Obtaining Soluble-grade Cellulose Pulp from *Agave tequilana* Weber var. Azul Bagasse

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Natural lignocellulosic biomass is a valuable feedstock for soluble-grade cellulose (α-cellulose > 90%) with advantageous features such as abundance, high strength, rigid structure, low weight, and it is biodegradable. The bagasse from *Agave tequilana* Weber var. azul is the main agroindustrial waste from the tequila industry and poses an environmental threat. However, due to its high cellulose content, *Agave tequilana* bagasse is an excellent candidate for the extraction and utilization of its components. In this study, alkaline cooking, followed by five stages of bleaching, was employed to obtain soluble-grade cellulose pulp. The obtained soda pulp material had a Kappa number (KN) of 23 ± 3, viscosity (μ) of 11.53 to 10.18 cp, degree of polymerization (DP) of 830 to 750, brightness of 87.5%, and percentage of alpha-cellulose (α) of 81 ± 3 at 94 ± 3%. The obtained material was characterized using X-ray diffraction, Fourier-transform infrared spectroscopy, and scanning electron microscopy to compare its properties with published information. The waste of *A. tequilana* Weber var. azul produced pulp with a high percentage of alpha cellulose and a high crystallinity degree. This methodology is novel and simple for the production of soluble-grade cellulose pulp, a raw material for the production of cellulose nanocrystals.

*Keywords:* *Agave tequilana* Weber var. azul; Bagasse; Agroindustrial waste; Bleached pulp; Sustainability

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**INTRODUCTION**

The bagasse of *Agave tequilana* Weber var. azul is a fibrous, highly moist feedstock (80%), mainly composed of cellulose (70 to 80%), lignin (15 to 20%), and hemicelluloses (5 to 10%) (Ponce-Reyes *et al.* 2014). Because the bagasse is hard and difficult to transport, use, and process, it is considered a waste that is being disposed in inadequate areas, causing several environmental concerns such as changes in soil fertility and phytosanitary problems. In addition, the waste is rich in residual sugars, which makes it an ideal substrate for microbial fermentation and pest proliferation, especially insects and rodents, causing an extremely bad odor and potential health hazards (Rodríguez Macías *et al.* 2010; Salcedo Luna *et al.* 2010; Satyanarayana *et al.* 2013). Although there are alternative uses to bagasse, such as a composting material and as an element in the manufacture of handicrafts, fill mattresses, and production of light bricks, the bagasse waste generated is much higher than its alternative uses (Granados Sánchez 1993; Colunga García-Marín 2007).
On the other hand, the increasing demands for greener technologies and products have led researchers to study the feasibility of biodegradable and renewable raw materials obtained from a variety of botanical species and agroindustrial wastes (Ramires and Dufresne 2011). In this sense, the present work aims to re-utilize the bagasse of Agave tequilana Weber var. azul as a raw material for the production of soluble-grade cellulose, in a way that decreases its environmental impact and contributes to the preservation of the environment. Besides, tequila bagasse possesses several advantages in comparison to alternative lignocellulosic feedstock, as it is considered a waste material with a higher content of cellulose and α-cellulose (Iñiguez-Covarrubias et al. 2001; Balam-Cocom et al. 2006).

Cellulose is the most abundant renewable plant material produced on earth, with an estimated annual production of $7.5 \times 10^{10}$ tons (Bolio-López et al. 2011; Carchi 2014). Cellulose is approximately one-third of plant tissue as it is widely distributed in the plant cell walls, particularly stems, trunks, and woody parts. Cellulose is exclusively composed of β-glucose molecules and its chemical formula is $(\text{C}_6\text{H}_{10}\text{O}_5)_n$. It is a solid, white, odorless, and tasteless polymer, and insoluble in water, alcohol, and ether (Kalía et al. 2011; Mariano et al. 2014). In most natural fibers, the holocellulose content, which comprises the cellulose and hemicellulose, is in the range 60 to 80%, complemented with 5 to 20% of lignin (phenolic reticulated polymer responsible for thickening and stiffening of the plant), and up to 5% of pectin and water. In addition, the fibers contain low percentages of wax, also known as extractives or low molecular weight sugars (Morán et al. 2008).

Cellulose pulp can be obtained from mechanical, chemical, thermo-mechanical, chemo-mechanical and biological processes from wood or other plants (natural fibers). The traditional pulping process renders relatively low yields of approximately 40 to 60% of the original material for plant leaves (Jiménez Muñoz et al. 2014). There are three standard chemical processes for making pulp, the sulfite, kraft, and soda processes and the yield of pulp is dependent on the chemical composition of the source, ranging between 40 and 55 for different kinds of wood (May 1980). Soluble-grade cellulose pulp is cellulose without contaminant molecules, such as lignin, and hemicellulloses, and it is commonly obtained through a series of processes consisting of pulping followed by bleaching. The whole process consists of several stages, where an isothermal bath at constant temperature in order to hydrolyze the hemicelluloses and facilitate its extraction (60 to 80 °C) for a certain period of time (2 to 4 h) (Ponce-Reyes et al. 2014; Robles et al. 2015). The bleaching procedures are applied depending on the Kappa number (KN) obtained from the pulp under the TAPPI T236 om-06 standard (2006), which determines the lignin content of the pulp. A higher KN indicates a higher lignin content. Chlorine dioxide (ClO$_2$) is one of the bleach reagents used to separate and eliminate residual lignin and some sugars of low molecular weight from cellulose in the pulp (Robles et al. 2015). Other common reagents include sodium hydroxide (NaOH), which extracts the residual lignin from the pulp after the ClO$_2$ stage and removes the hemicellulloses; alternatively hydrogen peroxide (H$_2$O$_2$) can help to whiten cellulose by eliminating those residues that could not be extracted by the ClO$_2$ and NaOH stages of treatment (Casillas et al. 2019).

Second, it is necessary to evaluate the molecular chain degradation of cellulose, which is the soluble grade-cellulose, performing a viscosity test ($\mu$) and the degree of polymerization (DP) following the TAPPI T230 om-13 (2013) standard, the DP values for “technical cellulose pulp” are suitable in the range of 200 to 1000 (Peng et al. 2011). This procedure is performed prior and after the bleaching process to determine possible internal structure damage of cellulose caused by chemical
degradation. Besides, is also necessary to obtain the percentage of α-, β-, and γ-cellulose using the TAPPI T203 cm-09 (2009) standard. The α-cellulose is one of the molecular structural components that cannot be degraded via NaOH due to its high molecular weight. A degree of polymerization greater than 500 and α-cellulose content greater than 90% are the principal characteristics of dissolving pulp (Casillas et al. 2019). β-cellulose is the structural part with the lowest molecular weight, and possess an amorphous structure. However, β-cellulose is responsible for molecular chain flexibility. In contrast, gamma-cellulose is hemicelluloses and residual low molecular weight sugars that do not belong directly to the molecular structure but are attached to it physically, so they do not represent any direct property of cellulose (Libby 1968; Hubbe et al. 2008).

Many reports in the literature describe obtaining cellulose pulp from various types of agave. Leaves of Agave angustifolia were used to obtain cellulose pulp (cellulose 97% and the DP was not reported); this pulp was recycled to produce cellulose nanocrystals (CNC) (Rosli et al. 2013). Nanoparticles of Agave sisalana fiber were produced with bleached cellulose pulp; this cellulose had a DP of 1035 and 90% of cellulose (Siqueira et al. 2010). Bagasse of Agave tequilana Weber var. azul was the source to get CNF and CNC, in the process, the cellulose pulp was obtained, but α-cellulose and DP were not reported (Siqueira et al. 2010). Nevertheless, bagasse of Agave tequilana Weber var azul was used to obtain cellulose pulp, and among the reported characteristics of this pulp, there was a DP of 173 and 89% of α-cellulose (Lomelí et al. 2018). However, a method to obtain only pure soluble-grade cellulose, α-cellulose, without drastically decreasing of the DP value, from the bagasse of A. tequilana Weber var. azul has not yet been described.

In this sense, in the present work it is proposed to utilize agroindustrial wastes, bagasse of Agave tequilana Weber var. azul, as raw material for the production of soluble-grade cellulose by means of a novel methodology consisting of a chemical cooking followed by a five-stage bleaching process, suggesting the use of this material as a possible solution to environmental problems.

**EXPERIMENTAL**

**Materials**

Bagasse from Agave tequilana Weber var. Azul was kindly donated by Mundo Agave (Tequila, Mexico). Sodium hydroxide (NaOH, 97% purity), potassium permanganate (KMnO₄, 0.1 N, 97% purity), sulfuric acid (H₂SO₄, 97% purity), potassium iodide (KI, 97% purity), sodium chlorite (NaClO₂, 97% purity), hydrochloric acid (HCl, 37% purity), and hydrogen peroxide (H₂O₂, 30% purity) were acquired from Golden Bell Reactivos (Zapopan, México). Anthraquinone (97% purity) and cupriethylenediamine (CED, 1 M, 97% purity) were obtained from Sigma-Aldrich (Toluca, México). Potassium dichromate solution (K₂Cr₂O₇, 0.5 N, 96% purity) and ferrous ammonium sulfate [(NH₄)₂Fe(SO₄)₂·6H₂O, 0.1 N, 9% purity) were obtained from HYCEL Reactivos Químicos (Zapopan, México). Sodium thiosulfate (Na₂S₂O₃, 0.1 N, 96% purity) was from Karal Reactivos Analíticos (León, México).
Methods

Obtaining and characterizing cellulose pulp

The bagasse of *Agave tequilana* Weber var. azul was washed until the mellow was removed and the sugars and parenchymal cells were not detected, and then dried at room temperature. Next, the bagasse was cooked using a solution of 20% NaOH, 0.1% anthraquinone, at a hydromodule ratio of 5:1, for 90 min at 170 °C in a rotating stainless steel reactor. The resulting pulp was washed and filtered using a stainless steel mesh (0.40 mm).

The characterization of the cellulose pulp was determined from the Kappa number (KN) under the TAPPI T236 cm-06 (2006) standard. The viscosity (μ) and degree of polymerization (DP) were determined under the TAPPI T230 om-13 (2013) standard. The amounts of α-, β-, and γ-cellulose were determined by the TAPPI T203 cm-09 (2009) standard. The reported values are the average of at least two independent measurements.

Scanning electron microscopy (SEM)

The morphology of the untreated fibers and treated fibers were analyzed via scanning electron micrographs for several samples, using a TESCAN MIRA 3 high-resolution microscope (Micra Nanotecnología, Ciudad de México, México) at 10 kV accelerating voltage and a working distance of 15 mm. The samples were coated with a sputter layer of gold beforehand (SPI-module sputter coater; Micra Nanotecnología, Ciudad de México, México).

Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra of the untreated fibers and treated fibers were measured with a spectrophotometer (Spectrum GX; Perkin Elmer, Guadalajara, México) using the attenuated total reflectance technique (ATR). The spectra were obtained from the average of 16 scans with a resolution of 4 cm⁻¹, in the frequency range of 4000 to 700 cm⁻¹.

X-ray diffraction (XRD)

X-ray diffraction of untreated fibers and treated fibers was completed using an X-ray diffractometer (EMPYREAN; PANanalytical, Ciudad de México, México) at 45 kV and 40 mA, with an angle of incidence of 5° to 70°, a step of 0.2 and a time per step of 30 s, using CuKα radiation in a Ni filter and a graphite monochromator. The percentage of crystallinity was calculated using the Rietveld method (Snyder 1993), with the MAUD program (University of Trento, MAUD version 2.1, Trento, Italia).

Obtaining soluble grade cellulose pulp

The cellulose pulp was subjected to a five-stage bleaching process consisting of: (1) the addition of chlorine dioxide until the concentration of ClO₂ was 15% of the KN value obtained just after the alkaline cooking ([ClO₂] = 0.15 KN), (2) alkaline extraction of residual lignin with NaOH, the NaOH concentration should be 60% of the ClO₂ concentration used in step 1 ([NaOH] = 0.6 [ClO₂]), (3) addition of 2.5% H₂O₂ and the addition of 2% NaOH in aqueous solution, (4) addition of 2% ClO₂ with a consistency of 10% m/v in an isothermal bath at 60 °C for 1 h, and (5) an alkaline extraction with 10% NaOH at 25 °C for 2 h for the elimination of hemicelluloses. In all stages, the pulp was washed, centrifuged, and homogenized.
**Soluble-grade cellulose pulp characterization**

After the bleaching process, the brightness percentage was determined under the TAPPI T452 om-08 (2008) standard, by making sheets for physical tests, under the TAPPI T205 SP-02 (2002) standard. The brightness was determined in a chromatic analyzer Elrepho 3000 series (Datacolor International, Lawrenceville, NJ, USA). The lightness value “L” and the color channels “a” and “b” from the chromaticity spectrum and the percentages of brightness at 440 and 460 nm of wavelength were obtained. The percentage of brightness was interpolated to 457 nm.

The viscosity and degree of polymerization were determined using the TAPPI T230 om-13 (2013) standard, and the percentage of α-cellulose was obtained according to the TAPPI T203 cm-09 (2009) standard.

**RESULTS AND DISCUSSION**

**Alkaline Cooking and Bleaching Process**

After pulp cooking, a yield of 40% of pulp in bagasse was achieved. After the bleaching processes, this value decreased to 30%, with respect to the amount of initial bagasse used. This effect was due to the elimination of hemicelluloses present in the pulp, which represent 15% of the total bagasse weight. Figure 1 shows the pulp before and after the bleaching treatment, where a change of color from brown to white is noticed.

![Pulp photographs: A) before and B) after the bleaching process](image)

For the unbleached cellulose pulp, a remarkably high KN of 23 ± 3 was obtained, indicating a high lignin content in the pulp (Iakovlev et al. 2014; Jaouadi et al. 2015). The analysis of the viscosity and DP before and after the bleaching process yielded values of 11.53 cp, 830, and 10.18 cp, 750, respectively. The decrease in DP indicated that the molecular structure of cellulose did not suffer severe chemical attack during the bleaching process. The percentage of α-cellulose increased considerably after the bleaching process, from 81% ± 3 to 94% ± 3, indicating that soluble-grade cellulose was obtained.

Several authors have reported obtaining soluble-grade cellulose from the bagasse of *Agave angustifolia* and *Agave sisalana* by cooking with a 4% NaOH solution, at temperatures ranging from 70 to 80 °C for 2 h in a digester. Then, a bleaching treatment was performed using 1.7% (w/v) NaClO₂ from 70 to 80 °C for 4 h (Rosli et al. 2013). In this case, the authors used agave leaves, the most rigid part of the plant, and obtained a material with α-cellulose of 97%. In the current case, the
value was lower because the agave came from waste residues. In the same way, *Agave salmiana* soluble-grade cellulose was obtained with 5% NaOH using similar reaction conditions previously reported. The bleaching treatment consisted of a solution of 11% H$_2$O$_2$ at 50 °C for 90 min (Chávez-Guerrero *et al.* 2017); cellulose pulp without lignin and hemicelluloses was obtained from *Agave tequilana* Weber var. azul, after addition of 5 M NaOH at 80 °C for 3 h in a rotating digester (Ponce-Reyes *et al.* 2014). In other reports, 1.8 M NaOH (1:20 w/v) at 120 °C for 90 min was used (Robles *et al.* 2016), obtaining an approximate pulp yield of 40% by weight, using bleaching treatments with H$_2$O$_2$ (3 M), NaClO$_2$ (0.2 M), and ClO$_2$ (2 to 3% by weight) at temperatures between 60 and 75 °C for 2 to 3 h in an isothermal bath. In some cases, the addition of anthraquinone (AQ) helps NaOH penetrate the fiber structure and subsequently to more easily extract the lignin and low molecular weight sugars (Hubbe *et al.* 2008; Robles *et al.* 2015). In all these cases, the production of pulp with yields of α-cellulose higher than 90% have not been reported, with the exception of Rosli *et al.* (2013). The best purity has been 89%, but with DP values less than 300 (Lomelí-Ramírez *et al.* 2018). In this work, the authors combined the processes mentioned earlier to obtain soluble-grade α-cellulose with purity above 90%.

**Pulp Brightness Process**

Table 1 shows the values of the chromaticity parameters and the percentage of brightness for wavelengths of 440 and 460 nm, after extrapolating to 457 nm and averaging, as stipulated by the TAPPI T452 om-08 (2008) standard. A brightness value of 87.5% was obtained, similar to the results reported in the literature (Andrade and Colodette 2014; Jaouadi *et al.* 2015). The obtained results give the material a high-added value, as special chemical methods were not employed, in comparison to other reports, where a near brightness of 90% was reported.

**Table 1. Chromaticity Parameters**

<table>
<thead>
<tr>
<th>Sample</th>
<th>L</th>
<th>a</th>
<th>b</th>
<th>Brightness (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>92.1</td>
<td>-0.45</td>
<td>2.94</td>
<td>87.31</td>
</tr>
<tr>
<td>2</td>
<td>91.97</td>
<td>-0.44</td>
<td>2.72</td>
<td>86.64</td>
</tr>
<tr>
<td>3</td>
<td>91.37</td>
<td>-0.44</td>
<td>2.79</td>
<td>85.23</td>
</tr>
</tbody>
</table>

**SEM Analysis**

Figure 2a shows a scanning electron micrograph of the longitudinal section of the agave bagasse fiber, which looks similar to many vegetable fibers (Esmeraldo *et al.* 2010; Ferraz *et al.* 2011; Carvalho *et al.* 2010). The longitudinal arrangement of a large number of fibrils in tube form is easily observed, with diameters of approximately 200 μm. Material is mixed between the fibers, which could be lignin, hemicelluloses, and extractives. The latter could be amorphous waxes and low molecular weight sugars (Bessadok *et al.* 2008; Satyanrayana *et al.* 2013). The fibers showed a space between them, and in some cases, pores that function as channels that can increase and retain the volume of water (Rodríguez Macías *et al.* 2010; Singha and Rana 2010). Defects in the surface were also visible, demonstrating that it is not a homogeneous surface. Some shapes and structural parts of other fibers are not perfectly cylindrical, as commonly occurs during the sugar extraction process to which the agave bagasse is subjected during the process of the production of tequila (Bessadok *et al.* 2008; Robles *et al.* 2018). Figure 2b shows the micrograph of the pulp after alkaline cooking. The fibrils that were observed in Fig. 2a are already
separated, which indicates that the cooking process was completed successfully. Another important feature is that the fibers are no longer rigid, which means that the extractives and a large portion of lignin were removed. The fiber diameters were approximately $18 \pm 5 \mu m$, which was consistent with the reported values of previous works for different agaves. *Agave tequilana* showed fiber diameters from 16 to 45 \( \mu m \), *A. angustifolia* from 9 to 110 \( \mu m \), and *A. salmiana* from 20 to 30 nm (Rosli et al. 2013; Espino et al. 2014; Chávez-Guerrero et al. 2017). Figure 2c shows the micrograph of the bleached pulp. It can be seen that the fibrils were, in turn, composed of smaller fibers with diameters between $5 \pm 3 \mu m$. This indicated that residual lignin and hemicelluloses that remained after the cooking process were eliminated, as the fibers were less rigid than they had been before cooking (Fig. 2b). It can be understood that these small fibers contain the molecular structure of cellulose, where \( \alpha-, \beta-, \) and \( \gamma- \)celluloses are found. Similar works with plants from the same family as *Agave tequilana* demonstrated that an aggressive hydrolysis of bleached pulp must be performed to break the amorphous regions (\( \beta- \) and \( \gamma- \)celluloses) of the cell wall, leaving only the crystalline region (\( \alpha- \)cellulose) (Siqueira et al. 2011; Espino et al. 2014).

![Fig. 2. SEM micrographs of a) agave bagasse, b) pulp after the cooking process, and c) bleached pulp](image)

**FTIR**

Figure 3 shows the FTIR spectra of the agave bagasse fiber, the pulp obtained from the alkaline cooking, and the pulp obtained after the bleaching process. The agave fiber shows characteristic peaks belonging to cellulose and lignin, where bands of C-H bonds (2918 and 2851 cm\(^{-1}\): asymmetric and symmetric tension vibrations, respectively), C-H bonds (1318 cm\(^{-1}\) [crystalline band] (Xu et al. 2013; Robles et al. 2017).
2018), C-O bonds (1054 and 1030 cm\(^{-1}\)), C-O-C bond (1160 cm\(^{-1}\)), and a broad O-H maximum between 3600 and 3200 cm\(^{-1}\), as well as bands at 1335 and 1205 cm\(^{-1}\) were observed. A small peak at 1734 cm\(^{-1}\) corresponding to carbonyl groups (C=O) was attributed to the presence of acetyl ester and carbonyl aldehyde groups in hemicelluloses and lignins (Bessadok et al. 2008; Singha and Rana 2010). A band observed at 1610 cm\(^{-1}\) corresponds to the vibration of the aromatic ring (C=C stretching). Another vibration in the aromatic ring at 1500 cm\(^{-1}\) and the C=C deformation at 1450 cm\(^{-1}\) corresponds to lignin (Garside and Wyeth 2006; Xu et al. 2013). The signal at 1625 cm\(^{-1}\) apparently did not belong to the fiber. However, its presence has been associated with absorbed water and is commonly observed in lignocellulosic spectra (Tronc et al. 2007). At 898 cm\(^{-1}\), there is the vibration of the anomic carbon group of the carbohydrate, C1-H, and at 1635 cm\(^{-1}\) the band corresponds to adsorbed water (Bessadok et al. 2008; Contreras et al. 2010; Yu et al. 2013; Anwar et al. 2015).

The signals recorded for the bleached pulp and pulp were similar to the signals observed in the fiber spectrum. However, a decrease of the peaks referring to hemicelluloses and lignin was clearly seen in the pulp spectrum, while the peaks were no longer observed in the bleached pulp at 1733, 1507, and 1232 cm\(^{-1}\), which were attributed to \(\nu_{\text{C=O}}\) and \(\nu_{\text{C=C}}\) aromatic ring, and \(\delta_{\text{C-H}}\) from aromatics substituted, respectively. This indicated that during alkaline cooking large proportions of the lignin and hemicelluloses were eliminated, and after bleaching they were totally eliminated. Another important point was the so-called crystalline band (1318 cm\(^{-1}\)), which is the peak that shows the preferential directional arrangement of \(\alpha\)-celluloses. It was observed that this band was more defined in the bleached pulp spectrum, which was a possible indication of obtaining soluble-grade pulp. Another indication was the signal observed at 3334 cm\(^{-1}\), which was bigger in the spectrum of bleached pulp, and
this can be a symptom of crystallinity, because the hydrogen bond is less flexible in a crystalline structure (Dong et al. 1998; Montanari et al. 2005).

The areas of the peaks from the FTIR spectra were obtained by deconvolution and divided by the peak area located at 896 cm$^{-1}$ that was constant for all spectra, the values are shown in Table 2. The results corroborate the disappearance of some peaks (1733, 1507, and 1232 cm$^{-1}$), where hemicelluloses and lignin exhibit signals, as well as the decrease of other peaks that overlap with those observed in soluble-grade cellulose (1318, 1598, and 1033 cm$^{-1}$).

**Table 2. Area Ratio from FTIR Spectra ($A_\lambda/A_\lambda = 896$)**

<table>
<thead>
<tr>
<th>$\lambda$ (cm$^{-1}$)</th>
<th>Fiber</th>
<th>Pulp</th>
<th>Bleached Pulp</th>
</tr>
</thead>
<tbody>
<tr>
<td>3408</td>
<td>1.07</td>
<td>1.64</td>
<td>1.46</td>
</tr>
<tr>
<td>1733</td>
<td>1.17</td>
<td>0.14</td>
<td>0.03</td>
</tr>
<tr>
<td>1598</td>
<td>1.48</td>
<td>0.72</td>
<td>0.78</td>
</tr>
<tr>
<td>1507</td>
<td>0.52</td>
<td>0.28</td>
<td>0.03</td>
</tr>
<tr>
<td>1318</td>
<td>0.93</td>
<td>1.57</td>
<td>1.57</td>
</tr>
<tr>
<td>1232</td>
<td>1.86</td>
<td>0.09</td>
<td>0.08</td>
</tr>
<tr>
<td>1033</td>
<td>3.81</td>
<td>3.78</td>
<td>3.51</td>
</tr>
</tbody>
</table>

**XRD**

The crystallinity in lignocellulosic materials is a characteristic attributed to the cellulose structure, which consists of polymeric glucopyranose chains arranged helically and strongly attached by hydrogen bonds (Tronc et al. 2007). Figure 4 shows the comparison of the diffractograms of the agave bagasse fiber, the pulp obtained from the alkaline cooking, and the pulp after the bleaching process. It was observed that the signals associated with cellulose were present in all cases, but with different intensity and broadening due to the amount of crystalline cellulose present in each sample and its different components. The widened signals were associated with the presence of non-crystalline particles. This can be seen in the agave fiber and in the pulp after cooking, in which the signals at 2$\theta$ = 14.63°, 16.85°, and 22.27° were wider and with lower intensity, showing the planes (1-10), (110), and (200). This was related to hemicelluloses or amorphous cellulose (Park et al. 2010; Poletto et al. 2013). In contrast, the bleached pulp had sharper signals and peaks at 2$\theta$ = 14.4° (101), 16.45° (10-1), 22.43° (002), and 34.92° (040).

It was observed that the planes were not the same and their angles changed, showing a clearer and weaker signal than the other samples. This indicates a higher crystallinity of the pulp and that $\alpha$-cellulose was obtained (Tronc et al. 2007; Robles et al. 2015). The diffractograms also illustrate the elimination of impurities and increase in crystallinity as the cooking and bleaching processes were completed, which was corroborated by the increase in the peak intensity.

Using the Rietveld method, a crystallinity value of 79.2% was determined for the bleached pulp (Fig. 5). This value was relatively greater than the values found in literature, which were in the range of 51 and 78.5% (Pires de Figueiredo and Furlan Ferreira 2014; Lomelí-Ramírez et al. 2018). This, together with the other characterization techniques, proves that soluble-grade cellulose was obtained.

This cellulose presents unique applications and high value, as it can serve as the raw material for the production of cellulose nanocrystals, a nanomaterial that is being used in various areas of research such as medicine, chemistry, biology, and engineering, among others.
Fig. 4. XRD diffractograms of agave fiber, pulp, and bleached pulp

Fig. 5. XRD of the bleached pulp analyzed via the Rietveld method
CONCLUSIONS

1. Cellulose pulp was successfully obtained with a Kappa number (KN) of $23 \pm 3$. The yield of alkaline cooking was 40%, the $\alpha$-cellulose was $83 \pm 3\%$, viscosity was 10.18 cp, and the degree of polymerization (DP) was 830. In the bleaching process, approximately 10% of the total pulp was lost.

2. Soluble-grade cellulose had an $\alpha$-cellulose value of $94 \pm 3\%$, a brightness of 87.5%, a viscosity of 11.53 cp, and a DP value of 750. With the bleaching process, the value of $\alpha$-cellulose increased, while viscosity and DP were not considerably affected.

3. The SEM micrographs showed that the agave fibers were gradually separated until they reached the soluble grade of cellulose pulp. In FTIR spectrograms, the disappearance of peaks attributed to lignin and extractives were observed, thereby contributing to the fact that the bleaching process eliminated these pulp components.

4. The high crystallinity was due to the fact that the $-\text{OH}$ peak area of the pulp increased. The XRD analysis indicated the elimination of impurities and the increase in crystallinity as the cooking and bleaching processes were performed. Using the Rietveld method, a crystallinity of 79.2% was determined for the bleached pulp. These results indicate that soluble-grade cellulose pulp was successfully obtained.

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