microscopy helped for the observation of the degree of fibrillation in the cellulose, and with the software ImageJ, the average diameter of the nanofibers was determined to be 75 ± 5 nm with a relatively uniform length of 1.0 – 1.2 μm . Finally, by means of thermogravimetric analysis, it was found that the obtained cellulose nanofibers (CNFs) supported high temperatures of thermal decomposition, so it was concluded that due to the diameter of the fibrils, the high resistance to pressure, and elasticity, the nanofibrils obtained in this investigation can be used in the elaboration of advanced materials.

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 280 pm^{-99} , while the extractable compounds in water with 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 chlorite (NaClO_2) 80% w/v, boric acid (H_3BO_3) 99.5% w/v, acetic acid (CH_3COOH) 99% w/v, and nitric acid (HNO_3) 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 NaClO_2 (80% w/v) and CH_3COOH (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 H_3BO_3 (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 with 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 degree of crystallinity of the cellulose nanofibers was determined by X-ray diffraction (XRD, Empyrean). The degree of crystallinity of cellulose was calculated using the Segal method [24]. This method is based on the relative intensity of two diffraction peaks of the X-ray spectrum using the following equation:

$$IC = \frac{I_{002} - I_{AM}}{I_{002}} * 100, \quad (1)$$

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 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 CNF thermal stability was determined by thermogravimetric analysis, using TA Instruments Q500 (TGA) equipment, in a temperature range between 50 and 800°C at 20°C/min under inert atmosphere.

3. Results and Discussion

The agave bagasse was air-dried and characterized using TAPPI standard methods. The results are shown in Table 1. In general, the composition of the agave bagasse corresponds well with values reported elsewhere for cellulose content (44.5%). With the same material, other authors [3, 11, 25, 26] have reported the values of 44.5%, 54.60%, 49.43%, and 64.9% respectively. On the contrary, the ash content obtained (2.8%) was lower than that reported in the literature (5.3%) from fibers of raw materials without cooking [20]. The large cellulose content makes it a suitable raw material for producing cellulose nanofibers.

In general, it can be considered that the differences between the percentages obtained in this research, and the results reported elsewhere, are basically due to the origin of the agave bagasse fibers and the pretreatment that the fibers were subjected to during the respective industrial processing. Some authors indicate that the composition of the plant species also depends on the type of pretreatment and operational factors such as temperature, reaction time, acid concentration, and particle size [9, 21]. In addition, other

influencing factors include the age of the plant at the time of harvesting and agronomical crop conditions, among others.

The morphology of the agave bagasse fibers without treatment and treated with the organosolv method is appreciated in the SEM images (Figure 1). After the decomposition of the lignocellulosic matrix, the microfibrils of cellulose are more accessible to obtain nanofibrils due to the elimination of noncellulosic components [27]. Figure 1(b) also indicates that the microfibril matrices become twisted after the organosolv treatment. Recently, this same observation was reported in an investigation [3].

The composition of the cellulose fibers treated with organosolv was confirmed through FTIR (Figure 2). The following peaks were observed: OH: 3335 cm^{-1} ; CH: 2895 cm^{-1} ; CC: 1495 cm^{-1} ; C=O, CC and CO: 1140 and 1060 cm^{-1} ; and COC: 905 cm^{-1} , which are the characteristics of cellulose. In addition, when comparing the three spectra, it can be seen that when the cellulose is not bleached, in the control, the following signal is also observed (C=O: 1735 cm^{-1}), which is associated with acetyl and aldehyde-carbonyl groups attributed to the presence of hemicellulose and lignin. Moreover, the stretching (C=C: 1630 cm^{-1}) characteristic of the benzene ring of lignin is also observed [2]. When the cellulose fiber is bleached, these last two signals were gradually decreased and became residual lignin [9].

The bleached agave cellulose nanofibers were obtained after 6 consecutive cycles in the microfluidizer. To analyse their morphology, the fibrils were observed by means of AFM (Figure 3). Nanosized fibrils can be seen with a width (ImageJ) of 75 ± 5 nm and relatively uniform sizes. The length was in the range 1.0–1.2 μm , and an entangled and self-aggregated morphology was observed, given the presence and interaction between the long fibrils.

It is worth noting that, after six cycles through the microfluidizer, the length of the agave cellulose fibers was reduced and an important portion of these became nanofibrils, as seen in the AFM image (Figure 3 and Table 2). Nanofibrils of different lengths were noted, and when averaging their size, it was evident that these values were similar to those reported elsewhere [28]. Recently, cellulose nanofibers with an average length of 85 nm were reported [26], which was obtained by high-pressure homogenization.

The value of the zeta potential indicates the degree of repulsion between adjacent particles in dispersion and is a measure of the stability of the suspension [29]. A greater zeta potential correlates with a more hydrophilic character. It was observed that the zeta potential of the analysed samples was negative and that this increased as the size of the fiber decreased (Table 2). These negative values indicate that the cellulose nanofibers are stable because the structure of the cellulose is oriented [12, 29]. It has been reported that cellulose in neutral water suspensions has a zeta potential with negative values from -20.72 to -41.32 mV [30]; and recently for agave cellulose nanofibers, a zeta potential was reported of -32.5 mV [12], this being lower than that found in this investigation, which was -42 mV, indicating that the obtained nanofibers were more stable.

TABLE 1: Chemical composition of the agave bagasse.

Ash (%)	Acetone extractables (%)	Water extractables (%)	Lignin (%)	Cellulose (%)	Hemicellulose (%)
2.8 ± 0.5	3.6 ± 0.8	3.7 ± 0.8	20.1 ± 1.5	44.5 ± 2.5	25.3 ± 3.4

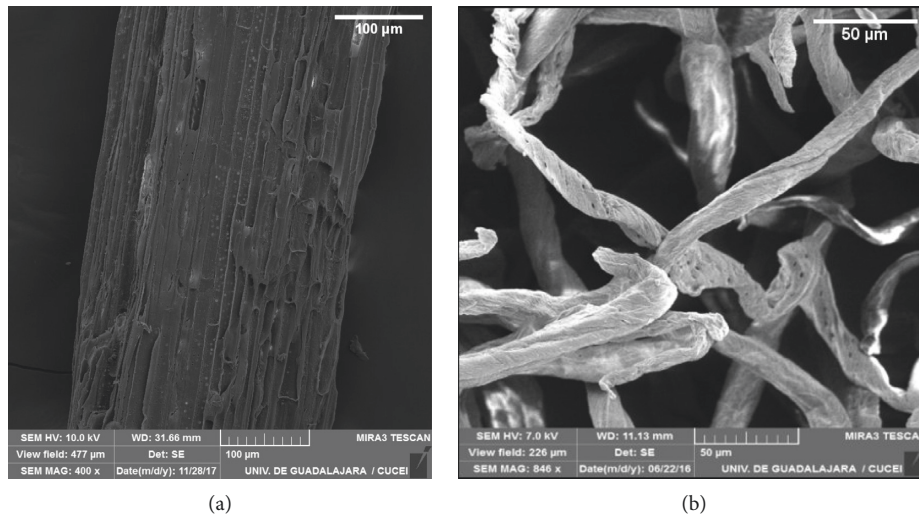
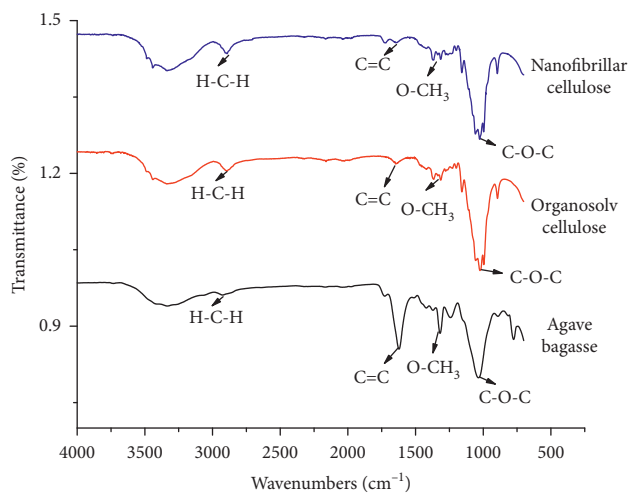
FIGURE 1: Morphology of agave cellulose fiber: (a) without treatment 100 μm ; (b) treated with organosolv method 50 μm .

FIGURE 2: FTIR spectra of agave bagasse fibers, cellulose obtained by the organosolv method, and nanofibrillar cellulose.

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

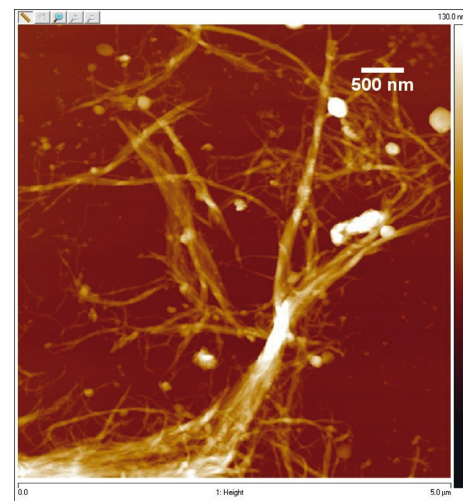


FIGURE 3: AFM image of cellulose bagasse nanofibril 100 nm.

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°C 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.

TABLE 2: Crystallinity index, ζ potential, and fiber size of the cellulose and nanocellulose.

Samples	Crystallinity index (%)	Zeta potential (mV)	Fiber size	
			Length	Width
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

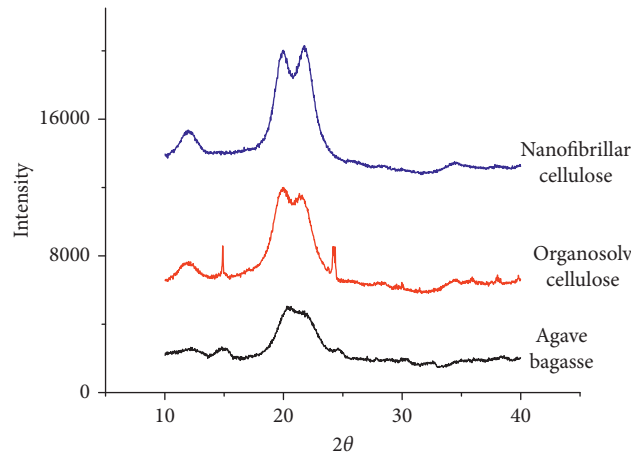


FIGURE 4: XRD spectra of agave bagasse fiber, organosolv cellulose, and nanofibrillar cellulose.

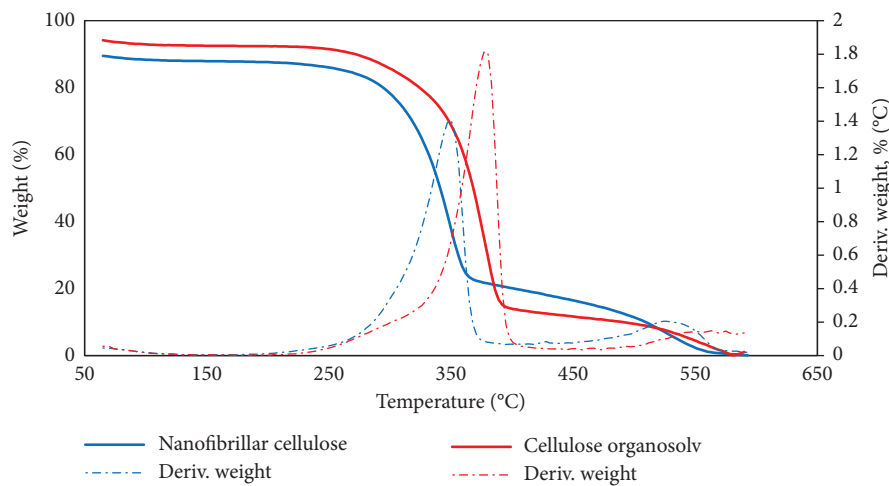


FIGURE 5: TG curves of organosolv cellulose and nanofibrillar cellulose.

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

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.

Acknowledgments

Financial support for this work was provided by the National Council of Science and Technology of Mexico (CONACYT) and Program for Professional Teacher Development (PRODEP). The authors thank Laboratory POLIUNA in the National University of Costa Rica and Aalto University in Finland for experimental support.

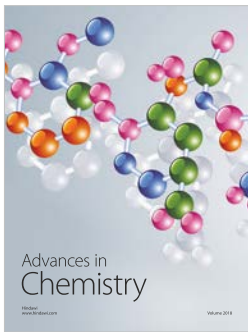
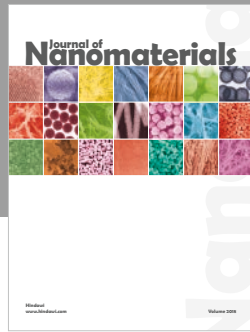
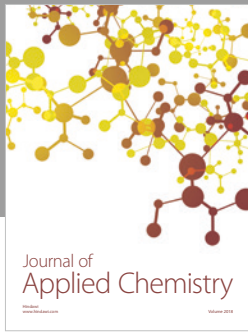
Supplementary Materials

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. Cyras, and A. Vázquez, "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. Alemán-Nava, I. A. Gatti, R. Parra-Saldivar, J.-F. Dallemand, B. E. Rittmann, and H. M. N. Iqbal, "Biotechnological revalorization 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-Saldivar, 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 consortium for bioconversion of lignocelluloses 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-Álvarez, 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. Rodríguez-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. Nantnarphirom, 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. Filpponen, A. Rodríguez, J. Laine, and O. J. Rojas, "Valorization of residual empty palm fruit bunch fibers (EPFBF) by microfluidization: production of nanofibrillated cellulose and EPFBF nanopaper," *Bioresource Technology*, vol. 125, pp. 249–255, 2012.
- [17] B. Nasri-Nasrabadi, M. Mehra, 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 electrospun 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 poly(lactic acid) to conform new composites," *Industrial Crops and Products*, vol. 71, pp. 44–53, 2015.
- [21] TAPPI, Technical Association of the Pulp Paper, *Industry 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 gradiented 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 microfibrils 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.



Hindawi
Submit your manuscripts at
www.hindawi.com

