

(Introduction)

Chloe: Good morning everyone. Panelists, advisers, and fellow researchers. My name is Annjela Chloe Alfonso,

Lady: I am Glenice Visha Torres,

Hugo: And I am Hugo Molina,

Chloe: And we are the presenters of the study: Recovery and evaluation of cellulose from agroindustrial residues of corn, grape, pomegranate, strawberry-tree fruit, and fava.

(Statement of the Problem)

Lady: Wood pulp remains the most popular source of cellulose due to its abundance and cost-effective extraction which enable large-scale production. However, the processes involved entail several environmental impacts, namely forest devastation and consequent contribution to global warming, which have driven researchers to seek environmentally friendly and biocompatible materials as sustainable alternatives to wood-derived cellulose (Pennells et al. 2020).

(Objectives)

Hugo: Therefore, the objectives of this study are: To offer an attractive alternative to wood pulp, from the use of non-wood cellulosic biomass sources, such as agricultural and industrial wastes.

Chloe: To offer inexpensive, readily available, renewable, and promising cellulose and fibers from non-wood sources, as these have lower lignin and higher hemicellulose contents compared to forestry materials, leading to lower energy and chemical consumption in delignification and purification processes.

Lady: To offer significant opportunities to convert biomass resources into valuable raw materials, considering the low economic value of the existing applications and the high value of potential cellulose products there is.

Hugo: To further boost research on sustainable biomass procurement, and to further promote circular economy practices to achieve environmentally sustainable economic growth, as the pulp industry follows the current trend of pursuing sustainable practices.

(Significance)

Chloe: In the upcoming years, the demand for cellulose is expected to exceed the available supply, considering the continual research that has led to the development of novel cellulose-based products, particularly in the food and biomedical fields (Hindi 2017; Abdul Khalil et al. 2020).

(Scope)

Lady: Hence, the focus of this study was to evaluate the cellulose isolation suitability from renewable agroindustrial residues through following a multi-step solvent extraction procedure. The isolated cellulose samples were characterized and compared to wood-derived microcrystalline cellulose, considering their further application in the biomedical field as a reinforcing material of composite scaffolds.

(Methodology)

Hugo: Now, onto the methodology of the study. The process started with Raw Material Preparation. The gathered ripened agricultural and industrial wastes were cleansed with tap water. The fruits were cut into pieces, and the arils were extracted from the peels through the use of a grape stem removing machine. The peels were then stored at a temperature of 20°C until further processing.

Chloe: Then followed the Multi-Step Cellulose and Lignin Isolation Process, which is schematically represented in the screen. As lignin acts as the “glue” that connects cellulose and hemi-cellulose, the lignin was extracted during the extraction process of the cellulose, as well.

Lady: This process started with Dewaxing. In the first fractionation step, the peels were dewaxed using a 2:1 (v/v) toluene-ethanol solvent mixture and a solid-to-solvent ratio of 1:20 (w/v) in a Soxhlet apparatus for six hours. This was then followed by the rinsing of the dewaxed peels with ethanol for under 30 minutes under vacuum filtration, and were then dried in an oven at a temperature of 105 °C until the material reached the persistent weight.

Hugo: This was then followed by the Bleaching of the dried dewaxed peels for delignification purposes using a 1:1 (v/v) mixture of acetate buffer and sodium chlorite aqueous solution (17 g/L) for 4 hours, at 100 °C, using a solid-to-solvent ratio of 1:40 (w/v). This was the step in which the lignin content from the peels were extracted. After the removal of lignin, the delignified celluloses were rinsed with a solution of sodium hydrogen sulfate (20 g/L), deionized water and ethanol under vacuum filtration. Successively, the rinsed and delignified celluloses were dried at the temperature of 105 °C.

Chloe: Next, Alkaline Treatment. As the Bleaching is executed, the researchers were able to extract holocelluloses, or the total polysaccharide fraction of lignocellulosic biomass: cellulose and hemicelluloses. The extracted holocelluloses then underwent alkaline treatment with a sodium hydroxide solution (175 g/L) in an orbital shaker for 45 minutes at room temperature and 60 rpm using a solid-to-solvent ratio of 1:50 (w/v). The crude cellulose fibers were then rinsed with 10% acetic acid, followed by deionized water.

Lady: Lastly, Acidic Treatment. After being dried at 105 °C, the crude cellulose fibers were put through an acidic treatment to remove impurities. A 10:1 (v/v) mixture of acetic acid (80%, v/v) and nitric acid (70%, w/v) were used at 120 °C in an oil bath for 15 min with a solid-to-solvent ratio of 1:40 (w/v). Again, the fibers were rinsed and dried, together with the extracted lignin from bleaching. This multi-step extraction process was repeated at least three times for each agroindustrial residue.

(Results)

Hugo: The moisture content of the agroindustrial residues at the end of the multi-step extraction procedure is reported in Table 1. The relative high moisture content of pomegranate peel (13.3%) may be related to its high content of soluble carbohydrates (Gullón et al. 2020), that are usually associated with higher hygroscopicity and difficulties during drying (Roos 1995).

Chloe: The percentage of mass removed at each extraction step was quantified and is presented in Fig. 2. to evaluate and compare the fractionation process for each agroindustrial residue. Based on previous studies (Sun et al. 2004; Morán et al. 2008; Maheswari et al. 2012; Madureira et al. 2018), it is expected that the fractions obtained at the end of steps I, II and III of the extraction procedure are rich in waxes/lipids, lignin and hemicellulose, respectively, leaving the purified cellulose isolated from each residue, after a final acidic treatment.

Lady: The cellulose yield obtained at the end of the multi-step extraction process for each agroindustrial residue, is shown in Figure 3. It was found that the mass losses during those operations varied between 0.2 and 0.4 g (dry basis) which may have possibly decreased cellulose yields from 2 to 4%.

Hugo: Figure 4, on the other hand, shows the FTIR spectra of the samples and of the wood-derived commercial MC obtained in an ATR-FTIR spectroscopy analysis. All spectra presented roughly the same peaks, indicating the presence of the same functional groups and confirming that the multi-step extraction process was successful in isolating purified cellulose from the residues.

Chloe: Next, Figure 5 shows the TGA and DSC curves for all cellulose samples obtained during the TGA and DSC analyses of cellulose, including the MC, and Table 2 reports the data obtained from these curves. The TGA curves of the MC and of the six cellulose samples obtained in this study were similar. A small weight loss was found in the range of 30–150 °C, due to the evaporation of low molecular weight compounds and water, which ranged from 4.1 to 5.3%, being of the same order of magnitude of the one observed for the commercial MC ($5.61 \pm 0.11\%$). No noticeable thermal events were observed after water evaporation confirming the effectiveness of the extraction procedure in removing hemicellulose and lignin from the residues.

Lady: Figure 6 then presents macroscopic and microscopic images of the cellulose samples obtained in the Morphological analysis of cellulose. The most evident differences were the color and brittleness which are mainly associated with the residual presence of lignin. The obtained celluloses can be classified from yellow to white, by the following order: grape stalk, fava pod, strawberry-tree fruit marc, pomegranate peel, corncob and corn husk. These observations suggest that the bleaching process was more efficient in the case of corncob and corn husk.

Hugo: Lastly, Figure 7 shows the SEM images under two different magnifications and reveals differences in the morphology of the two samples.

(Conclusion)

Chloe: In conclusion, this study suggests novel non-wood cellulosic biomass sources, thus contributing to the development of new trade opportunities for the involved processing industries, and following the current worldwide tendency of recycling agroindustrial residues.