FROM *CIRTUS AURANTIUM* PRUNING WASTE TO HIGH VALUE BIOMATERIAL: ISOLATION AND CHARACTERIZATION OF CELLULOSE FIBERS

DONIA FARHAT,[*,**] JIHEN TRABELSI,[***] RAMZI KHIARI,[****,*****]
FILOMENA BARRERIO[*] and NACEUR BELGACEM[*****]

[*]*CIMO, LA SusTEC, Instituto Politécnico de Bragança, Campus de Santa Apolónia, 5300-253, Bragança, Portugal*
[**] *University of Gafsa, Faculty of Sciences of Gafsa, Tunisia*
[***] *Higher Institute of Fashion of Monastir, University of Monastir, Tunisia*
[****] *Department of Textile, Higher Institute of Technological Studies of Ksar Hellal, Ksar Hellal 5070, Tunisia*
[*****] *University of Grenoble Alpes, CNRS, Grenoble INP, LGP2, Grenoble, F-38000, France*
✉ *Corresponding author: N. Belgacem, naceur.belgacem@grenoble-inp.fr*

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Valorizing agricultural pruning residue is a sustainable way to produce in-demand, bio-based materials and reduce environmental issues related to the disposal and incineration of agricultural waste. In this context, our work focuses on the valorization of *Citrus aurantium* pruning waste through cellulose isolation. First, the chemical composition of the biomass was evaluated according to TAPPI standards, revealing a high content of holocellulose and α-cellulose (60.7 ± 3.06, 37.2 ± 1.76, respectively), promoting its suitability as a cellulose feedstock. The isolation process consisted of alkaline delignification, followed by sodium chlorite and acetate buffer bleaching treatment. The resulting material was characterized by evaluating its crystallinity, thermal stability and morphology. The XRD analysis revealed a crystallinity index of 68%, indicating a well preserved crystalline cellulose structure. The TGA demonstrated a major degradation peak at 350 °C and low residual mass consistent with the measured ash content. Morphological study revealed well individualized fibers with average length of 0.926 mm and width of 22 µm, and a moderate fine elements fraction. These results confirm that *Citrus aurantium* pruning waste is promising as alternative lignocellulosic feedstock for cellulose production, highlighting its potential for applications in biobased material fields.

Keywords: pulping, cellulose fibers, *Citrus aurantium* pruning, characterization

INTRODUCTION

Citrus aurantium, also known as sour orange, is a wildly cultivated plant due to its importance in numerous fields. The essential oil produced from this plant's fruit, fruit peel, leaves and flowers has been gaining research attention mainly due to its benefits and applications in multiple industries. It is mostly known for its bioactive compounds,[1] antioxidant activities,[2] antidiabetic effects,[3] aromatherapy application to treat anxiety, stress, and depression[4] and use in the food industry.[5,6] To maintain good fruit, flower and extractives quality, a pruning step is applied at least once a year. This step results in fruit disease prevention, promotes flowering, and facilitates light penetration by offering a manageable canopy size, promoting yield and quality improvement. While the other components of the tree are highly consumed, the resulting wood is often discarded in landfills or incinerated, causing major land and air pollution problems. In Tunisia, *Citrus arantium* covers 1100 ha, while the average residual biomass of its pruning waste varies between 1 to 3 t/ha and 2.5 to 7.5 kg/tree.[7] The woody fraction resulting from this process is rich in lignocellulosic materials, with important potential in the production of high value added materials, such as cellulose.

Cellulose, the most abundant biopolymer on Earth, is broadly present in plant cell walls and is widely valued for its renewability, biodegradability, and remarkable versatility. Owing to these intrinsic properties, cellulose plays a pivotal role not only in conventional industries, such as textiles and

papermaking, but also in emerging high-value applications, including sustainable food packaging,[8–10] biocomposites,[11–13] water treatment technologies[14–16] and advanced functional materials.[17–19]

The isolation of cellulose from lignocellulosic biomass relies on the selective removal of lignin and hemicelluloses, which form a complex matrix surrounding cellulose microfibrils within the plant cell wall.[20,21] This process typically involves two main stages: (i) delignification, which disrupts the lignin–carbohydrate complex, leading to lignin solubilization and partial hemicellulose removal, and (ii) bleaching, which further eliminates residual lignin and hemicelluloses to yield purified cellulose.

In the present study, the raw material was subjected to a series of standardized procedures based on TAPPI methods to determine its chemical composition. Following confirmation of a significant cellulose fraction, the biomass underwent alkaline hydrolysis, coupled with sodium chlorite treatment in an acetate buffer medium, to obtain high-purity cellulose.

The isolated cellulose was subsequently characterized using complementary analytical techniques. X-ray diffraction (XRD) was employed to assess its crystallinity, while thermogravimetric analysis (TGA) provided insights into its thermal stability and degradation behavior. In addition, the morphological features of *Citrus aurantium*-derived cellulose were examined using Scanning Electron Microscopy (SEM) and Morfi analysis.

To the best of our knowledge, the valorization of *Citrus aurantium* pruning waste as a cellulose source remains largely unexplored. This study therefore aims to investigate the chemical composition of this agricultural residue and evaluate its potential for high-value applications. The upcycling of such underutilized biomass offers a promising pathway toward sustainable resource management and contributes to advancing the circular economy within the *Citrus aurantium* production chain.

EXPERIMENTAL

Materials

Citrus aurantium pruning waste (CPW) was collected in the Ariana region, located in northern Tunisia, during the pruning season in February 2024. The collected stems were washed, air-dried under natural conditions, and cut into pieces of 2 to 3 cm prior to pulping. Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), acetic acid (CH_3COOH), sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and sodium chlorite ($NaClO_2$) were purchased from Sigma-Aldrich and used without further purification.

Chemical composition of *Citrus* pruning waste

The chemical composition of CPW was studied following common protocols, mainly TAPPI (Technical Association of the Pulp and Paper Industry) standards. The extractives were evaluated according to solubility in hot and cold water (T207 cm-08), in ethanol–toluene (T204 cm 07) and in 1% sodium hydroxide (T212 om-07). The ash content is defined as the amount of remaining solid residue after biomass calcination at 600 °C; it was determined following the TAPPI method T211 om-07.

The Klason lignin, defined as the acid insoluble lignin, was studied by subjecting biomass to acid hydrolysis (H_2SO_4) and filtering the suspension. The obtained insoluble lignin is then dried and weighed, following the TAPPI method T222 om-06.

Holocellulose content was determined following the protocol proposed by Wise *et al*.[22] Biomass was placed in a flask containing distilled water, sodium chlorite and acetic acid were added, and the mixture was put under reflux for 1 h at 80 °C. The procedure was repeated until the material turned white. After filtration, the holocellulose obtained was repeatedly washed until neutralization and oven dried.

Alpha-cellulose content was determined according to the TAPPI standard T203 cm-99. In this method, a holocellulose sample was subjected to alkaline hydrolysis using a 17.5% sodium hydroxide solution under constant stirring. Five minutes after the start of the reaction, 40 mL of 17.5% sodium hydroxide was added to the mixture over a 10 min interval in equal portions. After 30 min from the beginning of the reaction, 100 mL of distilled water was added, and the beaker was maintained under reflux for an additional 30 min. At the end of the process, the pulp was filtered, washed, and oven dried. All analyses were carried out in triplicate to ensure reproducibility.

Cellulose fibre production

Cellulosic fibers from *Citrus aurantium* pruning waste were extracted following the procedures described by Razzak *et al*.[23] and Khiari *et al*.,[24] with slight adjustments. The extraction process consisted of two main stages:

alkaline delignification followed by oxidative bleaching, both aimed at isolating high-purity cellulose from the lignocellulosic source.

In the first stage, the raw CPW biomass was subjected to alkaline pulping to disrupt the rigid and recalcitrant structure of the plant cell wall. This treatment primarily induces lignin depolymerization through the cleavage of ether linkages (notably β–O–4 bonds), thereby increasing its solubility in the alkaline medium, while also promoting partial solubilization of hemicelluloses. The reaction was carried out in an autoclave using a 10 wt% sodium hydroxide (NaOH) solution at 160 °C for 2 h. After treatment, the solid fraction was separated by filtration and thoroughly washed with distilled water until neutral pH was reached, ensuring the removal of residual alkali and dissolved organic compounds.

In the second stage, the delignified fibers were subjected to a bleaching treatment to remove residual lignin and remaining hemicellulosic fractions, thereby enhancing cellulose purity and improving optical properties. The bleaching was performed using an aqueous solution containing 1.7 wt% sodium chlorite ($NaClO_2$) in an acetate buffer, which maintains the acidic conditions required for the *in-situ* generation of chlorine dioxide, the active oxidizing agent. The reaction was conducted at 80 °C under constant mechanical stirring for 2 h and repeated twice to ensure thorough purification. After each cycle, the fibers were filtered and extensively washed with distilled water until neutral pH was achieved. The final cellulose product was then oven-dried at 105 °C for 24 h until constant weight.

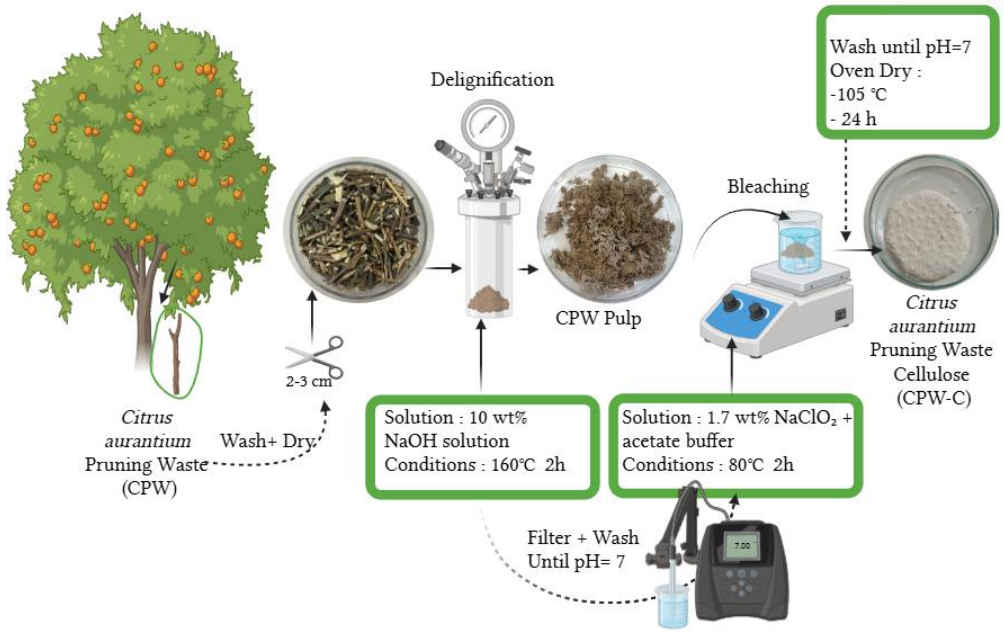

Figure 1: A schematic representation of different processes involved in cellulosic fiber extraction

Cellulose fiber characterization

Total charge determination by conductometric titration

The total charge of the raw material and the fibre were determined using the conductometric titration method described by Katz *et al*.[25] and Fras *et al*.[26] For each measurement, 1 g of the sample (on an oven-dry basis) was placed in an airtight titration vessel. Then, 500 mL of distilled water and 1 mL of 0.5 mol/L NaCl were added to achieve a final NaCl concentration of 1 mol/L during titration. Additionally, 0.5 mL of 0.1 mol/L HCl was introduced into the system.

The suspension was stirred constantly with a magnetic stirrer at 25 °C throughout the experiment. Titration was carried out by incrementally adding 0.1 mol/L NaOH with a precision burette at one-minute intervals; each drop contained 0.04 mL. Conductivity was continuously monitored using a conductometer.

Titration continued until a pH of approximately 10.5 was reached, allowing for an accurate endpoint determination through extrapolation. A blank titration was performed under identical conditions but without the sample to correct for background conductivity and potential impurities.

The total charge was determined by extrapolating the second and third linear regions of the titration curve to their intersection point. It was calculated according to Equation (1):

$$Total\ charge(\mu eq.g^{-1}) = \frac{C_{OH}-V_t}{m_{dry}} \quad (1)$$

where C_{OH} is the concentration of the sodium hydroxide solution, and V_t is the volume of NaOH consumed at the second equivalence point, and m_{dry} is the oven-dry mass of the sample.

Spectroscopic analysis: X-ray diffraction (XRD)

The crystalline structure of the CPW-C fibers was investigated by an X-ray diffractometer (D8-Advance Bruker AXS GmbH) at ambient temperature. This technique is used to evaluate the degree of crystallization and the arrangement of cellulose chains inside the fiber structure. The crystallinity index was calculated following the principle proposed by Segal *et al*.:[27]

$$C_1(\%) = 100x\frac{I_{002}-I_{Am}}{I_{002}} \quad (2)$$

where I_{002} is the intensity of the highest peak relative to the crystalline zone, I_{Am} – the intensity of the amorphous minimum.

Thermogravimetric analysis (TGA)

The thermal stability and decomposition behavior of the obtained cellulose were evaluated using thermogravimetric analysis (TGA). This method measures weight loss as a function of temperature. The dried cellulose fibers were heated from room temperature to 600 °C. Weight loss was recorded as the temperature increased.

Morphological analysis

The morphological aspect of CPW-C was evaluated by a Scanning Electron Microscope (SEM) employed to investigate the sample morphology at 15 kV acceleration voltage. Each sample was prepared by coating with gold via sputtering to prevent charging effects. The coated sample was then loaded on the stub placed with carbon tape before viewing. This method provides a direct visualization of fiber surface, texture and arrangement at high resolution. The Morfi laboratory image analyzer was also used to investigate the principal morphological parameters of the fiber suspension, providing information about fiber length, diameter and length of fine elements.[28]

RESULTS AND DISCUSSION

Chemical composition of *Citrus aurantium*

The chemical composition of CPW lignocellulosic biomass was systematically investigated following standard analytical protocols, and the results are summarized in Table 1. These findings provide a comprehensive insight into the potential of this biomass as a promising feedstock for holistic valorization strategies, including biorefinery applications and material production. The studied biomass exhibits a high holocellulose content of 60.67 ± 3.06%, which is significantly higher than that reported for papaya residues (46.8 ± 1.0%), garlic straw (59%), grape pomace (26.5%) and olive husk (47.6%) and comparable to that of vine stems (65.4%) and *Dendrocalamus giganteus* (65%) and other biomass sources ranging from 65% to 81%. Notably, the holocellulose content of CPW lies within the mid-range of values reported for other lignocellulosic biomass indicating a balanced polysaccharide composition, primarily cellulose and hemicelluloses, which can serve as precursors for high-value products, such as cellulose derivatives, nanocellulose, and bio-based materials.

The α-cellulose content, corresponding to the high-molecular-weight and alkali-insoluble fraction of cellulose, was determined to be 37.17 ± 1.7%. This value is comparable to those reported for persimmon tree pruning twigs (34.85 ± 0.48%), vine stems (≈35%), and other sources like grape pomace, corn husk, papaya residues, pineapple leaf, and olive husk, ranging between 19.3-35.6. The relatively elevated α-cellulose content indicates a significant proportion of well-ordered, crystalline cellulose domains within the biomass. Such characteristics are generally associated with enhanced mechanical strength, thermal stability, and resistance to chemical degradation. Consequently, this material appears particularly suitable for high-value applications requiring cellulose with a high degree of crystallinity, such as fiber production, film formation, and reinforcement in bio-based composite materials.

Furthermore, the α-cellulose fraction typically represents approximately two-thirds of the holocellulose content in lignocellulosic biomass, which is consistent with the value obtained in this study. This proportion is also commonly observed in wood, suggesting a comparable structural organization of the

polysaccharide matrix. The similarity in α-cellulose-to-holocellulose ratio indicates that the studied CPW exhibits a chemical composition and hierarchical organization analogous to that of conventional woody biomass. This resemblance reinforces its potential as an alternative lignocellulosic feedstock for material and chemical valorization processes.

The Klason lignin content of 21.23 ± 2.64% is within the typical range for woody and agricultural residues and is like for other citrus species, such as *Citrus lemon* pruning waste (25.1%) and *Citrus aurantium* fruit residue (19.7%). It is higher than the values reported for garlic straw (6.3%), corn residues (11.4-19%) and Red prickly pear peels (9%), while remaining lower than those of olive husk (48.4%) and other sources. This moderate lignin content provides a balanced lignocellulosic matrix, facilitating the separation of structural components through acid or alkaline hydrolysis. It also presents an opportunity for lignin valorization, enabling the extraction of aromatic compounds for chemicals, adhesives, or bio-based polymers.

The biomass shows significant solubility in hot and cold water, measured at 24.33 ± 3.06% and 18.33 ± 2.08%, respectively. These values exceed those of vine stems and *D. giganteus*, while being lower than for papaya residues. The high solubility reflects the presence of water-soluble compounds, such as free sugars, pectins, and organic acids, and the greater hot water solubility suggests thermal sensitivity of components like hemicelluloses. Additionally, these water-extractable fractions may contain phenolic compounds, providing potential antioxidant activity and possible recovery in biorefinery processes.

Solubility in toluene-ethanol was measured at 14.00 ± 1.00%, positioning CPW between papaya residues (22.2 ± 1.6%) and persimmon twigs (5.19 ± 0.13%). This moderate solubility indicates the presence of non-polar constituents, including waxes, fatty acids, and other hydrophobic compounds, which could be valorized as natural additives or used to enhance the hydrophobicity of cellulose-based materials.

The alkaline solubility (1% NaOH) was notably high at 44.67 ± 2.52%, reflecting a substantial fraction of alkaline-soluble components, including lignin, low molecular weight carbohydrates, and amorphous polysaccharides. This behavior indicates that delignification and hemicellulose removal via alkaline hydrolysis can be efficiently achieved, resulting in a purified cellulose fraction suitable for high-value applications.

Finally, the ash content of 4.10 ± 0.36% is comparable to vine stems (3.9%) and *Citrus lemon* pruning waste (3.5%) and lower than for red prickly pear (16.3%) peels and papaya residues (15.7%), suggesting a relatively low presence of mineral matter, such as potassium, calcium, and magnesium. This moderate ash content also indicates minimal contamination by soil or external impurities, making the biomass favorable for material and chemical conversion processes.

In summary, the chemical composition of CPW demonstrates a well-balanced lignocellulosic structure with high holocellulose and α-cellulose content, moderate lignin levels, significant water solubility, and manageable ash content. This composition reflects a stable and organized biomass matrix where cellulose and hemicelluloses are sufficiently abundant, while lignin remains at a moderate level, ensuring both structural integrity and processability. Such balance is particularly advantageous for lignocellulosic valorization, since it facilitates the efficient separation of components during chemical treatment, without degrading the fibrous structure.

Table 1
Chemical composition of CPW compared to other lignocellulosic biomasses

Biomass \ Composition	C.W. (%)	H.W. (%)	1%NaOH (%)	S.Tol. (%)	Ash (%)	K.L. (%)	Holo (%)	α.C (%)	Ref.
Citrus aurantium pruning waste (CPW)	18.3±2.1	24.3± 3.1	44.7± 2.5	14.0±1.0	4.1± 0.3	21.2±2.6	60.7±3.1	37.2±1.8	This work
Citrus aurantium fruit	5.6	7.9	14.9	-	2.7	19.7	81.2	-	[29]
Citrus lemon pruning	3	2.5	18.9	-	3.5	25.1	-	40.1	[30]
Red pickely pear peels	-	-	-	21.5	16.3	9	72	38	[28]
Prsimmon tree pruning	-	-	-	5.2±0.1	0.8± 0.1	27.5±0.6	-	34.8± 0.5	[31]
Garlic straw	-	-	-	3.2	10	6.3	59	41	[32]
Corn straw	-	-	-	-	-	19	73	45.5	[33]
Grape pomace	-	-	-	-	-	15.6	26.5	19.3	[34]
Sugarcane bagasse	-	-	-	0.7	0.7	23.8	75	44.6	[35]
Corn husk	-	-	-	-	-	11.4	69	29.3	[36]
Corn cob	-	-	-	3±0.8	-	12±0.8	68±5	40±0.3	[37]
Vine stems	8.2	13.9	37.8	11.3	3.9	28.1	65.4	35	[38]
Papaya residues	-	40.7±0.4	51±4	22.2±1.6	15.7	8.5±0.2	46.8±1.0	31.5±0.2	[39]
Dendrocalamus giganteus	6.2	7.8	24.5	6.3	-	27.7	65	-	[40]
Pineapple leaf	-	-	-	-	6.4 ± 0.2	27.3 ± 0.4	-	35.6 ± 1	[41]
Soft wood	0.5-4	1-5	8-10	-	0.2-0.5	25.3	63-70	38.9	[29]
Hard wood	0.2-4	1-8	12-25	-	0.2-0.7	18-26	79.1	42.7	
Tabacco stalk	-	-	-	-	2.4	27	70.2	42.4	[42]
Olive husk	-	-	-	9.4	4.1	48.4	47.6	24	[43]
Lupine husk	-	-	-	15±1.4	-	20±0.8	65±1.3	42±1.1	[44]

C.W.\H.W.: Cold\ hot water solubility, 1%NaOH: soubility in 1% NaOH solution, S.Tol.: Solubility in toluene-ethanol, Ash : Ash content, K.L.: Klason lignin, Holo: Hollocellulose, α.C: Alpha cellulose

Moreover, the presence of an important alpha cellulose fraction highlights the potential of CPW as a reliable source of high-quality cellulose suitable for fiber production and advanced applications. The moderate content of extractives further contributes to the feasibility of processing by limiting interference during purification steps, while offering opportunities for the recovery of high-value bioactive compounds.

These characteristics position CPW as a highly promising feedstock for sustainable biorefinery applications, including cellulose and nanocellulose production, lignin valorization, extraction of bioactive compounds, as well as the development of bio-based functional materials. The combination of compositional balance and resource abundancy positions this biomass as an attractive alternative to conventional lignocellulosic sources.

Pulping and bleaching of *Citrus aurantium* pruning waste fibers

Pulping efficiency was assessed in terms of cooking yield and bleaching yield, which were found to be 63.7% and 45.3%, respectively. The overall screening yield was 96.2%. Compared to other lignocellulosic resources, CPW exhibits competitive performance. The screening yield is notably important and comparable to almond residues (97.5-98%), and fig trimmings (98.3%),[45] indicating efficient recovery of high quality fibers. In terms of cooking and bleaching yields, CPW demonstrates well balanced results among various biomass groups. Within other agricultural residues, CPW outperforms almond shells (65.7%, 33.3%), fig trimmings (39.7%, 33.3%), almond trimmings (44.2%, 25.0%),[45] and bagasse (41.7%, 39.3%),[46] which displayed lower bleaching efficiencies, despite in some cases comparable or higher cooking yields. Compared to wood, such as beech wood (47.2%, 44.6%),[47] CPW achieved a significantly higher cooking yield, while maintaining a similar bleaching performance. In terms of comparison with grass biomass, bamboo exhibits the highest cooking yield (69.7%), but a lower bleaching yield (37.0%),[48] indicating a less favorable overall balance compared to CPW. In contrast, non-wood fibers, such as *Cannabis sativa* (49.5%, 45.0%),[49] *Hibiscus cannabinus* (51.8%, 42.8%)[50] and *Hibiscus sabdariffa* (58.5%, 49.3%)[51] show consistently high and balanced performance, however, CPW remains in the same range, particularly in terms of bleaching efficiency, and surpasses some of these materials in cooking yield.

Figure 2 provides a comparative overview of cooking yield versus bleaching yield for *Citrus aurantium* pruning waste (CPW) and various representative lignocellulosic biomasses, such as agricultural residues, grasses, and wood-based fibers. This comparison aims to position CPW within the broader landscape of fiber extraction efficiency, while considering the consistency of processing conditions across the datasets.

It is important to note that only a subset of the reported materials: almond trimmings, almond shells, fig trimmings, and CPW, were processed under comparable delignification and bleaching conditions. These samples allow for a direct, scientifically robust comparison of overall fiber recovery and process efficiency, since both the cooking and bleaching steps were conducted under similar operational frameworks. Within this coherent dataset, CPW clearly exhibits one of the most balanced performances, combining a relatively high cooking yield with an efficient bleaching yield. This indicates the effective removal of non-cellulosic components while preserving cellulose-rich fractions.

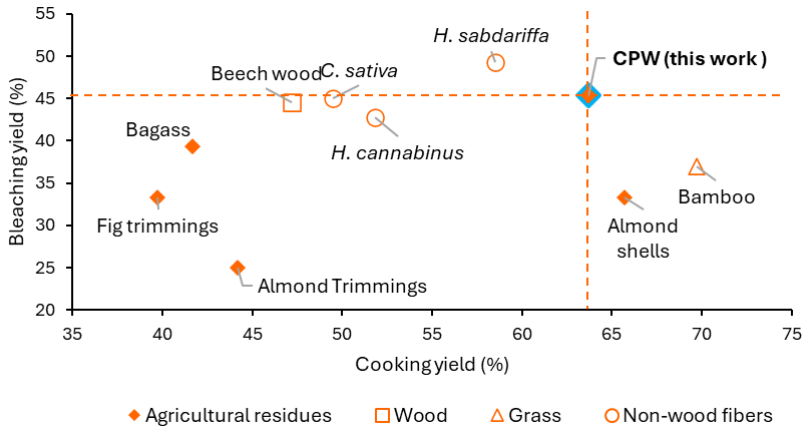

Figure 2: Comparison of cooking and bleaching yields of CPW with various lignocellulosic biomasses reported in the literature[45–51]

Other lignocellulosic materials, such as bagasse, bamboo, and non-wood fibers (*e.g.*, *H. cannabinus*, *C. sativa*, and *H. sabdariffa*), are included to provide broader context. However, it is important to emphasize that, although their cooking stages may be comparable in some cases, their bleaching protocols differ significantly in terms of chemical composition, severity, and processing conditions. Consequently, their bleaching yields cannot be strictly compared on an equal methodological basis. They should therefore be interpreted as indicative trends rather than direct performance benchmarks.

Despite the methodological differences, Figure 2 reveals a general trend highlighting CPW's position as a top performer, particularly regarding the combined efficiency of delignification and bleaching. CPW's proximity to regions with high cooking and bleaching yields demonstrates that its treatment strategy effectively preserves cellulose, while sufficiently removing lignin and hemicelluloses.

Overall, these results confirm that CPW is a competitive lignocellulosic feedstock when processed under controlled, comparable conditions. The simultaneous achievement of high pulp yield and efficient bleaching highlights its potential for producing high-quality cellulose fibers. This performance further establishes CPW as a sustainable and promising alternative for cellulose-based applications where yield efficiency and fiber purity are critical.

Cellulosic fiber characterization

Total charge measurement analysis

To assess the total charge of CPW and CPW-C, conductometric titration was applied. The resulting measurements are presented in Figure 3, together with other results reported in the literature.

CPW biomass exhibited a total charge of 110.44 µeq.g^{-1}, which is comparable to fig stems[52] and *Prunus amygdalus* (110 µeq.g^{-1}), slightly higher that *Tamarisk* sp. (100 µeq.g^{-1}),[53] and considerably lower than date palm rachis (291 µeq.g^{-1}).[24] This intermediate value suggests a balanced presence of ionizable functional groups, mainly carboxylic, associated with hemicellulose and lignin's phenolic content. The relatively high total charge of CPW biomass indicates a great potential for chemical reactivity and interactions, which is advantageous for subsequent processing steps, such as surface modification and cellulose extraction.

The total charge of the cellulosic fibers extracted from CPW is 45.48 µeq·g^{-1}. This result is comparable to that of other natural fibers, such as cotton, which has a total charge of 43.2 µeq·g^{-1}.[54] In contrast, it is considerably higher than that of regenerated fibers, such as modal and lyocell fibers, which have total charges of 20.6 and 27.2 µeq·g^{-1}, respectively.[54] This parameter is considered important in various industries, particularly the paper industry, since it can positively or negatively affect the papermaking process. Indeed, it provides insight into the fiber's behavior toward cationic additives, its softness, its water adsorption, and its strength.[55,56]

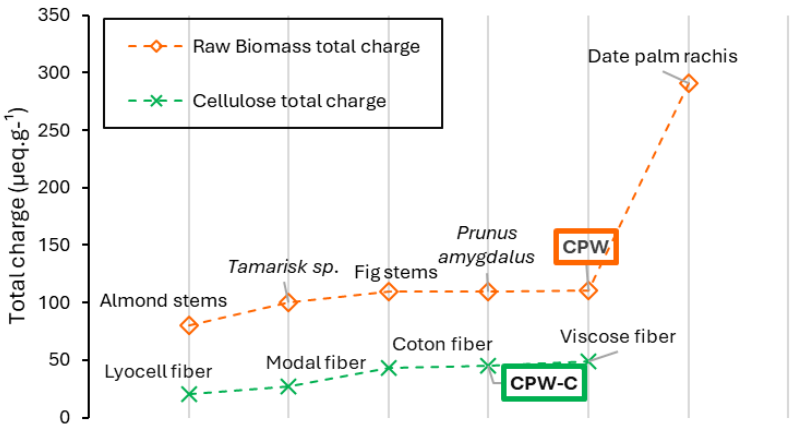

Figure 3: Total charge analysis of CPW raw biomass and cellulose compared to other sources reported in the literature[24,52–54]

Spectroscopic analysis

X-ray diffraction (XRD) analysis was conducted to examine the crystalline structure and degree of organization of CPW-C. The obtained diffraction pattern (Fig. 4) exhibits distinct peaks characteristic of semicrystalline cellulose, indicating a complex structural organization. The diffractogram shows that both Cellulose Iβ and Cellulose II allomorphs are present, suggesting that the chemical treatments partially transformed the native cellulose structure. Cellulose Iβ typically corresponds to the native crystalline form found in plant cell walls, whereas Cellulose II is associated with regenerated or mercerized cellulose arising from alkaline processing. The presence of these two phases highlights the effectiveness of the extraction and purification procedures, while reflecting the structural rearrangements that occurred during alkaline hydrolysis.

In addition to the cellulosic phases, the XRD pattern shows diffraction peaks attributable to a mineral crystalline phase identified as calcite ($CaCO_3$). This inorganic component may originate from the biomass' intrinsic mineral content or residual impurities that were not completely removed during the treatment process. Its detection further emphasizes the heterogeneous nature of the raw material and suggests potential interactions between organic and inorganic constituents within the structure.

The crystallinity index (CrI) of CPW-C was determined using the widely adopted Segal method,[27] which provides a semi-quantitative estimation of the relative crystalline fraction. Based on the measured intensities of the crystalline peak ($I_{200} \approx 34{,}000$) and the amorphous region ($I\_am \approx 11{,}000$), the crystallinity index was calculated to be approximately 68%. Compared to other sources (Table 2), the obtained Crl (68%) falls within the range reported for cellulose extracted from agricultural residue, such as maize stalk (66.1%), olive tree pruning (70.6%), *Coffea arabica* L. (54.9-66.3%) and citrus pomace (61.9-77.9%), and is slightly lower than that of corn husk (72.9%). However, it is significantly higher than the values reported for rice husk (50%) and *Pachygone ovata* leaf (43.6%). This relatively high CrI value indicates a significant proportion of ordered domains within the cellulose structure, reflecting the efficient removal of amorphous components, such as lignin and hemicelluloses, during the treatment process. Such a level of crystallinity is generally associated with enhanced mechanical strength, thermal stability, and resistance to chemical degradation, which are desirable properties for advanced materials.

Overall, the XRD results confirm the successful isolation of a highly structured cellulose material from CPW and provide valuable insights into its polymorphic composition and structural organization.

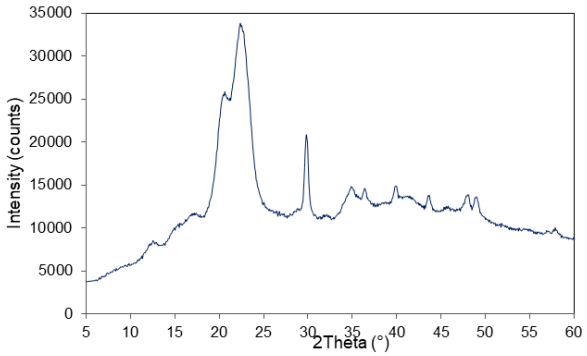

Figure 4: XRD pattern of CPW-C

Table 2
Crystallinity index of CPW-C compared to other cellulosic sources

Source of cellulose	Crystallinity index (%)	Reference
CPW	68	This work
Corn husk	72.9	44
Rice husk	50	57
Coffea arabica L.	54.9-66.3	58
Cirtus pomace	61.9-77.9	59
Pachygone ovata leaf	48.6	60
Maize stalks	66.1	61
Olive tree pruning	70.6	62
Oil palm mesocarp fiber	51-55	63

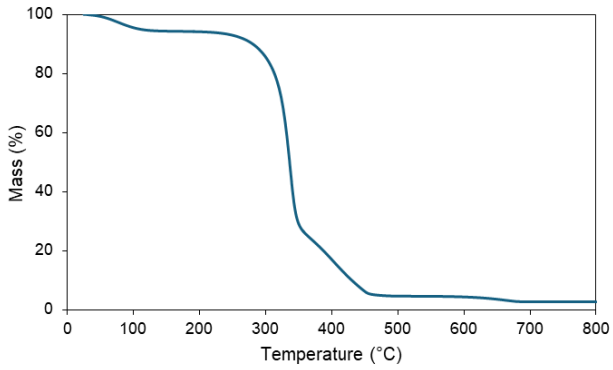

Figure 5: Thermogravimetric analysis of CPW-C

Thermogravimetric analysis

The thermogravimetric analysis (TGA) curve of CPW-C is presented in Figure 5, providing insight into its thermal stability and decomposition behavior. The thermal degradation profile can be divided into three distinct stages, reflecting the complex composition of the biomass. The first stage, occurring around 100 °C, corresponds to a minor weight loss of the sample, which can be attributed to the evaporation of residual moisture physically adsorbed or trapped within the hydrophilic cellulose matrix. This initial dehydration phase is typical of lignocellulosic materials and confirms the presence of bound water within the purified cellulose.

The second stage, spanning the temperature range from approximately 100 °C to 450 °C, is characterized by a pronounced and rapid mass loss, with a peak degradation rate near 350 °C. This stage is primarily associated with the thermal decomposition of the organic constituents, including cellulose, hemicellulose residues, and minor lignin fragments. The relatively high decomposition temperature

indicates a well-ordered crystalline cellulose structure, consistent with the XRD results, and suggests a high degree of thermal resistance suitable for potential high-temperature applications.

The final stage exhibits a plateau in the mass loss curve, indicating that the remaining material exhibits considerable thermal stability. The residual mass at the end of the analysis amounts to approximately 4% of the initial sample weight, which closely aligns with the ash content (4.1%) determined from the chemical composition analysis of the biomass. This residue likely corresponds to inorganic mineral components, such as calcium carbonate ($CaCO_3$) and other trace minerals inherent to the raw material.

Overall, the TGA analysis confirms that CPW-C possesses a high thermal stability, with a well-defined degradation profile. The results suggest that this cellulose source is not only structurally robust, but also suitable for applications requiring heat resistance, such as in bio-composites, packaging materials, and functional cellulose-based products.

Morphological analysis

Cellulosic fibers obtained after delignification and bleaching of CPW (CPW-C) were characterized using Scanning Electron Microscopy and MorFi analysis to evaluate their morphology, size distribution, and degree of fibrillation. These complementary techniques provide both qualitative and quantitative insights into the structural organization of the extracted cellulose.

Figure 6 reveals well individualized fibers, confirming the effective removal of lignin and hemicelluloses, which act as a binding matrix within the plant cell wall. The disappearance of this matrix facilitates the separation of fiber bundles into elementary fibers. The observed fibers exhibit an elongated and relatively cylindrical morphology, with some heterogeneity in length and surface structure. This variability may be attributed to partial fibrillation occurring during the chemical treatment process.

The surface of the fibers appears relatively clean and free of non-cellulosic residues, indicating the efficiency of the delignification and bleaching steps. In some regions, slight peeling and fibrillation of the fiber surface can be observed, suggesting the beginning of microfibril exposure without complete structural degradation.

Morfi analysis provided quantitative measurements of fiber dimensions as shown in Figure 7. CPW-C fibers exhibited an average length of 0.926 mm, a width of 22 µm, and a fine element fraction of 31.9%. These results indicate that the fibers are relatively long and moderately thin, which is advantageous for mechanical reinforcement and fiber bonding applications.

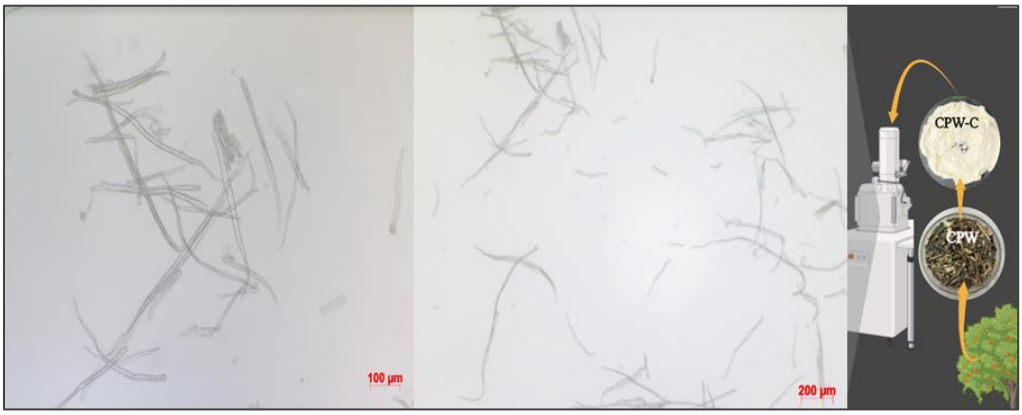

Figure 6: Scanning electron microscopy images of CPW-C

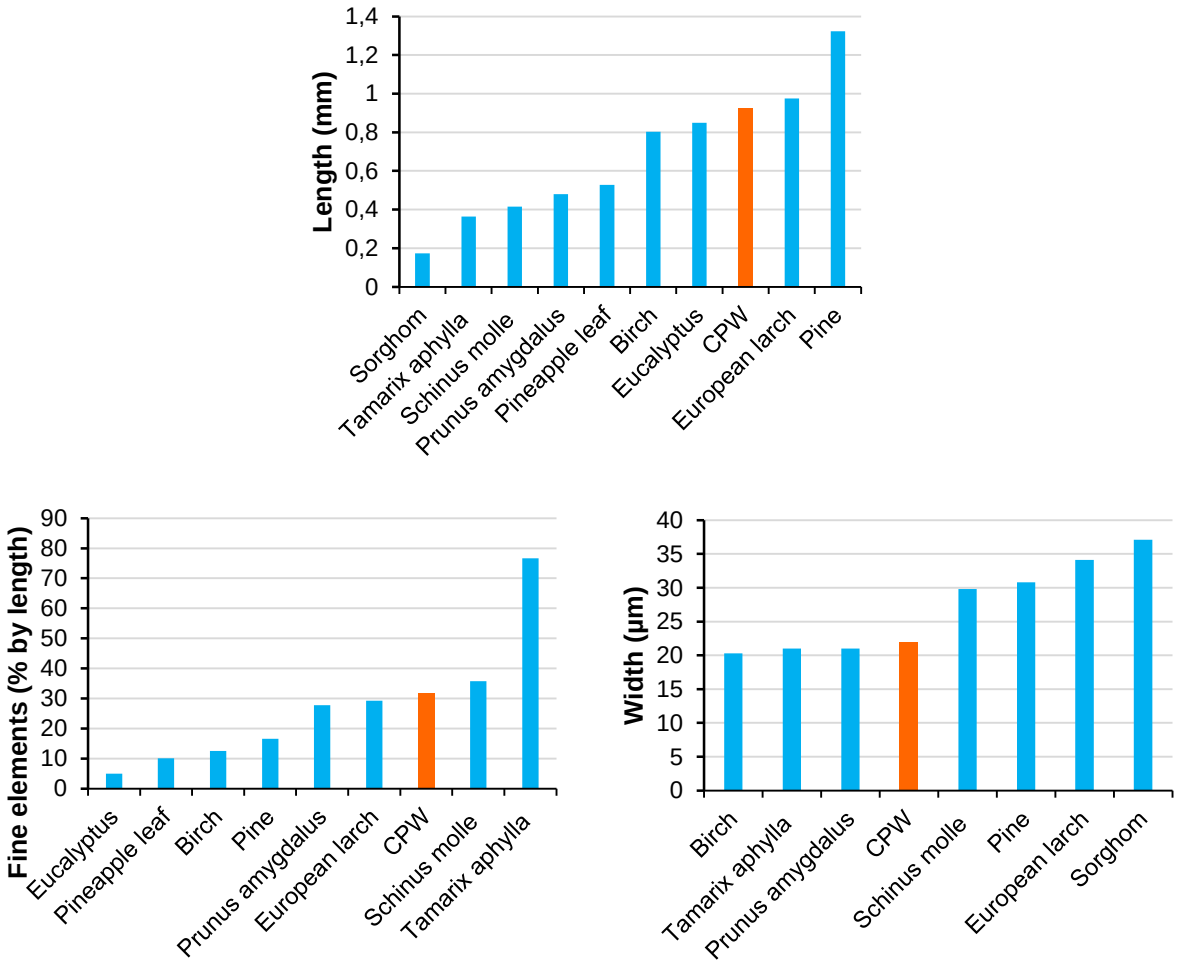

Figure 7: Morfi analysis of cellulosic fiber obtained from CPW and other sources reported in the literature[23,43–45,64–66]

Compared to other lignocellulosic sources such as *Schinus molle* (0.415 mm)[23] and *Tamarix aphylla* (0.364 mm),[43] *Prunus amygdalus* (0.48 mm),[44] pineapple leaf (0.528 mm)[45] and sorghom (0.174 mm),[64] CPW-C fibers show significantly greater length, while remaining comparable to other sources, like Eucalyptus (0.850 mm),[65] European larch (0.975 mm), pine (1.323 mm) and birch (0.803 mm),[66] highlighting their strong structural integrity after chemical treatment. Fiber width (22 µm) is comparable to that of *Tamarix aphylla*, *Prunus amygdalus* and birch (≈21 µm) and lower than that of *Schinus molle* (29.8 µm), sorghom (37.1 µm), European larch (34.1 µm) and pine (30.8 µm), reflecting a controlled treatment process, without excessive fiber swelling or collapse.[23]

The fine element content (31.9%) is lower than that of *Tamarix aphylla* (76.6%) and slightly lower than *Schinus molle* (35.8%),[23] while it remains higher than the value reported for Eucalyptus (5%) and other sources ranging from 10.1% to 27.8%.[65] This moderate value suggests a balanced degree of fibrillation, where sufficient fines are generated to enhance surface area and bonding ability, while maintaining fiber integrity. Excessive fines, as observed in *Tamarix aphylla*[43] may indicate over-processing and fiber degradation.

CPW-C fibers present characteristics comparable to those of conventional wood fibers and certain annual plant fibers. Wood fibers typically exhibit longer lengths and lower fines content, contributing to high mechanical strength, while fibers from annual plants often show higher variability and increased fibrillation. In this context, CPW-C fibers demonstrate a favorable compromise between structural integrity and surface reactivity, making them suitable for applications in papermaking and biocomposites.

CONCLUSION

This study demonstrates the efficient utilization of *Citrus aurantium* pruning waste by extracting cellulose through an alkaline hydrolysis process combined with sodium chlorite-assisted delignification and bleaching. Compositional analysis revealed high levels of holocellulose and α-cellulose, which supports the use of this lignocellulosic biomass as a sustainable and renewable source of cellulose. The chemical treatments effectively removed the lignin and hemicelluloses, producing a purified cellulose fraction with desirable physicochemical properties.

Structural characterization by X-ray diffraction (XRD) revealed a crystallinity index of around 68%, indicating a well-organized, predominantly crystalline cellulose structure. This relatively high crystallinity suggests improved intermolecular hydrogen bonding and enhanced mechanical performance. Thermogravimetric analysis (TGA) further demonstrated the good thermal stability of the extracted cellulose, with a primary degradation peak observed around 350 °C, which is consistent with the thermal decomposition behavior of typical cellulose.

Morphological investigations using Scanning Electron Microscopy (SEM) and Morfi fiber analysis confirmed the efficient defibrillation and separation of cellulosic fibers. The obtained fibers exhibited relatively high average lengths, moderate diameters and balanced proportions of fine elements, indicating favorable aspect ratios and homogeneous fiber distributions. These morphological features are particularly advantageous for applications requiring mechanical reinforcement, such as composite materials or functional fillers. Furthermore, the extracted cellulose has great potential for processing into high-value nano-cellulosic materials, such as cellulose nanofibrils or nanocrystals.

Overall, pruning waste from *Citrus aurantium* is a promising, yet underutilized source of cellulose. Valorizing this waste contributes to the development of sustainable materials and provides a more environmentally friendly alternative to conventional wood-based feedstock within the framework of a circular bioeconomy.

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