Reviewer 2: comments and Suggestions for Authors

The authors used Opuntia ficus-indica L. Miller as a new source for extracting cellulose by oxidation of the lignins. The extracted cellulose was identified by FTIR, NMR, X-ray diffraction, TGA-DTG, DSC and SEM. The manuscript was well organized. However, some points need to be clarified in the manuscript before being recommended to be published in the journal. Therefore, I would like to suggest a minor revision. If the requested points listing bellow could be well addressed and supported in revised version, the publication will be further considered:

Reply: Thanks to the referee comments. Several points in the manuscript was modified to better understanding of this study. Correction of the language was done. The change is highlighted in the yellow in the manuscript text".

1. Did you use any analytical method to confirm the complete elimination of lignin?

Reply: The quantification was performed by Klason methodology.

*P. Klason, Svensk Papperstidn., 26, 319 (1923)

2. Better to include the DSC and TGA thermograms of holocellulose in the manuscript for comparison.

Reply: Thanks for the suggestions. We understand the importance in include the DSC and TGA of holocellulose. However, there is a trend towards cost reduction (energy, materials, etc.), thus the researches are using data available in the literature for this. In this sense, we have selected manuscripts with this information and are published in peer-reviewed journals and great impact factor. However, this fact not decrease the innovation of manuscript that is obtaining of cellulose from Opuntia ficus-indica L. Miller, never made previously.

Please, see the highlighted changes bellow.

Thermogravimetry

"Figure 3 shows that the highest extent of mass loss was observed at 340 °C, which is very similar to the temperature found by Jonoobi (2011) [33] for purified cellulosic fibers and by Harini et al. (2018) [3] for cellulosic fibers extracted from banana peel."
Previous report on the TGA-DTG curve for holocellulose [34] that a mass loss occurred around 220 °C due to the thermal decomposition of the polysaccharide. On the other hand, lignocellulosic materials exhibited decomposition steps that start within the 200-260 °C range, which are attributed to the thermal depolymerization of hemicellulose or pectin, whose processes are not observed in purified cellulose [35].

Differential scanning calorimetry

“No characteristic events of vitreous transition (Tg) are observed. According to Almeida (2009) [38], such events may not be observed with some polymers such as cellulose where the decomposition takes place right before or parallel to the glass transition temperature, which may result in masking of such event.

As previously demonstrated by Miao et al. (2016) [39], the thermal profile of holocellulose comprises two exothermic events: the first one occurring within the range of 220-315°C (degradation of hemicelluloses) and the second around 353°C (decomposition of the cellulose fraction). These results corroborate with our finding and might indicate the purity of the analyzed cellulose.”

3. In FT-IR results, authors mentioned that the absence of bands between 1850-1650 cm−1 244 in the spectrum of cellulose confirms the disappearance of the 245 carbonyl groups in the sample. But there is not much difference in the FT-IR spectra of cellulose and holocellulose. Please give the magnified spectra in the region between 1850 and 1650 cm-1 to confirm the disappearance of peaks between this region.

Reply: Thanks for the comment. We highlight the absorption band in 1737 of carbonyl group (C=O) in figure 6. This band is not present in the cellulose spectrum, indicating the disappearance of the hemicelluloses. In this way the synthesis step was performed successfully, corroborating with the events demonstrated in the XRD after the alkaline treatment. The original data is being sent attached for better viewing of the band.

The spectrum of native fiber is showed in manuscript of Wan et al., 2019. The absorption bands are same to holocellulose results demonstrated in our work.


Please, see the changes below.

“The FT-IR spectrum of cellulose (Figure 6) shows a gradual decrease in the intensity of the bands related to -OH (3650-3207 cm−1), CH (2920 cm−1), the absorbed water (1634 cm−1) and C = C (1517 cm−1), which corroborates the elimination of lignans from holocellulose [50]. In addition, the absence of band in 1737 cm−1 in the spectrum of cellulose confirms the disappearance of the carbonyl groups (hemicelluloses) [53], corroborating with the events demonstrated in the XRD after the alkaline treatment.

Finally, the decrease in the intensity of the band at 1152 cm−1 corroborates the purity of the cellulose obtained from Opuntia ficus-indica L. Miller [54, 55].
Figure 6. FT-IR spectra of holocellulose and cellulose.