

ISOLATION AND CHARACTERIZATION OF CELLULOSE NANOFIBRILS FROM *PHORMIUM TENAX*

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Abstract. This study investigates the production and characterization of cellulose nanofibers (CNF) extracted from *Phormium tenax* using environmentally friendlier treatments based on water, ethanol, and NaOH under controlled pressure, agitation, and temperature conditions. Four pretreatments (T1-T4) were evaluated to promote selective lignin removal while reducing the use of aggressive chemical reagents. FTIR analysis showed that the NaOH-based treatments (T3 and T4) were the most effective for lignin removal, enabling the production of CNF in gel form. SEM and TEM analyses revealed homogeneous nanofiber networks, particularly for T4, with diameters ranging from 19 to 28 nm. X-ray diffraction demonstrated a marked increase in crystallinity for T3 (69.2%) and T4 (66.0%) compared with untreated fibers (36.7%), indicating effective removal of amorphous components while preserving the crystalline cellulose structure. These findings demonstrate that *Phormium tenax* is a promising and underexplored lignocellulosic source for high-quality CNF production and highlight a simpler, more sustainable extraction route with potential applications in polymer composites and advanced biomaterials.

Keywords: *phormium tenax*; nanocellulose; lignin removal; characterization; sustainable processing.

EXTRAÇÃO E CARACTERIZAÇÃO DE NANOFIBRILAS DE CELULOSE DE *PHORMIUM TENAX*

Resumo. Este estudo investiga a produção e a caracterização de nanofibras de celulose (CNF) extraídas de *Phormium tenax* utilizando tratamentos ambientalmente mais sustentáveis à base de água, etanol e NaOH, sob condições controladas de pressão, agitação e temperatura. Quatro pré-tratamentos (T1-T4) foram avaliados com o objetivo de promover a remoção seletiva de lignina, reduzindo simultaneamente o uso de reagentes químicos agressivos. A análise por FTIR mostrou que os tratamentos à base de NaOH (T3 e T4) foram os mais eficazes na remoção de lignina, possibilitando a obtenção de CNF na forma de gel. As análises por MEV e MET revelaram redes homogêneas de nanofibras, especialmente para o tratamento T4, com diâmetros variando entre 19 e 28 nm. A difração de raios X demonstrou um aumento significativo da cristalinidade para T3 (69,2%) e T4 (66,0%) em comparação com as fibras não tratadas (36,7%), indicando a remoção eficiente dos componentes amorfos e a preservação da estrutura cristalina da celulose. Esses resultados demonstram que *Phormium tenax* é uma fonte lignocelulósica promissora e ainda pouco explorada para a produção de CNF de alta qualidade, além de evidenciar uma rota de extração mais simples e sustentável, com potencial para aplicações em compósitos poliméricos e biomateriais avançados.

Palavras-chave: *phormium tenax*; nanocelulose; remoção de lignina; caracterização; processamento sustentável.

1 INTRODUCTION

Cellulose is the most abundant natural biopolymer and the main structural component of plant cell walls, providing rigidity and mechanical support. Its linear β -(1 $\rightarrow$ 4)-D-glucose chains are organized into highly ordered microfibrils stabilized by hydrogen bonding. Owing to its renewability, biodegradability, low density, and excellent mechanical properties, cellulose has become an important sustainable raw material, particularly for nanocellulose production (Mahmoud *et al.*, 2024).

Among cellulose-derived nanomaterials, cellulose nanocrystals (CNC) and cellulose nanofibers (CNF) stand out for their high aspect ratio, large specific surface area, low density, and outstanding mechanical properties, enabling applications in composites, packaging, biomedical materials, coatings, barrier systems, and environmental technologies. In particular, CNF exhibit an entangled fibrillar network that enhances reinforcement efficiency and the rheological behavior of composite matrices (Zha *et al.*, 2024). However, conventional nanocellulose extraction often relies on aggressive chemical treatments involving strong acids, chlorinated compounds, or concentrated oxidants, increasing environmental impact, energy consumption, and processing costs. Consequently, greener extraction routes with lower chemical consumption have received increasing attention. Among these, the cellulose-first approach prioritizes selective cellulose recovery for high-value materials while allowing subsequent valorization of the solubilized fractions (Shen and Sun, 2021).

Phormium tenax, an evergreen species native to New Zealand and Norfolk Island, is a promising lignocellulosic source due to its high cellulose content and favorable fiber morphology. Although CNC and CNF have previously been produced from chemically and oxidatively pretreated fibers (Di Giorgio *et al.*, 2020; Ortiz *et al.*, 2018), environmentally friendlier extraction methods remain scarce. In this study, cellulose nanofibers were extracted from *Phormium tenax* using a low-cost, environmentally friendlier process based on elevated temperature and pressure with reduced chemical consumption. Four pretreatments were evaluated, and two produced high-quality cellulose nanofibers while minimizing the use of aggressive reagents and mechanical processing.

2 EXPERIMENTAL

2.1 Pretreatments and mechanical defibrillation

The *Phormium tenax* leaves were collected in the municipality of Quatro Barras (Paraná, Brazil). After drying by the natural convection, they were manually cut into strips measuring 5 cm in length and subjected to four chemical treatments (P1, P2, P3 and P4) for delignification and the removal of extractives, pectin, and hemicellulose, with the aim of isolating cellulose for the subsequent production of cellulose nanofibers (CNFs) via mechanical defibrillation. The treatments were designed to evaluate the efficacy of using the minimal possible amount of polluting reagents, beginning with water, ethanol, or combinations thereof with NaOH. All treatments were conducted using a hydromodule of 1:4 at 140 °C for 1 hour in 2 L capsules placed inside a stainless-steel rotary reactor (REGMED, model HB-21). The reactor consisted of four electrically heated internal capsules equipped with an automatic time and temperature control system, operating at 2.5 rpm under a pressure of 2.8 kgf/cm². Treatment 1 (P1) involved the use of water only. Treatment 2 (P2) followed the Organosolv method, employing a 70:30 ethanol: water solution with ethanol constituting 46% of the dry mass (Robles *et al.*, 2018). Treatment 3 (P3) used an alkaline solution containing 15% NaOH (analytical grade). Finally, Treatment 4 (P4) combined alkaline treatment with the Organosolv method, using 15% NaOH and 46% ethanol on the dry mass (Berhanu *et al.*, 2018). All reagents are analytical grade from Sigma Aldrich-Merck. In the second stage, the raw pulp was filtered to discard the liquid fraction, and the solid fraction (raw pulp) was washed with distilled water using a Bauer disc shredder (REGMED), operating under atmospheric (non-pressurized) conditions. The equipment had a capacity of 60 L and a motor power of 7 kW. During this process, the fibrous material was disaggregated and washed at a speed of 1,725 rpm with a low consistency of 0.05% (dry mass basis) to remove residual cooking liquor. The process was followed by centrifugation to purify the pulp further. The third stage involved mechanical defibrillation using a Super Masscolloider mill (Masuko Sangyo, model MKCA6-2), with a 0.1 mm gap between the grinding stones, operating at 1,500 rpm and a consistency of 1%. The number of passes through the mill was set at 10 for pulps derived from treatments P1 and P2, and 5 for those from P3 and P4. After the mechanical treatments, the samples were named as T1, T2, T3 and T4. The difference of pass number used (10 or 5) was based on prior experiments with fibers such as jacitara, silk-floss, coconut, pampas grass, and peach palm, which demonstrated that 10 passes are sufficient to obtain high-quality CNFs (Franco *et al.*, 2019). Finally, films were produced using the casting

2.2 Chemical analysis of extractives and lignin in *phormium tenax* fibers

The total extractive and lignin contents of *Phormium tenax* fibers after chemical treatments were determined from raw pulps P1, P2, P3 and P4, as well as of sawdust from its dried leaves *in natura*, in accordance with the standards of the Technical Association of the Pulp and Paper Industry (1997; 1954; 2021), in triplicate. The sawdust was produced in a Marconi knife mill, and the sample was collected from the material retained in a sieve with a mesh size of 60. The total extractive content was determined according to the TAPPI T264 standard. The lignin content was determined according to the TAPPI T13-os-54 and T222 standards. Additionally, we analyzed the material's composition using Energy Dispersive Spectroscopy (EDS), performed with an FEI Quanta FEG 450 microscope, with six spectra analyzed for each pulp sample.

2.3 XRD characterization of chemically treated and mechanically defibrillated *Phormium tenax* pulps

The XRD measurements on *Phormium tenax* treated pulps after mechanical defibrillation were performed using an Ultima IV/Rigaku X-ray diffractometer with continuous scanning and grazing analysis. The 2θ interval started at 5.00° and ended at 90.01° , with a step of 0.6 degrees and a speed of 96 s by step. The wavelength used was $1.541 \text{ \AA}$, with a voltage of 45 kV and current of 35 mA. The crystallinity index (*CI*) of the samples was calculated according to the Segal equation (Nam *et al.*, 2024).

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

Where I_{002} and I_{am} refer to the diffraction peaks in the crystallographic plane (002) and to the minimum intensity, respectively.

2.4 FTIR Spectroscopic Characterization of Chemically Treated and Mechanically Defibrillated *Phormium tenax* Pulps

FTIR spectroscopy analyses were performed on an IRPrestige-21 infrared spectrometer (Shimadzu) equipped with a DRS-8000 diffuse reflectance accessory (Shimadzu) in transmittance mode. A total of 64 scans were performed with a resolution of 2 cm^{-1} , and the measurement range varied from 400 to 4000 cm^{-1} . Images by Scanning Electron Microscopy (SEM) were performed using a FEI Quanta 450 FEG. For *Phormium tenax* raw pulp, the surfaces were metallized with gold and magnifications of 100, 500, 1500 and 3500 times were applied. In the case of *Phormium tenax* CNF images were made only of samples CNF-T3 and CNF-T4 since treatments 1 and 2 did not present gel consistency. In addition, the composition of the material was analyzed using the Energy Dispersive Spectroscopy (EDS) technique. Images by Transmission Electron Microscopy (TEM) on samples CNF-T3 and CNF-T4 were performed with a JEM 1200EX-II (Jeol).

3 RESULTS AND DISCUSSION

3.1 Chemical Composition of *Phormium Tenax*

This study was conducted using a single extraction of *Phormium tenax* fibers to minimize the intrinsic variability of natural lignocellulosic materials, whose chemical composition, crystallinity, lignin content, moisture, physiological age, and growth conditions may vary even within the same species. This approach enabled a more consistent comparison of the chemical treatments (Satyanarayana; Flores-Sahagun; Bowman, 2022). The extractive and lignin contents of sawdust and raw pulps of *Phormium tenax* (P1, P2, P3 and P4) are presented in Table 1, after the four treatments.

Tabel 1: Extractive and lignin content (%) of sawdust and raw pulp of *Phormium tenax*.

	P1	P2	P3	P4	Sawdust
Lignin	16.2 ± 0.1	17.2 ± 0.5	9.5 ± 0.6	6.1 ± 0.2	11.9 ± 0.9
Total Extractives	3.1 ± 0.1	2.4 ± 0.5	1.9 ± 0.1	0.9 ± 0.4	26.7 ± 0.7

Hot water has been shown to be efficient in removing tannins, gums, sugars, starch and pigments (Kilpeläinen, Liski and Saranpää, 2023). It can be observed that sample P1, treated only with water, presented a slightly higher total extractive content than sample P2, extracted with ethanol-water in the proportion 70/30. The lignin content is rather the same for samples P1 and P2. In any case, the total extractive values of samples P1 and P2, $3.1 \pm 0.1\%$ and $2.4 \pm 0.5\%$ respectively, are much lower than the $26.7 \pm 0.7\%$ found in the sawdust. After extraction, P1 and P2 were lighter than the ground sawdust, which suggests that gums, tannins, sugars, starch and pigments were extracted. The differences in lignin content between the raw fiber and treated samples are attributed to the natural heterogeneity of *Phormium tenax* leaves. Similar variability has been reported for lignocellulosic fibers, particularly in long leaves such as *Phormium tenax* and sisal, whose basal and apical regions differ in lignin content, chemical composition, crystallinity, and extractives (Dube; Daba; Muleta, 2023). To reflect industrial practice, the entire biomass was cut, homogenized, and used without selecting specific leaf regions. Despite homogenization, the natural heterogeneity of the fibers resulted in the expected variation in lignin content, consistent with previous studies (Park *et al.*, 2022).

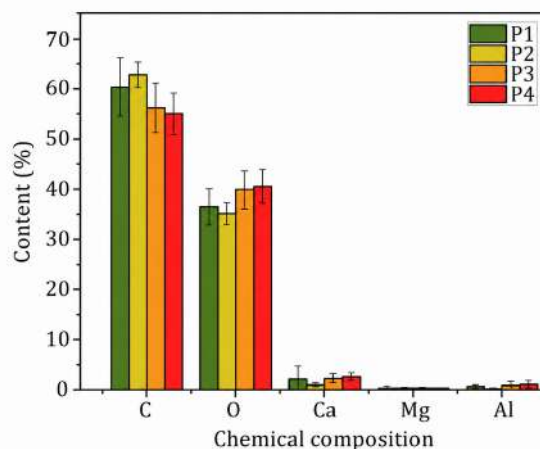
Also, the pleasant scent of *Phormium tenax* fibers remained in all treated pulps, showing that no degradation occurred, even with the use of a relatively high pressure and temperature. Since lignin is an amorphous polyphenol, a highly cross-linked material, it was not expected that water or water-ethanol would be able to remove lignin from the material, even under heating and pressure (Safian *et al.*, 2022). And although treatments with a pressure of 2.8 kgf/cm² and a temperature of 140 °C with water or water-ethanol were not effective for the removal of lignin, the effectiveness in the removal of other compounds indicated that this treatment is a simpler and more environmentally friendly way to extract cellulose, if used in conjunction with an appropriate reagent such as NaOH.

Fiber delignification with NaOH was more effective for samples P3 and P4. Sample P4 (NaOH-ethanol) presented a lignin content of 6.1%, a lower value than that of sample P3 (NaOH-water), which was 9.5%. Therefore, the cellulose content of sample P4 is higher than that of sample P3, which suggests that the nanocellulose obtained from sample P4 must have a greater reinforcing potential than that obtained from sample P3. P3 fibers presented the lignin content close to that of the *Phormium tenax* fibers studied by Sammartino *et al.* (2010), whose value was 9.1%. The use of NaOH in combination with ethanol for the chemical treatment of leaf fibers to extract cellulose offers notable advantages over the traditional NaOH-water approach. Sodium hydroxide is commonly employed to remove components such as lignin, hemicellulose, waxes, and fats from natural fibers. The addition of ethanol, however, enhances this process by acting as an auxiliary solvent. Ethanol facilitates the dissolution of nonpolar compounds, such as waxes and resins, that are poorly soluble in water, thereby improving the removal efficiency of lignin and hemicellulose. Moreover, ethanol helps reduce cellulose chain degradation during the treatment by minimizing excessive hydrolysis, which contributes to the preservation of the fibers' mechanical properties. The NaOH-ethanol combination enables a more effective extraction of non-cellulosic substances, leading to a more selective and higher-yield cellulose yield. This is particularly beneficial for leaf fibers, which often contain waxes and resins that interfere with the treatment when only NaOH and water are used.

The EDS chemical mapping results of the pulps are shown in Fig. 1.

For the minor inorganic elements (Ca, Mg, and Al), low concentrations and slightly higher relative variations were observed. This behavior is expected because trace elements are more sensitive to localized heterogeneity of the biomass surface and to limitations inherent to EDS semi-quantitative analysis. Even so, the low standard deviations obtained demonstrate satisfactory consistency among the replicates.

Although Thandavamoorthy, Devarajan, and Thanappan (2023) report that alkaline treatments typically lead to increased carbon content and decreased oxygen content, due to the removal of oxygen-rich groups from hemicellulose and lignin, it is important to consider the oxygen composition of cellulose itself. Cellulose is a polysaccharide composed of long chains of glucose units (C₆H₁₂O₆), making it a carbohydrate with a high oxygen content. These oxygen atoms are present in both hydroxyl (C-OH) and glycosidic (C-O-C) bonds. Hemicellulose, although also a carbohydrate, has a more heterogeneous and branched structure, incorporating various sugar monomers. This complexity can lead to a relatively higher oxygen density compared to cellulose, though hemicellulose is more easily removed by alkaline treatment. Lignin, in contrast, is a complex, irregular polymer composed of aromatic phenolic units. It contains less oxygen than both cellulose and hemicellulose and is comparatively richer in carbon. Therefore, an observed increase in oxygen content following NaOH treatment may appear counterintuitive, given

Figure 1: Chemical composition of *Phormium tenax* pulp determined by EDS.

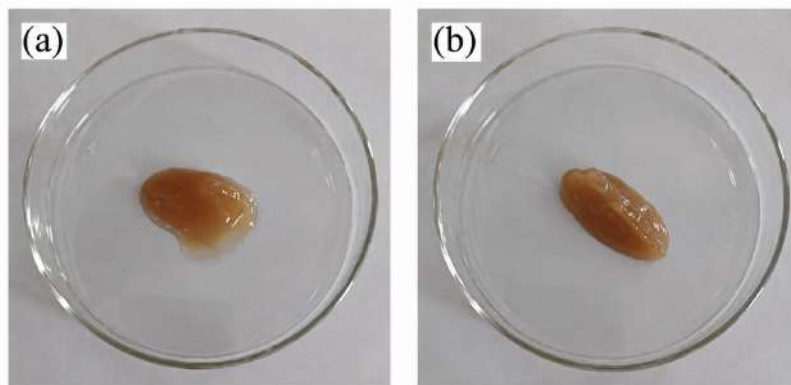
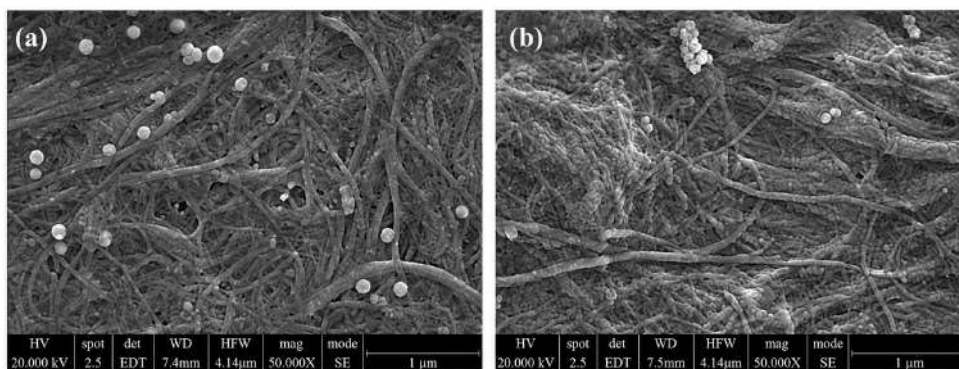
that this process typically removes lignin and hemicellulose, components generally associated with higher oxygen content. However, the selective removal of these substances and the relative enrichment of cellulose, with its high proportion of oxygenated functional groups, can explain this outcome. This phenomenon can be attributed to several factors. The removal of lignin and hemicellulose exposes a greater surface area of cellulose, which, despite having a lower overall oxygen-to-carbon ratio, still contains oxygen in its glycosidic bonds and hydroxyl groups. This increased exposure can lead to an apparent rise in surface oxygen content. Additionally, NaOH may induce oxidation reactions on the fiber surface, generating new oxygen-containing functional groups. The alkaline treatment also removes non-oxygenated impurities, such as waxes and oils, thereby increasing the relative proportion of oxygen in the remaining material. In some cases, oxygenated compounds may form or be adsorbed onto the fiber surface, further contributing to the observed increase in oxygen content (Rao *et al.*, 2025).

3.2 Extraction of Nanocellulose from *Phormium Tenax* Fibers

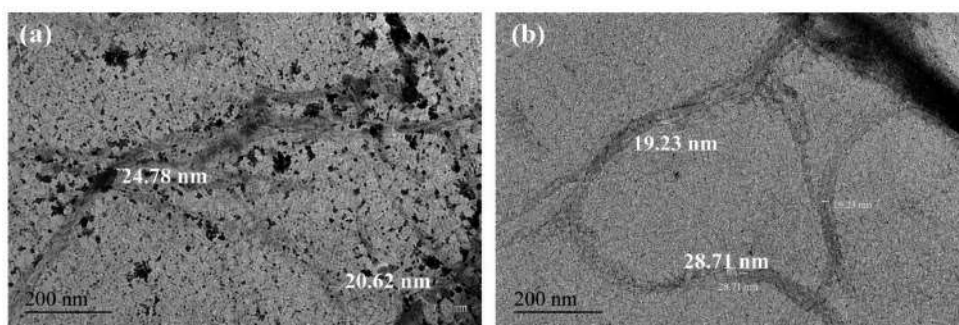
For pulps P1 and P2, 10 milling passes produced only aqueous suspensions, indicating insufficient cellulose extraction. In contrast, P3 and P4 formed the desired gel after five passes, demonstrating that NaOH was essential for lignin removal and gel formation. P4 also showed the highest extractives removal and, owing to its lower lignin content, is expected to provide greater reinforcement potential than P3. Figure 2 shows the *Phormium tenax* nanocelluloses obtained. As can be seen in Fig. 2(a), the nanocellulose obtained after treatment with NaOH presented a gel consistency; however, as can be seen in Fig. 2(b), this effect is more pronounced after treatment with NaOH-ethanol. Another aspect to be observed in the brownish samples is the less intense coloration in the sample in Fig. 2(b). The nanocellulose samples in this work were not subjected to bleaching reactions, which would whiten them.

Figure 3(a) shows a SEM micrograph of nanofibrillated cellulose T3 (CNF-T3) at 50k x magnification. The micrograph of the CNF-T3 sample revealed a dense network of cellulose nanofibers, exhibiting good fiber dispersion with only occasional localized agglomerations. Additionally, small spherical particles were observed adhered to the fibril surfaces, likely originating from contaminants or residual by-products of the production process. The nanofibers displayed uniform dimensions and a fine, well-individualized structure, characteristic of nanofibrillated cellulose. Figure 3(b) shows a SEM micrograph of nanofibrillated cellulose T4 (CNF-T4) at 50k x magnification.

The CNF-T4 sample also exhibited a network of cellulose nanofibers, with a more uniform distribution compared to CNF-T3. Adhered particles were less apparent in this image, and the nanofibers appeared elongated and well-dispersed. The nanofibers also showed slightly greater thickness than those in the CNF-T3 sample, suggesting a lower degree of fibrillation or variations in the treatment conditions applied during production.

Figure 2: Nanofibrillated cellulose: (a) T3, (b) T4.**Figure 3:** SEM micrography of: (a) CNF-T3, (b) CNF-T4.

A TEM micrograph of CNF-T3 is shown in Fig. 4(a), and a TEM micrograph of CNF-T4 is shown in Fig. 4(b).

Figure 4: TEM micrography of: (a) CNF-T3, (b) CNF-T4.

The TEM micrograph of the CNF-T3 sample revealed the presence of nanofibers with a thickness around 20.62 to 24.76 nm. This complements the SEM observation (Fig. 3(a)), where the nanofibers are thin and well dispersed, showing more details about the real dimension of the nanofibers observed in the SEM analysis. In the TEM micrograph of CNF-T4, the nanofibers exhibited consistent dimensions, with diameters ranging from 19.23 to 28.11 nm. These measurements align with the observations from the SEM micrograph (Fig. 3(b)), which also

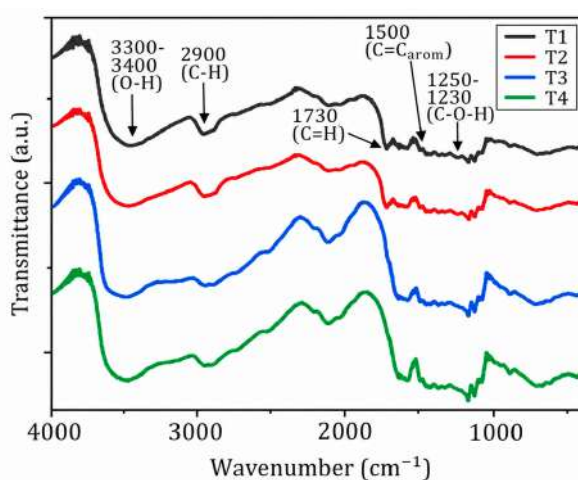
revealed slightly thicker, uniformly distributed nanofibers.

Treatments 3 and 4 used for the extraction of nanofibrillated cellulose (CNF) from *Phormium tenax* in this study proved to be effective, as demonstrated by the SEM and TEM analyses. Notably, the CNF-T3 and CNF-T4 samples were obtained using a more environmentally friendly chemical process, incorporating high pressure and temperature, in contrast to the method employed by Di Giorgio *et al.* (2020), which relied on more polluting reagents.

3.3 FTIR Spectroscopy Results

Figure 5 shows the FTIR spectra of samples T1, T2, T3 and T4.

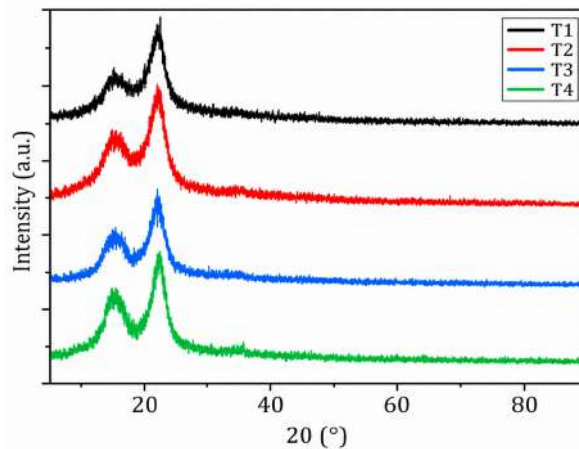
Figure 5: FTIR spectra of treated *Phormium Tenax* pulp after mechanical defibrillation.



At 3340 cm^{-1} , an increase was observed for T3 and T4 of this band, which corresponds to the stretching vibration of the O-H bond, characteristic of the hydroxyl groups present in cellulose (Vârban *et al.*, 2021). The increase in the intensity of this band can be considered an indication of the increase in the cellulose content in samples T3 and T4, compared to T1 and T2. In addition, the decrease in the peak at 2900 cm^{-1} for T3 and T4 indicated the removal of amorphous portions of cellulose from these samples, which may have promoted an increase in their crystallinity indices, compared to T1 and T2 (Bhagia *et al.*, 2022). The disappearance of the band at 1730 cm^{-1} in samples T3 and T4, corresponding to the stretching of the acetyl and uronic groups of hemicellulose, demonstrated the effectiveness of chemical treatments 3 and 4 in removing this constituent (Chambre and Dochia, 2021). The decrease in the intensity of the band at 1500 cm^{-1} in T3 and T4 indicated the partial removal of lignin and hemicellulose, since this band is related to the stretching of the C=C bonds present in the conjugated system of the aromatic rings of these components. In addition, the absence of the band between $1250\text{--}1230\text{ cm}^{-1}$ in T3 and T4, attributed to the angular deformation of primary alcohols of the C-O-H bond in lignin and hemicellulose, indicated the removal of these constituents by the treatments applied (Vârban *et al.*, 2021; Bhagia *et al.*, 2022). Considering the increases in the characteristic bands of functional groups related to the presence of cellulose, and at the same time, decrease or disappearance of bands related to the presence of lignin and hemicellulose, it can be stated that the chemical treatments with NaOH (treatment 3) and NaOH-ethanol (treatment 4) were efficient in removing the non-cellulosic components from the treated samples, offering a more sustainable delignification route with reduced chemical consumption, while highlighting *Phormium tenax* as a promising and underexplored source of cellulose nanofibers.

3.4 XRD Results

Figure 6 shows the XRD diffractograms of the treated *Phormium tenax* pulps.

Figure 6: XRD diffraction patterns of treated *Phormium tenax* pulp after mechanical defibrillation.

In Fig. 6, characteristic peaks of cellulose I are observed, located around 16.5° , referring to the (110) plane, and 22.5° , referring to the (200) plane (Liu *et al.*, 2022). The peak around 35° , referring to the (400) plane, also indicative of the presence of cellulose I, appears, although subtly, in the T2, T3 and T4 curves. When conversion to cellulose II occurs, a change in the peak around 22° of cellulose I is observed, which decreases. However, no change in this peak was observed in the XRD diffractograms of samples submitted to treatments 1 to 4. Cellulose I, naturally present in plants, is more rigid and resistant than cellulose II, formed by mercerization or regeneration. To preserve these properties, treatments that maintain cellulose I are preferred (Stanciu, Tanasa and Teaca, 2025). The crystallinity indices of the films of the treated pulps of *Phormium tenax* after mechanical defibrillation are presented in Table 2.

Tabel 2: Crystallinity index of treated *Phormium tenax* pulp after mechanical defibrillation.

Treatment	I_{002}	I_{am}	CI (%)
T1	290 ($2\theta=22.54^\circ$)	107	63.1
T2	329 ($2\theta=22^\circ$)	127	61.4
T3	260 ($2\theta=22^\circ$)	80	69.2
T4	303 ($2\theta=22.52^\circ$)	103	66.0

The CI obtained in this work were comparable to those obtained by Duchemin *et al.* (2009). Treatments 3 and 4, used in this study, were the most suitable, considering the CI obtained, of 69.2 and 66%, respectively. It should be noted that fibers with high crystallinity can provide greater reinforcement in composite materials (Karthik *et al.*, 2024). Compared to samples T1 and T2, it can be seen that the results discussed in the previous section, on FTIR spectroscopy, are corroborated by the higher CI of T3 and T4, confirming the effectiveness of treatments with NaOH and NaOH-ethanol, respectively, for the removal of non-cellulosic components such as lignin and hemicellulose, in addition to amorphous portions of cellulose. These findings support the development of simpler, efficient, and more sustainable nanocellulose production routes from *Phormium tenax*.

4 CONCLUSIONS

Four treatments with different solvents (water, ethanol, and NaOH) were tested under controlled conditions of pressure, agitation, and temperature, aiming to remove lignin in a sustainable manner. FTIR analysis showed that treatments with NaOH (T3 and T4) were more effective in removing lignin, allowing to obtain CNF in gel form. Microscopy images (SEM and TEM) revealed dense and homogeneous networks of nanofibers, especially

in sample T4, with diameters between 19 and 28 nm. The crystallinity of the treated cellulose increased significantly (69.2% for T3 and 66.0% for T4), compared to the original fiber (36.7%). It is concluded that treatments with NaOH and NaOH/ethanol combined to elevated pressure and temperature (140 °C), are efficient, simple and more sustainable for obtaining high-quality CNF from *Phormium tenax*, with potential for use in composite materials. This work contributes both scientifically and technologically to the advancement of greener strategies for nanocellulose production from alternative natural fibers.

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