



Biodegradable nanocomposites from wolf fruit: reinforcement of starch films with cellulose nanofibers extracted from peel waste

Amanda Lélis Souza · Letícia Moraes Costa Teixeira · Maria Betânia de Freitas-Marques · Sandra de Oliveira Silva · Guilherme Carneiro · Franciele Maria Pelissari

Received: 28 July 2025 / Accepted: 14 June 2026 / Published online: 22 June 2026
© The Author(s) 2026

Abstract Cellulose nanofibers (CNFs) were extracted from the peel of *Solanum lycocarpum* (wolf fruit) using sulfuric acid hydrolysis at concentrations of 0.1%, 1%, and 10%. These nanofibers were incorporated into starch-based films produced via casting, and the resulting materials were analyzed for their structural, mechanical, thermal, and optical characteristics. The addition of CNFs led to significant enhancements in tensile strength, Young's modulus, and opacity, along with a reduction in moisture content. Among the formulations, the film reinforced with CNFs from the 0.1% acid treatment exhibited the best mechanical performance (tensile strength = 7.45 MPa;

Young's modulus = 471.57 MPa), attributed to improved nanofiber dispersion and stronger interactions within the starch matrix. In contrast, nanofibers obtained using higher acid concentrations (1% and 10%) showed partial structural degradation, resulting in nanocomposites with reduced tensile strength compared to F0.1—likely due to reduced hydrogen bonding between CNFs and starch chains. Additionally, the use of 0.1% H₂SO₄ is favorable in terms of environmental and cost considerations, reducing both reagent usage and energy demand during processing. Overall, the study demonstrates the feasibility of producing fully bio-based nanocomposites by utilizing both starch and cellulose nanofibers derived from the same fruit, offering a sustainable solution for biodegradable packaging applications.

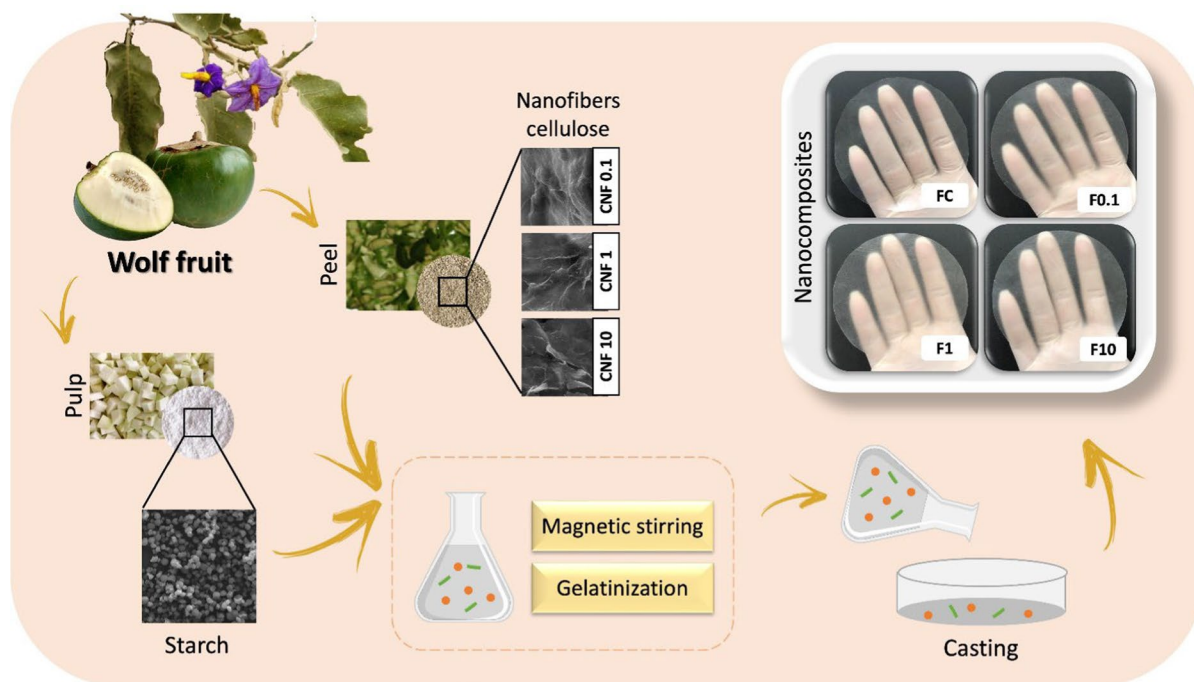
Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s10570-026-07115-y>.

A. L. Souza · L. M. C. Teixeira · S. O. Silva ·
F. M. Pelissari (✉)
Laboratory of Green Materials, Institute
of Science and Technology, Federal University
of Jequitinhonha and Mucuri Valleys, Diamantina,
Minas Gerais CEP 39100-000, Brazil
e-mail: franciele.pelissari@ict.ufvjm.edu.br

M. B. de Freitas-Marques
Department of Chemistry, Federal University of Minas
Gerais, Belo Horizonte, Minas Gerais CEP 31270-901,
Brazil

G. Carneiro
Department of Pharmacy, Faculty of Biological and Health
Sciences, Federal University of Jequitinhonha and Mucuri
Valleys, Diamantina, Minas Gerais CEP 39100-000, Brazil

Graphical Abstract



Keywords Sustainable materials · Acid hydrolysis · Natural polymer reinforcement · Agro-industrial valorization · Brazilian Cerrado

Introduction

Synthetic polymers, widely used in food packaging, are valued for their mechanical strength, low cost, lightweight nature, and ease of processing (Kim et al. 2020). However, their environmental persistence and resistance to degradation have raised concerns about pollution and sustainability. In this context, biodegradable alternatives such as starch have drawn attention due to their natural abundance, renewability, and environmental compatibility (Soni et al. 2020). Despite these advantages, starch-based films often suffer from drawbacks like high water sensitivity and limited mechanical flexibility, which restrict their broader application.

A promising strategy to overcome these limitations involves the application of nanotechnology to develop

nanocomposites incorporating nanostructured reinforcements. These materials exhibit improved physical and functional properties, including enhanced mechanical performance and barrier characteristics, often surpassing those of conventional polymer composites (Abdul Khalil et al. 2012; Pelissari et al. 2017).

Among the nanostructured reinforcements, cellulose-based nanomaterials stand out due to their biodegradability, availability from diverse sources (plants, bacteria, fungi), and cost-effectiveness. Cellulose nanofibers (CNFs), in particular, are extracted from plant biomass and exhibit impressive strength and stiffness, comparable to some synthetic fibers (Balasubramaniam et al. 2020). One of the most common techniques for CNF isolation is acid hydrolysis using sulfuric acid (H_2SO_4), which not only breaks the cellulose chains but also introduces sulfate groups, improving dispersion in aqueous media (Harahap et al. 2025; Siqueira et al. 2010; Tibolla et al. 2020).

Agricultural residues have emerged as an attractive and renewable source of cellulose for CNF production. Research has highlighted the use of agro-industrial waste—such as pineapple leaves (Cherian et al. 2011), wheat straw (Kaushik and Singh 2011), banana peels (Pelissari et al. 2017), cassava bagasse (Travalini et al. 2019), citrus peels (Soofi et al. 2021) and oil palm empty fruit bunches (Gea et al. 2020)—as viable raw materials for reinforcing polymer matrices. The integration of these nanofibers into biodegradable polymers contributes to improved structural integrity, reduced moisture absorption, and preservation of environmental benefits (Pelissari et al. 2017).

Fruit processing byproducts, due to their high volume and cellulosic content, represent a strategic source of raw material for sustainable nanocomposites. Globally, over 800 million tons of dry agricultural waste are produced annually (Kassab et al. 2020). In Brazil, the wolf fruit (*S. lycocarpum*), native to the Cerrado biome, generates a lignocellulosic peel residue during pulp extraction, which presents potential as a reinforcing agent in bio-based materials (Souza et al. 2025). Additionally, the fruit contains significant starch content—ranging from 1.8% to 20% depending on harvest and maturity stage (Pascoal et al. 2013)—which makes it a candidate for film-forming matrices.

Recent work has shown that it is possible to isolate CNFs from wolf fruit peel using varying concentrations of sulfuric acid (Souza et al. 2025). Building upon this advancement, the present work introduces an innovative and fully integrated approach to the valorization of *S. lycocarpum*, a native species of the Brazilian Cerrado. In the present study, both the pulp and peel of this fruit were utilized to develop a fully integrated nanocomposite system: starch extracted from the pulp served as the film-forming polymer, while CNFs from the peel acted as the reinforcing phase. The proposed nanocomposite promotes circularity and the integral utilization of natural resources, while also highlighting the Cerrado's biodiversity as a promising and renewable source for the development of sustainable materials. The resulting films were evaluated for their mechanical, barrier, thermal, structural, and optical properties. To our knowledge, this is the first report of starch-based biodegradable films entirely derived from wolf fruit, integrating its own starch and nanocellulose components into a

Table 1 Formulation of control and nanocomposite films

Sample	Wolf fruit starch (g/100 g of film solution, dry basis)	Glycerol concentration (g/100 g of starch)	CNF content (g/100 g of starch, dry basis)	Acid concentration used for CNF extraction (H ₂ SO ₄ , w/w)
FC	2.5	22.5	0	–
F0.1	2.5	22.5	5	0.1%
F1	2.5	22.5	5	1%
F10	2.5	22.5	5	10%

single bio-based system with enhanced functional performance.

Material and methods

Material

Wolf fruits (*S. lycocarpum* A. St.-Hill) were harvested during the green maturation stage in January 2019, in the district of Conselheiro Mata, Diamantina, Minas Gerais, Brazil (coordinates: – 18.228930, – 43.610849). After collection, the fruits were cleaned and refrigerated at 4 °C until further processing for starch extraction. All chemicals utilized in the study were of analytical grade.

Starch extraction

The extraction of starch from wolf fruit pulp was performed following a procedure adapted from Louzada et al. (2021). Initially, the fruits were washed, peeled, and had their seeds removed. The pulp was cut into pieces and treated with a 1% (w/v) potassium metabisulfite solution for 15 min to inhibit enzymatic browning. Subsequently, the treated pulp was blended with distilled water in a ratio of 1:3 (pulp:water), followed by sequential sieving through 65 and 100 mesh screens to separate the starch milk from insoluble residues. The starch suspension was left to settle for 3 h at room temperature. The precipitated starch was washed with 70% ethanol to remove non-starch components and then dried in a forced-air oven (TE-394/2, Tecnal Equipamentos Científicos Ltda) at 45 °C for 48 h. The dried starch was ground using a

knife mill (226,172, Lucadema Científica Ltda) and stored at 4 °C in airtight containers.

Proximate composition of wolf fruit starch

The proximate composition of the starch was determined following AOAC methods (Latimer 2023). Moisture content was assessed by drying at 105 °C until constant weight. Ash content was measured by incinerating 2 g of dry sample in a muffle furnace (N480, Novus) at 550 °C. Protein content was estimated using the Macro-Kjeldahl method with a nitrogen-to-protein conversion factor of 6.25. Lipid content was determined according to the Bligh and Dyer (1959) method, while starch content was quantified using the Lane-Eynon titration approach (Lane 1923). All analyses were conducted in triplicate.

Isolation of cellulose nanofibers

Cellulose nanofibers were obtained from wolf fruit peel according to the method established by Souza et al. (2025). The process involved acid hydrolysis using sulfuric acid solutions at three concentrations: 0.1%, 1%, and 10% (w/w). The CNF suspensions obtained after hydrolysis presented fiber concentrations of 0.70 g/100 g (CNF 0.1), 0.79 g/100 g (CNF 1), and 0.78 g/100 g (CNF 10). These concentrations were taken into account when formulating the nanocomposite films.

The nanofibrillar morphology of the CNFs was previously confirmed by scanning electron microscopy (SEM). It is important to note that SEM has limited resolution for visualizing individualized nanofibers, and therefore the observed structures should be interpreted as nanofibrillar assemblies rather than fully individualized fibrils. The fiber diameters were determined using ImageJ® software based on 50 independent measurements, yielding average diameters of 158, 124, and 93 nm for nanofibers hydrolyzed with 0.1%, 1%, and 10% H₂SO₄, respectively (Souza et al. 2025). A representative SEM image with diameter annotation is provided in Supplementary Material 1. These values are consistent with the size range commonly reported for cellulose nanofibers, which may extend beyond 100 nm depending on the biomass source, extraction conditions, and degree of fibrillation (Abdul Khalil et al. 2012; Siqueira et al. 2010; Tibolla et al. 2020). Indeed, several studies have

reported CNFs with diameters exceeding 100 nm, particularly when derived from lignocellulosic residues or obtained under milder chemical treatments, while still maintaining characteristic nanofibrillar morphology and reinforcing behavior (Kaushik and Singh 2011; Yang et al. 2020; Pelissari et al. 2017).

Production of nanocomposites

Film-forming solutions were prepared by dispersing the CNF suspensions in distilled water. For each formulation (Table 1), 5 g of nanofibers per 100 g of starch (dry basis) were incorporated. Initially, the suspensions were stirred magnetically (TE-0851, Tecnal Equipamentos Científicos Ltda) for 20 h to ensure adequate dispersion. The starch solution (2.5% w/w, dry basis) was separately prepared and homogenized for 15 min before blending with the CNFs. After mixing, the combined solution was subjected to mechanical stirring for 30 min and then heated to 80 °C.

Once the target temperature was reached, glycerol was added at a proportion of 22.5 g per 100 g of starch as a plasticizer. The mixture was maintained at 80 °C for 15 min to complete gelatinization. Afterwards, 100 g aliquots of the film-forming solution were poured onto Teflon-coated plates (20 cm diameter) and dried at 53 °C in a forced-air oven (TE-394/2, Tecnal Equipamentos Científicos Ltda). A control film without CNFs was also produced following the same procedure (see Supplementary Material 1).

All films were conditioned at 25 °C and 58% relative humidity for 48 h, except for samples used in microstructural, FTIR, and XRD analyses, which were dehydrated over silica gel (~0% RH) for three weeks.

Characterization of nanocomposites

Microstructural analysis

Surface and cross-sectional morphology of the films was examined by scanning electron microscopy (SEM). Film samples were fractured manually, fixed on aluminum stubs with double-sided tape, and coated with a thin gold layer using a sputter coater (SDC050, Polaron). SEM imaging was performed at 10 kV using a TESCAN VEGA-LMH microscope at

the Multiuser Laboratory of Advanced Microscopy (LMMA, UFVJM).

Thickness, density, and moisture content

Film thickness was measured with a digital micrometer (Mitutoyo, Series 293 MDC-Lite, Japan) with 0.1 μm precision, using ten random points per sample. Density (ρ) was calculated based on mass-to-volume ratio from 20 $\times$ 20 mm film samples, dried at 105 $^{\circ}\text{C}$ for 24 h (Eq. 1):

$$\rho = \frac{m}{A \times L} \quad (1)$$

where (m) is the mass (g), (A) is the area (cm^2), and (L) is the thickness (cm) of the film.

Moisture content was determined gravimetrically following ASTM D644-99 (ASTM International 2007), by drying the samples at 105 $^{\circ}\text{C}$ until constant weight.

Water solubility

Water solubility was determined using the method described by Gontard et al. (1992), with modifications. Film discs (1.9 cm diameter) of known dry mass were immersed in 50 mL distilled water at 25 $^{\circ}\text{C}$ for 24 h under intermittent stirring. After soaking, the undissolved residue was filtered, dried at 105 $^{\circ}\text{C}$ for 24 h, and weighed. The percentage of solubilized material was calculated from the difference between initial and final dry mass.

Mechanical properties

Mechanical performance was evaluated using a texture analyzer (TA.TX Plus, Stable Micro Systems, UK) following ASTM D882-12 (ASTM International 2012). Film strips (20 $\times$ 80 mm) were tested with an initial grip separation of 70 mm at a constant speed of 1 mm/s until rupture. Six replicates were performed for each sample to determine tensile strength, elongation at break, and Young's modulus.

Optical properties

The color properties of the nanocomposite films were analyzed using a CM-5 colorimeter (Konica

Minolta) operating in transmittance mode, under standard conditions of D65 illumination (representing daylight) and a 10 $^{\circ}$ observation angle. Measurements were conducted in quadruplicate, and results were expressed within the CIELab system (Hunterlab 1996), reporting L^* (lightness, where 0 = black and 100 = white), a^* (green–red axis, with positive values indicating red), and b^* (blue–yellow axis, where positive values correspond to yellow tones). The total color difference (ΔE^*) compared to a polyethylene reference film was calculated using Eq. 2:

$$\Delta E = \left[(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2 \right]^{1/2} \quad (2)$$

where L^* , a^* , and b^* are the measured values for each film sample, while L_0^* , a_0^* , and b_0^* refer to the corresponding parameters of the standard reference material.

Film opacity was also determined following the protocol described by Núñez-Flores et al. (2012). Samples were carefully cut and placed against the inner face of a spectrophotometer cuvette, ensuring full coverage without folds. Absorbance was measured at 600 nm using a UV-5100 model spectrophotometer (Tecnal Equipamentos Científicos Ltda), with a blank reading taken from an empty cuvette. Opacity was calculated using Eq. 3:

$$\text{Opacity} = \frac{A_{600}}{X} \quad (3)$$

where A_{600} is the absorbance value at 600 nm, and X is the thickness of the film expressed in millimeters.

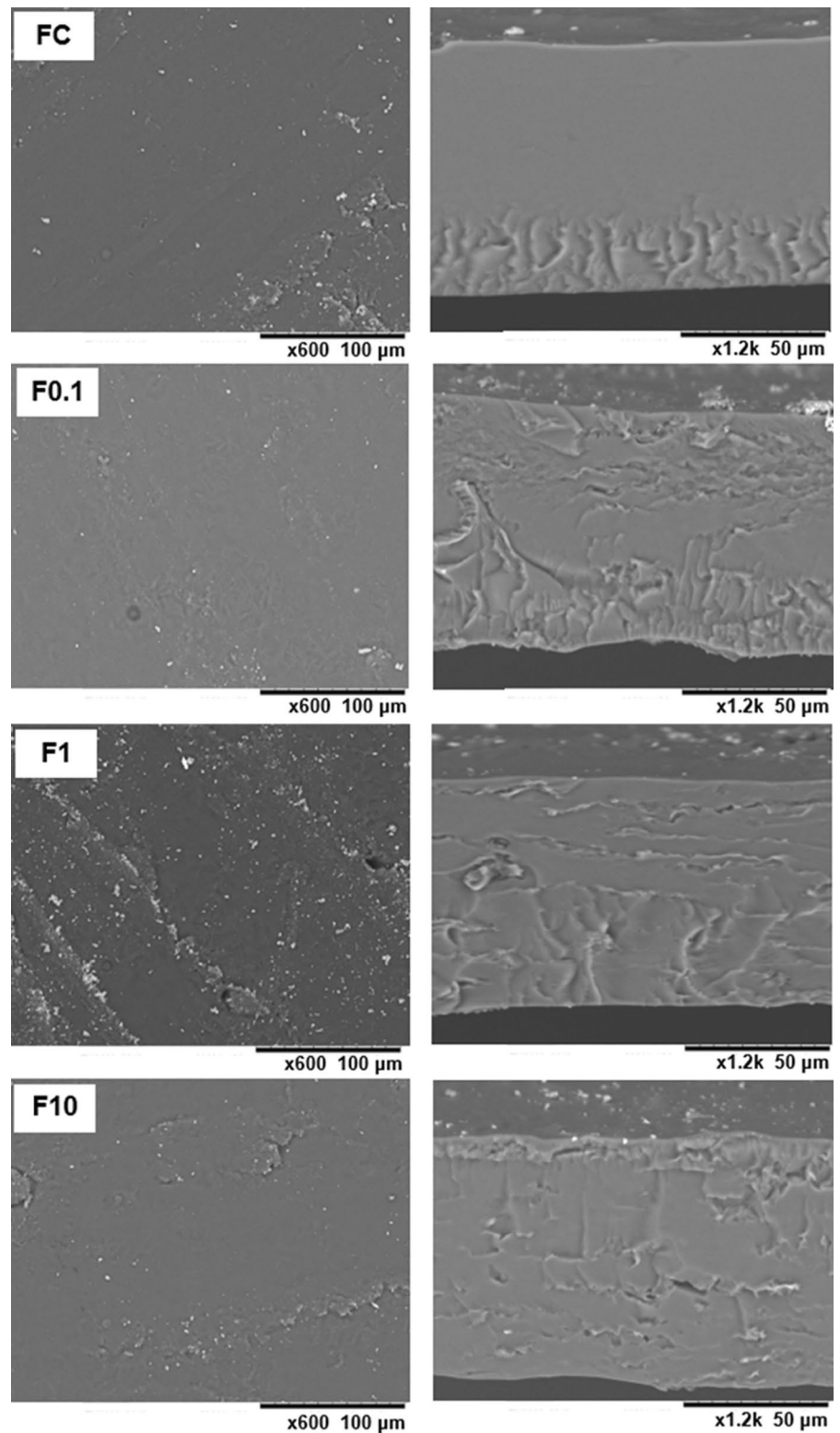
X-ray diffraction (XRD)

XRD patterns were recorded using a Shimadzu RXD6000 diffractometer with $\text{Cu-K}\alpha$ radiation at

Table 2 Starch content of wolf fruit and other botanical starches (wet basis)

Starch source	Starch content (%)	Reference
Wolf fruit	75.9	Present study
Barley	67.9	Hor and Gastl (2025)
Potato	87.4	Zhang et al. (2025)
Corn	91.0	Li et al. (2025)
Rice	93.0	Martinez-Munoz et al. (2025)
Cassava	78.8	Qi et al. (2024)

Fig. 1 Surface and cross-sectional microstructure of the wolf fruit starch-based control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4



40 kV and 30 mA. Scans were performed over a 2 θ range of 10–50° at a scanning rate of 1.2°/min. XRD patterns were qualitatively evaluated to identify structural features associated with starch and cellulose in the films. The interpretation focused on the presence, position, and relative intensity of diffraction features. The XRD graphical data used in this study are provided in Supplementary Material 2.

Fourier transform infrared spectroscopy (FTIR)

FTIR spectra were obtained with an Agilent Cary 630 spectrometer equipped with an ATR accessory. Measurements were conducted from 4000 to 650 cm⁻¹, with a resolution of 4 cm⁻¹ and 64 scans per sample.

Thermal analysis

The thermal behavior of the nanocomposites was evaluated using differential scanning calorimetry (DSC) and thermogravimetric analysis (TG)/derivative thermogravimetry (DTG). DSC curves were obtained using a Shimadzu® DSC60 cell, calibrated with Indium (melting: $T_{\text{onset}} = 156.63$ °C, $\Delta H_m = 28.45$ J g⁻¹), under a dynamic nitrogen atmosphere at a flow rate of 50 mL min⁻¹. The heating rate was set to 10 °C min⁻¹, within a temperature range of 30 to 400 °C, using a sealed aluminum crucible with an exact sample mass of approximately 1.5 mg.

TG/DTG curves were obtained using a Shimadzu® DTG60 thermobalance, with a heating rate of 10 °C min⁻¹, within a temperature range of 30 to 600 °C, under a dynamic nitrogen atmosphere at a flow rate of 50 mL min⁻¹. Analyses were conducted in an alumina crucible with an exact sample mass of approximately 2.5 mg.

TA60 software version 2.30 SP1 of manufacturer was used to calculate the enthalpy of the phenomena (ΔH), mass variation (Δm), and first derivative of the TG curves.

Statistical analysis

The mean properties of starch films reinforced with CNFs obtained from different sulfuric acid concentrations were compared through analysis of variance (ANOVA), followed by Tukey's post-hoc test at a 5% significance level. All statistical analyses were conducted using Statistica 7.0 software (StatSoft Inc., Tulsa, OK, USA).

Results and discussion

Proximate composition of wolf fruit starch

The dry basis starch yield was 6.5%, aligning with values previously reported in the literature, such as 7.4% by Louzada et al. (2021) and 4.8% by Clerici et al. (2011). The proximate composition of the extracted wolf fruit starch (wet basis) was as follows: moisture 7.61% $\pm$ 0.07, protein 0.87% $\pm$ 0.04, lipids 0.02% $\pm$ 0.03, ash 0.17% $\pm$ 0.02, and starch content 75.90% $\pm$ 0.14. In comparison, Clerici et al. (2011) reported higher values for moisture (12.15%), protein (6.27%), ash (0.38%), and lipids (0.18%). These differences are likely related to factors such as harvest season, extraction procedures, and the ripening stage of the fruit during processing. Notably, unripe fruits generally exhibit higher starch content.

To better contextualize these findings, Table 2 compares the starch content of wolf fruit with other common starches reported in the recent literature.

Table 3 Thickness, density, moisture content, and water solubility of the control film (CF) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H₂SO₄

Sample	Thickness (mm)	Density (g/cm ³)	Moisture content (%)	Water solubility (%)
FC	0.14 $\pm$ 0.01 ^a	0.86 $\pm$ 0.02 ^a	21.34 $\pm$ 0.19 ^a	22.22 $\pm$ 1.48 ^a
F0.1	0.15 $\pm$ 0.01 ^a	0.82 $\pm$ 0.01 ^a	20.74 $\pm$ 0.28 ^{ab}	19.13 $\pm$ 1.23 ^a
F1	0.15 $\pm$ 0.01 ^a	0.82 $\pm$ 0.04 ^a	20.50 $\pm$ 0.41 ^b	20.31 $\pm$ 1.11 ^a
F10	0.15 $\pm$ 0.01 ^a	0.83 $\pm$ 0.01 ^a	20.87 $\pm$ 0.15 ^{ab}	22.01 $\pm$ 1.34 ^a

^{a, b}Means with different superscripts in the same column indicate a statistical difference ($p < 0.05$)

As shown in Table 2, the starch content of wolf fruit (75.9%) is comparable to cassava (78.8%), a widely used source of industrial starch. It is moderately lower than that of potato (87.4%), corn (91.0%), and rice (93.0%), which are among the most starch-rich crops. However, wolf fruit presents a clear advantage over barley (67.9%), indicating its viability as an alternative starch source. These comparisons highlight that wolf fruit offers a competitive starch yield, supporting its potential for applications in biodegradable film production.

Nanocomposite characterization

The nanocomposite films produced with wolf fruit starch and CNFs from its peel showed uniform surfaces without visible cracks or air bubbles. All films exhibited good mechanical integrity, were easily removed from the casting plates without tearing, and did not display stickiness or excessive brittleness. During manual bending in both directions, no cracking or breaking was observed.

Scanning electron microscopy

Figure 1 presents the micrographs of the film surfaces and cross-sections. The absence of visible starch granules suggests that the casting process achieved complete gelatinization.

The incorporation of CNFs notably affected the internal structure of the nanocomposite films (F0.1, F1, and F10) when compared to the control sample (FC). While the control film exhibited a smooth and uniform surface, the nanocomposites displayed a more irregular and heterogeneous morphology with surface imperfections. In cross-sectional views, the nanocomposites revealed a less dense and more

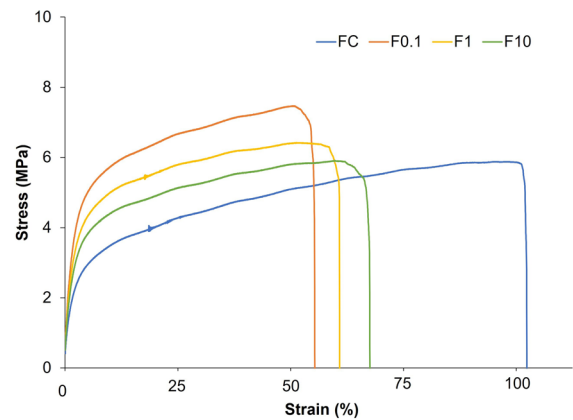


Fig. 2 Typical tensile test curves for the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

porous structure, with the presence of small fissures, contrasting with the denser appearance of the control film. These morphological features are consistent with partially fibrillated cellulose, as expected for mild chemical treatments, which typically result in heterogeneous fibrillar structures rather than fully individualized nanofibers.

The acid hydrolysis conditions, varying in H_2SO_4 concentration, resulted in CNFs with distinct morphological features (Souza et al. 2025), which influenced the film microstructure (Fig. 1) and the interaction between cellulose and the starch-based polymer matrix, along with glycerol and water components (Tibolla et al. 2020).

Despite the morphological differences, SEM analysis indicated good dispersion of the nanofibers within the matrix, with minimal signs of nanoparticle agglomeration, suggesting effective integration. This outcome is likely due to hydrogen bonding between

Table 4 Mechanical properties of the control film (CF) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

Sample	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (MPa)
FC	5.19 ± 0.34^c	89.33 ± 0.22^a	274.38 ± 13.05^b
F0.1	7.45 ± 0.54^a	49.52 ± 0.18^b	471.57 ± 23.41^a
F1	6.47 ± 0.50^{ab}	53.09 ± 0.49^b	438.95 ± 39.58^a
F10	6.35 ± 0.56^b	58.83 ± 0.13^b	429.15 ± 16.63^a

^{a, b, c} Means with different superscripts in the same column indicate a statistical difference ($p < 0.05$)

hydroxyl groups in the cellulose and polar sites within the starch polymer chains (Azeredo et al. 2009). Similar structural patterns were previously observed by Kaushik et al. (Kaushik et al. 2010) for wheat straw CNFs in thermoplastic starch and by Travalini et al. (2019) when using cassava bagasse-derived CNFs in starch-based films.

Thickness, density, and moisture

Table 3 shows the measurements of thickness, density, and moisture content for all film samples. The comparable thickness values across the different formulations reflect the strict control applied to the dry mass per area during film casting.

The incorporation of CNFs did not cause statistically significant variations in film density ($p \geq 0.05$, Table 3). Nevertheless, SEM images (Fig. 1) indicated that CNF addition contributed to a more porous and less compact internal structure, which may explain the slight reduction in density values observed.

Regarding moisture content, no significant differences ($p \geq 0.05$) were detected among the samples, with the exception of film F1, which exhibited a lower moisture level following CNF incorporation. This decrease could be attributed to the higher water-binding capacity of starch relative to the nanofibers. A similar trend was described by Ortega et al. (2021) in studies with corn starch films containing silver nanoparticles, where moisture content remained mostly unaffected.

Water solubility

The nanocomposite films showed a tendency towards reduced water solubility compared to the control; however, the differences among the samples were

not statistically significant ($p \geq 0.05$, Table 3), even considering the variations in microstructure (Fig. 1) and the higher moisture content observed in the control film. The incorporation of 5% CNFs was not sufficient to cause a significant decrease in solubility levels.

This behavior is probably related to strong water affinity with the hydrophilic components of the matrix, such as starch and cellulose, as well as possible structural changes like increased tortuosity, which can hinder water migration through the material.

Several factors affect the water solubility of polymeric films, including the origin and characteristics of the raw materials, the nature and intensity of molecular interactions within the matrix, the presence and concentration of plasticizers, processing conditions, and the storage or conditioning environment before testing (Pelissari et al. 2013; Galus and Kadzińska 2015; Wang et al. 2015). Therefore, understanding and controlling these variables is essential for tailoring the solubility behavior of biodegradable films.

Mechanical properties

The tensile properties of the films, including tensile strength, elongation at break, and Young's modulus, are summarized in Table 4, with the corresponding stress–strain curves illustrated in Fig. 2.

Incorporation of CNFs into the starch matrix led to a noticeable improvement in tensile strength and stiffness (as indicated by Young's modulus), while reducing film flexibility, as shown by the lower elongation at break values. The F0.1 formulation, in particular, showed a 44% increase in tensile strength and a 72% rise in Young's modulus when compared to the control film. This enhancement may be associated with improved stress transfer promoted by effective

Table 5 Optical properties of the control film (CF) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

Sample	L^*	a^*	b^*	ΔE^*	Opacity
FC	97.48 ± 0.14^c	0.07 ± 0.01^c	0.66 ± 0.02^c	3.16 ± 0.14^c	1.91 ± 0.01^b
F0.1	98.57 ± 0.05^a	0.15 ± 0.01^a	0.93 ± 0.04^b	4.27 ± 0.04^a	2.97 ± 0.06^a
F1	98.05 ± 0.13^b	0.10 ± 0.01^b	1.03 ± 0.07^{ab}	3.83 ± 0.10^b	3.06 ± 0.08^a
F10	98.05 ± 0.08^b	0.12 ± 0.01^b	1.10 ± 0.05^a	3.86 ± 0.09^b	3.06 ± 0.09^a

^{a, b, c} Means with different superscripts in the same column indicate a statistical difference ($p < 0.05$)

Fig. 3 Appearance of the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

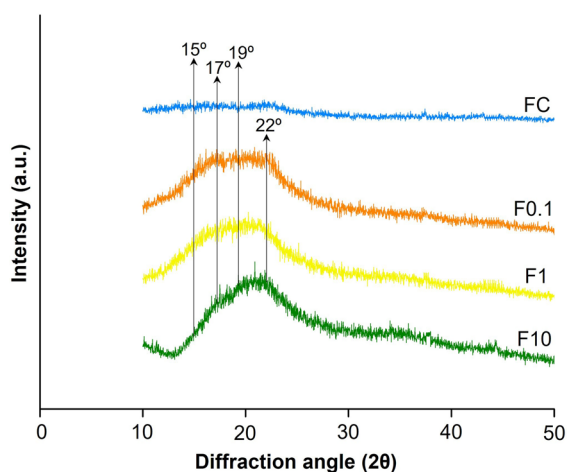
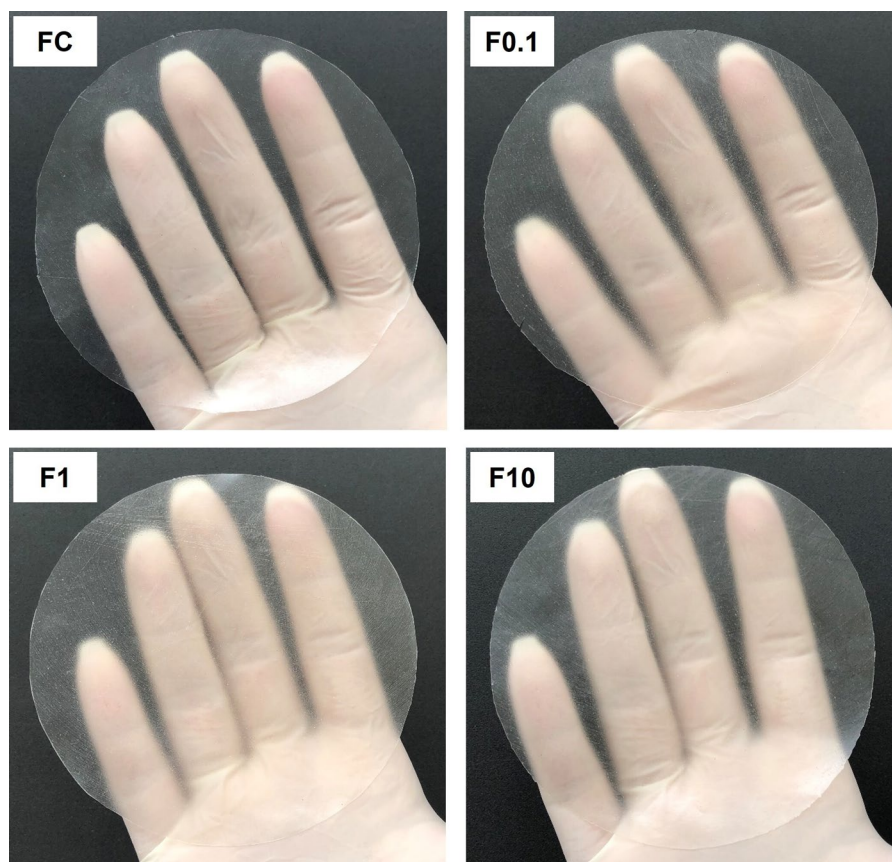


Fig. 4 X-ray diffraction pattern of the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

nanofiber dispersion and stronger interfacial interactions between CNFs and the starch matrix (Li et al. 2018). These results were supported by the typical profiles of the stress–strain curves, which confirmed the higher flexibility of the control sample and the superior mechanical performance of the nanocomposites. Similar trends have been reported in other studies involving CNF-reinforced biodegradable films (Li et al. 2018; Yang et al. 2020; Tibolla et al. 2020).

The lower sulfuric acid concentration (0.1%) used during hydrolysis proved to be the most effective condition for improving mechanical performance. The F0.1 film achieved the highest values of tensile strength and modulus of elasticity, which can be associated with its more negative zeta potential (−19.3 mV) compared to CNF 1 (−17.2 mV) and CNF 10 (−10.5 mV). This negative zeta potential arises from sulfate ester groups ($-\text{OSO}_3^-$) introduced onto the CNF surface during sulfuric acid hydrolysis, which impart electrostatic repulsion and improve colloidal stability. A higher zeta potential indicates

better colloidal stability, minimizing fiber aggregation and promoting a well-dispersed, interconnected nanofiber network within the starch matrix (Yang et al. 2020). Future studies should investigate whether sulfuric acid concentrations lower than 0.1% could further enhance the mechanical properties of the nanocomposites, while preserving the environmentally friendly nature of the process.

Conversely, films reinforced with CNFs obtained from higher acid concentrations (F1 and F10) exhibited lower mechanical strength and stiffness compared to F0.1. This reduction can be attributed to the more severe degradation of cellulose crystals promoted by stronger acid hydrolysis (Souza et al. 2025), which compromised the reinforcing ability of the nanofibers. As previously described in our related work (Souza et al. 2025), higher acid concentrations not only reduced fiber length but also altered the crystalline structure of CNFs, thereby limiting their interfacial interactions with the starch matrix. These structural modifications are consistent with the weaker reinforcement effect observed in F1 and F10 films, highlighting the importance of controlling hydrolysis severity to preserve CNF performance in nanocomposite systems.

Optical properties

All color parameters showed statistically significant differences between the control film and the nanocomposites ($p < 0.05$; Table 5). Figure 3 illustrates

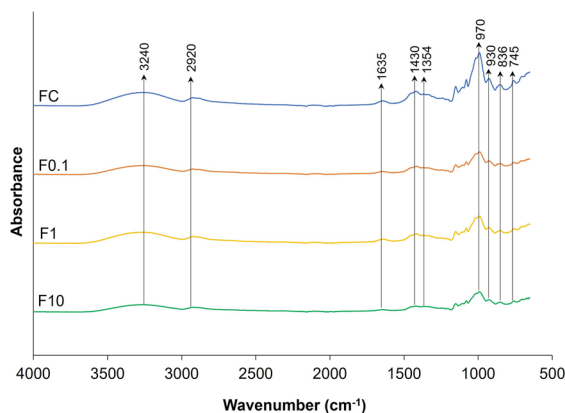


Fig. 5 FTIR spectra of the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

the appearance of the nanocomposites reinforced with CNFs isolated from wolf fruits.

Based on the L^* , b^* , and ΔE^* values, incorporating CNFs resulted in lighter, more yellowish nanocomposites with less uniform color than the control film. The a^* parameter values were positive and close to zero for all films, indicating the absence of characteristic green tones.

The control film was more transparent than the nanocomposites (Table 5), confirming that reinforcement with CNFs increased opacity by approximately 55–60%, probably due to the random distribution of nanoparticles within the polymer matrix. The opacity values align with the methodology used, as Núñez-Flores et al. (2012) reported values ranging from 0.39 to 1.98 for commercial fish skin gelatin films with eucalyptus wood lignosulfonate.

Visual evaluation further confirmed that the control film exhibited greater transparency and gloss than the nanocomposites, reinforcing the observation that adding CNFs increased film opacity. The light transmittance of the control film and nanocomposites within the 200–800 nm wavelength range was as follows: 62.95 nm for FC, 40.09 nm for F0.1, 41.98 nm for F1, and 41.50 nm for F10. Visible light spans wavelengths from 380 to 780 nm, while ultraviolet (UV) radiation covers wavelengths from 200 to 380 nm.

The control film exhibited higher light transmittance in the UV region compared to the nanocomposites. This behavior can be attributed to the presence of nanofillers, which hinder light transmission through the polymer matrix. The reduction in light transmittance depends on the filler characteristics, as the fine dispersion and high aspect ratio of CNFs promote strong interfacial interactions with the polymer matrix (Tibolla et al. 2020). These results are consistent with the opacity values observed for the nanocomposites, indicating that more opaque films act as more effective barriers against light and UV radiation. Therefore, the use of such materials represents a promising strategy for preserving products susceptible to lipid oxidation, as they provide an additional barrier against light, one of the main factors that accelerate oxidative processes.

Fig. 6 DSC analysis of the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4

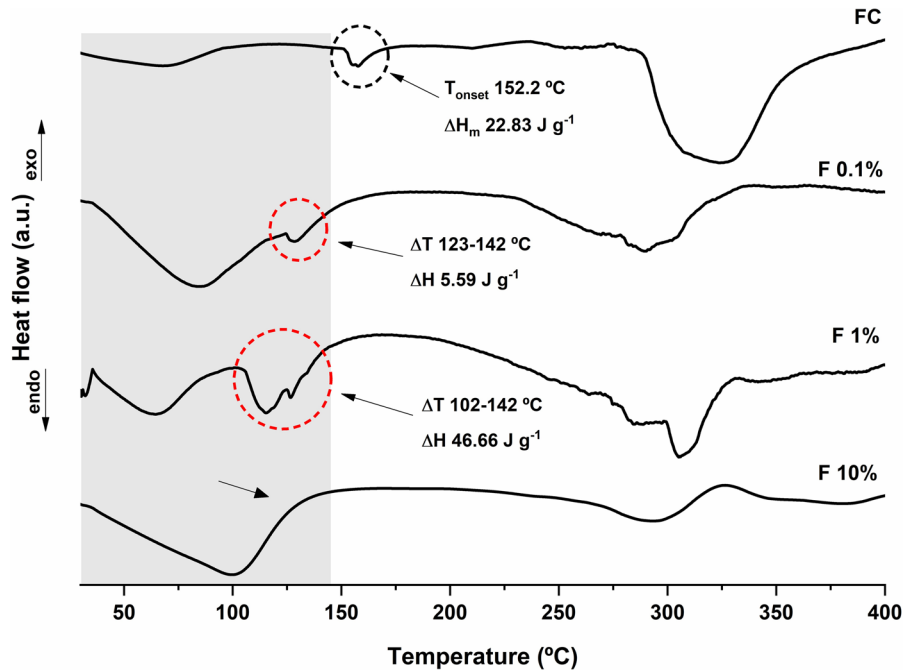
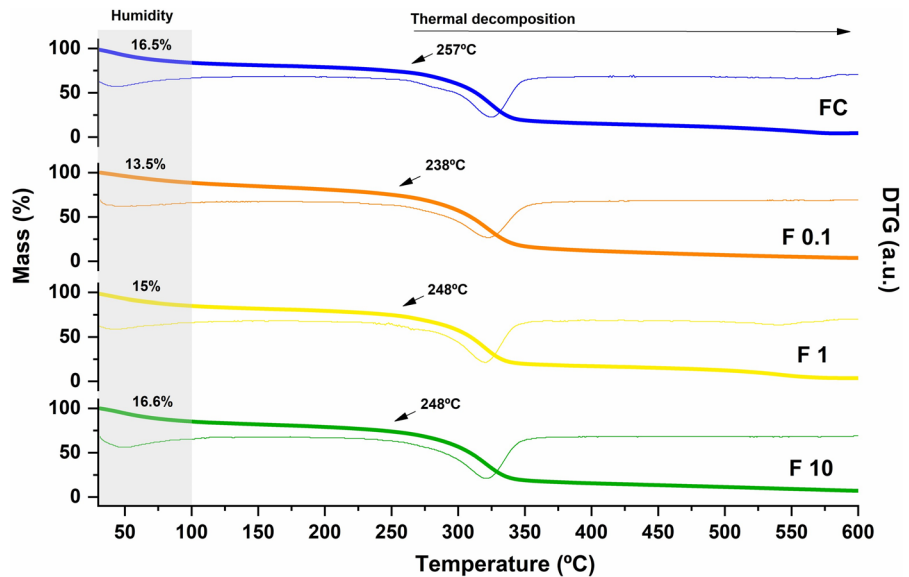


Fig. 7 Thermogravimetric analysis (TG) and derivative thermogravimetry (DTG) curves of the control film (FC) and nanocomposites reinforced with cellulose nanofibers obtained via acid hydrolysis using 0.1% (F0.1), 1% (F1), and 10% (F10) H_2SO_4



X-ray diffraction (XRD)

XRD patterns for both the control wolf fruit starch film and the CNF-reinforced nanocomposites are shown in Fig. 4. The interpretation of the XRD patterns focused on the $10^\circ\text{--}50^\circ$ region, where the main diffraction features associated with starch and cellulose structures are observed. In this range, the

control film (FC) exhibited weak and broad diffraction features around $2\theta \approx 15^\circ$, 17° , and 19° , which are typically associated with type-C starch crystallinity. These low-intensity and broad diffraction features are consistent with a predominantly amorphous structure, although the presence of short-range ordered domains or very small crystallites cannot be excluded. This behavior is likely related to extensive

gelatinization during film formation, which disrupts the native crystalline arrangement of starch (Pascoal et al. 2013). Such structural disruption is commonly observed in thermoplastic starch-based films, where the reorganization of starch chains after gelatinization (retrogradation) is limited and does not lead to highly defined crystalline peaks (Pelissari et al. 2017; Cao et al. 2008).

With the incorporation of CNFs, the diffraction features became more evident compared to the control film, indicating modifications in the structural organization of the nanocomposites. It is important to note that the observed changes may also be influenced by differences in nanofiber organization, dispersion, and aggregation state, rather than solely by intrinsic crystallinity. Additionally, a diffraction feature at $2\theta \approx 22^\circ$, associated with cellulose I (Yang et al. 2020), became evident in the nanocomposite films and was absent in the control sample. This observation suggests that the CNFs retained structural features associated with cellulose I within the polymer matrix (Robles-García et al. 2018).

Small variations in the relative intensity of the cellulose-related diffraction features were observed among the nanocomposites prepared with CNFs obtained under different hydrolysis conditions, suggesting that the extraction conditions influenced the structural organization of the cellulose domains.

Fourier transform infrared spectroscopy (FTIR)

FTIR analysis was performed to investigate the types of molecular interactions present in the pure starch film and in the nanocomposite samples. The resulting spectra and characteristic bands are shown in Fig. 5. The overall spectral profiles of the nanocomposites were similar to that of the control film (FC), reflecting the chemical resemblance between starch and cellulose, as well as the relatively low CNF content (5% w/w) incorporated into the matrix.

All samples exhibited a broad absorption peak near 3240 cm^{-1} , related to O–H stretching vibrations, characteristic of starch molecules (Sutrisno et al. 2020). The band observed around 2920 cm^{-1} corresponds to C–H stretching, indicating the presence of glycerol used as a plasticizer (Ma et al. 2017).

Absorption bands at 1635 cm^{-1} and 1430 cm^{-1} were associated with water retained in the films (O–H

bending) and C–H₂ bending vibrations, respectively (Prachayawarakorn et al. 2013; Patel et al. 2015). In the nanocomposite spectra, a distinct peak at 1354 cm^{-1} appeared, linked to the bending of –COH and –CH groups in cellulose (Balakrishnan et al. 2017). For the control film, this region was related to amide III vibrations, likely due to residual proteins from starch extraction ($0.87\% \pm 0.04$ protein content).

A noticeable band near 970 cm^{-1} in the nanocomposites was attributed to C–O and C–H vibrations, a typical signal of cellulose presence. In contrast, in the control film, this band was mainly linked to C–O stretching modes from starch molecules (Zhao et al. 2019).

Additionally, all films showed a peak close to 930 cm^{-1} , associated with glycosidic bonds, particularly the α -1,6 linkages of amylopectin (Ribeiro Sanches et al. 2021). The absorption at 836 cm^{-1} was assigned to symmetric C–O–C stretching and deformations involving CH and CH₂ groups in the starch structure (Tibolla et al. 2020).

Lastly, a band around 745 cm^{-1} was detected in all samples, suggesting the presence of aromatic compounds (Pelissari et al. 2013), which may originate from phenolic substances naturally present in wolf fruit, the raw material used for starch extraction.

Thermal analysis

DSC, TG, and DTG were conducted to assess the thermal properties of the films. DSC curves for the control film and the nanocomposites conditioned at 58% relative humidity are shown in Fig. 6. FC melts at $T_{\text{onset}} 152.2^\circ\text{C}$ ($\Delta H_m 22.83\text{ J g}^{-1}$) an endothermic phenomenon observed in the DSC curve (short dashed black circle) without mass loss, as observed in the TG curve (Fig. 7 blue). The evaluation of the thermal behavior of the films allowed inferences characteristics such as moisture presence (shaded region of the DSC curves, Fig. 6), stability (confirmed in the TG curves, Fig. 7), and therefore critical process temperatures, as well as the influence of the acid concentration used for CNF extraction. The temperature variation (ΔT) and enthalpy (ΔH) in the endothermic phenomenon after dehydration of samples F0.1 and F1, from the start of the analysis up to $\sim 100^\circ\text{C}$ (red circle short dashed in Fig. 6), indicate that the increase in the concentration of the acid used in the

extraction causes disorder of the cellulose fibers and total rupture, a fact observed at the highest concentration used, 10%. For this film (F10), there is no heat fluctuation in this temperature range, the wide endotherm observed refers to dehydration.

TG (thick lines)/DTG (thin lines) curves for film samples are presented in Fig. 7. All films exhibit moisture content around 15%, confirmed by the mass loss between 30 and 100 °C (shaded region of Fig. 7), as previously described phenomenon (Daudt et al. 2017). Films exhibit thermal stability above 230 °C a significant mass reduction up to 380 °C due to the decomposition of starch and glycerol, driven by the removal of hydroxy groups and the depolymerization of starch carbon chains (Travalini et al. 2019). This stage marks the highest rate of thermal degradation, reflected in the drastic weight loss of the films.

The derivatives of the TG curves (thin lines in Fig. 7) indicate that the thermal decomposition of the films occurs through a complex mechanism with overlapping steps.

These materials typically exhibit three main stages of thermal behavior: (1) evaporation of absorbed environmental moisture and the water available within the material beginning at 100 °C; (2) evaporation of the glycerol-rich phase at 290 °C; and (3) maximum thermal degradation of the starch-rich phase at 330 °C (Gutiérrez et al. 2015, 2016; Gutiérrez and Alvarez 2018).

Upon heating, the initial mass of the films decreased due to volatile loss: FC lost 16% of its initial mass, while F0.1, F1, and F10 lost 13%, 15%, and 16%, respectively. Notably, moisture evaporation occurred earlier in the control film compared to the nanocomposites (Fig. 7), attributable to the higher moisture content in FC (Table 3), resulting in a greater mass loss.

Thermal decomposition began at 238 °C for film F0.1, at 248 °C for films F1 and F10, and at 257 °C for the control film. All films exhibited similar degradation behavior at the onset. Complete degradation was observed above 400 °C for both the nanocomposites and the control film. The TG/DTG curves indicate that the maximum degradation temperature, between 280 °C and 380 °C, was slightly higher for the nanocomposites compared to the control film. The higher I_c of the CNFs, relative to the wolf fruit bran, further contributes to the enhanced thermal stability of the nanocomposites (Yang et al.

2020), and similar behavior has been reported by other researchers (Ghanbari et al. 2018). Therefore, the nanocomposites proved efficient for processing at temperatures below 230 °C.

Conclusion

The results indicate that CNFs are crucial in starch-based nanocomposite films and offer significant potential for food packaging applications aimed at enhancing quality and safety. The nanocomposites demonstrated substantial improvements in optical, mechanical, and thermal properties compared to the control starch film. This makes them promising candidates for packaging food products that require protection against lipid oxidation, weight gain, and the preservation of texture and flavor during storage. The enhanced performance of the nanocomposites can be attributed to the chemical similarities between starch and cellulose, as well as the nanofibrillar dimensions and high aspect ratio of the fibers. However, the various CNF samples (CNF 0.1, CNF 1, and CNF 10) incorporated into the starch matrix showed no statistically significant differences in elongation at break and Young's modulus, while tensile strength was affected, with F0.1 exhibiting significantly higher values.

Thus, sample CNF 0.1 emerges as a promising reinforcing agent for biopolymer-based films, as its corresponding nanocomposite (F0.1) achieved superior mechanical properties (tensile strength=7.45 MPa and Young's modulus=471.57 MPa). Moreover, the synthesis of this nanoparticle occurs under the mildest chemical treatment conditions, rendering it economically viable in terms of energy and environmentally beneficial. The minor differences observed in the nanocomposite properties relative to the chemical treatment do not justify employing more aggressive processes. This study presents a potential application for a material derived from a byproduct of wolf fruit processing as an innovative product for future use in the food industry. Future studies will include TEM and/or AFM analyses to further resolve nanofiber individualization and provide higher-resolution structural characterization, complementing the findings reported here.

Author contributions AL Souza, LMC Teixeira, SO Silva and MB de Freitas-Marques conducted the main investigation, data curation and writing. G Carneiro conducted the review

and editing of the material. FM Pelissari conducted conceptualization, review, editing, and project administration. All authors contributed to the review paper. All authors approve of and agree to take responsibility for the final version of this material.

Funding The Article Processing Charge (APC) for the publication of this research was funded by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) (ROR identifier: 00x0ma614). The authors wish to acknowledge the financial support provided by Fundação de Amparo à Pesquisa do Estado de Minas Gerais – Brazil (FAPEMIG – n° APQ-00768-16) and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brazil (CAPES – n° 001). Furthermore, we would like to thank the support from LMMA sponsored by FAPEMIG APQ-00370-22—“EDITAL 002/2022—PROGRAMA DE APOIO A INSTALAÇÕES MULTIUSUÁRIOS” and FAPEMIG APQ-03088-2.

Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

Consent for publication We consent that the publisher has the author's permission to publish research findings. We confirm that the manuscript has not been published previously, is not under consideration for publication elsewhere, and is not being simultaneously submitted elsewhere. We confirm that the manuscript has been read and approved by all named authors and that there are no other persons who satisfied the criteria for authorship but are not listed. We further confirm that the order of authors listed in the manuscript has been approved by all of us.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

- Abdul Khalil HPS, Bhat AH, Ireana Yusra AF (2012) Green composites from sustainable cellulose nanofibrils: a review. *Carbohydr Polym* 87:963–979. <https://doi.org/10.1016/j.carbpol.2011.08.078>
- ASTM International (2007) ASTM D644-99. In: standard test method for moisture content of paper and paperboard by oven drying
- ASTM International (2012) ASTM D882-12. In: standard test method for tensile properties of thin plastic sheeting
- Azeredo HMC, Mattoso LHC, Wood D et al (2009) Nanocomposite edible films from mango puree reinforced with cellulose nanofibers. *J Food Sci* 74(5):N31–N35. <https://doi.org/10.1111/j.1750-3841.2009.01186.x>
- Balakrishnan P, Sreekala MS, Kunaver M et al (2017) Morphology, transport characteristics and viscoelastic polymer chain confinement in nanocomposites based on thermoplastic potato starch and cellulose nanofibers from pineapple leaf. *Carbohydr Polym* 169:176–188. <https://doi.org/10.1016/j.carbpol.2017.04.017>
- Balasubramaniam SL, Patel AS, Nayak B (2020) Surface modification of cellulose nanofiber film with fatty acids for developing renewable hydrophobic food packaging. *Food Packag Shelf Life* 26:100587. <https://doi.org/10.1016/j.fpsl.2020.100587>
- Bligh EG, Dyer WJ (1959) A rapid method of total lipid extraction and purification. *Can J Biochem Physiol* 37:911–917. <https://doi.org/10.1139/o59-099>
- Cao X, Chen Y, Chang PR et al (2008) Green composites reinforced with hemp nanocrystals in plasticized starch. *J Appl Polym Sci* 109:3804–3810. <https://doi.org/10.1002/app.28418>
- Cherian BM, Leão AL, de Souza SF et al (2011) Cellulose nanocomposites with nanofibres isolated from pineapple leaf fibers for medical applications. *Carbohydr Polym* 86:1790–1798. <https://doi.org/10.1016/j.carbpol.2011.07.009>
- Clerici MTPS, Kallmann C, Gaspi FOG et al (2011) Physical, chemical and technological characteristics of *Solanum lycocarpum* A. St.-HILL (Solanaceae) fruit flour and starch. *Food Res Int* 44:2143–2150. <https://doi.org/10.1016/j.foodres.2011.01.060>
- Daudt RM, Sinrod AJG, Avena-Bustillos RJ et al (2017) Development of edible films based on Brazilian pine seed (*Araucaria angustifolia*) flour reinforced with husk powder. *Food Hydrocoll* 71:60–67. <https://doi.org/10.1016/j.foodhyd.2017.04.033>
- Galus S, Kadzińska J (2015) Food applications of emulsion-based edible films and coatings. *Trends Food Sci Technol* 45:273–283. <https://doi.org/10.1016/j.tifs.2015.07.011>
- Gea S, Siregar AH, Zaidar E, HarahapM IDP, Perangin-Angin YA (2020) Isolation and characterisation of cellulose nanofibre and lignin from oil palm empty fruit bunches. *Materials* 13(10):2290. <https://doi.org/10.3390/ma13102290>
- Ghanbari A, Tabarsa T, Ashori A et al (2018) Thermoplastic starch foamed composites reinforced with cellulose nanofibers: thermal and mechanical properties. *Carbohydr Polym* 197:305–311. <https://doi.org/10.1016/j.carbpol.2018.06.017>
- Gontard N, Guilbert S, Cuq J (1992) Edible wheat gluten films: influence of the main process variables on film properties using response surface methodology. *J Food Sci* 57:190–195. <https://doi.org/10.1111/j.1365-2621.1992.tb05453.x>
- Gutiérrez TJ, Alvarez VA (2018) Bionanocomposite films developed from corn starch and natural and modified nano-clays with or without added blueberry extract. *Food Hydrocoll* 77:407–420. <https://doi.org/10.1016/j.foodhyd.2017.10.017>

- Gutiérrez TJ, Morales NJ, Pérez E et al (2015) Physico-chemical properties of edible films derived from native and phosphorylated cush-cush yam and cassava starches. *Food Packag Shelf Life* 3:1–8. <https://doi.org/10.1016/j.fpsl.2014.09.002>
- Gutiérrez TJ, Guzmán R, Medina Jaramillo C, Famá L (2016) Effect of beet flour on films made from biological macromolecules: native and modified plantain flour. *Int J Biol Macromol* 82:395–403. <https://doi.org/10.1016/j.ijbiomac.2015.10.020>
- Harahap M, Purwandari V, Zebua DA, Daulay N, Situmorang SA, Perangin Angin YA, Ong AJ, Siow J, Goei R, Tok ALY, Gea S (2025) Extraction and characterization of cellulose nanofibers from rice husk: potential for electrospun PVA/CNF mat composites. *Kuwait J Sci* 52(3):100403. <https://doi.org/10.1016/j.kjs.2025.100403>
- Hor S, Gastl M (2025) Starch properties of field-grown malting barley under variable weather: Interpreting results in light of controlled-environment studies. *J Cereal Sci* 125:104261. <https://doi.org/10.1016/j.jcs.2025.104261>
- HUNTERLAB (1996) CIE L*a*b* color scale. In: Applications note
- Kassab Z, Abdellaoui Y, Salim MH, El Achaby M (2020) Cellulosic materials from pea (*Pisum Sativum*) and broad beans (*Vicia Faba*) pods agro-industrial residues. *Mater Lett* 280:128539. <https://doi.org/10.1016/j.matlet.2020.128539>
- Kaushik A, Singh M (2011) Isolation and characterization of cellulose nanofibrils from wheat straw using steam explosion coupled with high shear homogenization. *Carbohydr Res* 346:76–85. <https://doi.org/10.1016/j.carres.2010.10.020>
- Kaushik A, Singh M, Verma G (2010) Green nanocomposites based on thermoplastic starch and steam exploded cellulose nanofibrils from wheat straw. *Carbohydr Polym* 82:337–345. <https://doi.org/10.1016/j.carbpol.2010.04.063>
- Kim J-K, Choi B, Jin J (2020) Transparent, water-stable, cellulose nanofiber-based packaging film with a low oxygen permeability. *Carbohydr Polym* 249:116823. <https://doi.org/10.1016/j.carbpol.2020.116823>
- LANE, J. H. (1923) Determination of reducing sugar by means of Fehling's solution with methylene blue as internal indicator. *J Soc Chem Ind* 17:32–37
- Latimer GW (ed) (2023) Official methods of analysis of AOAC international. Oxford University Press, New York
- Li M, Tian X, Jin R, Li D (2018) Preparation and characterization of nanocomposite films containing starch and cellulose nanofibers. *Ind Crops Prod* 123:654–660. <https://doi.org/10.1016/j.indcrop.2018.07.043>
- Li X, Yan J, Gao J, Xing F, Gu Q, Zhao C, Liu J (2025) Structure, physicochemical properties and in vitro digestibility of starch in corn flour with different milling processes. *Food Chem X* 30:102901. <https://doi.org/10.1016/j.fochx.2025.102901>
- Louzada LB, Lopes AT, Molina G, Pelissari FM (2021) Effect of casting process conditions on mechanical properties and water solubility of films made from wolf fruit and its optimization. *J Polym Environ* 29:2435–2446. <https://doi.org/10.1007/s10924-021-02047-z>
- Ma X, Cheng Y, Qin X et al (2017) Hydrophilic modification of cellulose nanocrystals improves the physicochemical properties of cassava starch-based nanocomposite films. *LWT* 86:318–326. <https://doi.org/10.1016/j.lwt.2017.08.012>
- Martinez-Munoz PE, Gutierrez-Cortez E, Hernandez-Urbola MI, Lopez-Leon A, Barron-Garcia OY, Rodriguez-Garcia ME (2025) Physicochemical characterization of isolated nanocrystals in starch and isolated rice starch: a new perspective. *Food Chem* 495:146590. <https://doi.org/10.1016/j.foodchem.2025.146590>
- Núñez-Flores R, Giménez B, Fernández-Martín F et al (2012) Role of lignosulphonate in properties of fish gelatin films. *Food Hydrocoll* 27:60–71. <https://doi.org/10.1016/j.foodhyd.2011.08.015>
- Ortega F, Arce VB, Garcia MA (2021) Nanocomposite starch-based films containing silver nanoparticles synthesized with lemon juice as reducing and stabilizing agent. *Carbohydr Polym* 252:117208. <https://doi.org/10.1016/j.carbpol.2020.117208>
- Pascoal AM, Di-Medeiros MCB, Batista KA et al (2013) Extraction and chemical characterization of starch from *S. lycocarpum* fruits. *Carbohydr Polym* 98:1304–1310. <https://doi.org/10.1016/j.carbpol.2013.08.009>
- Patel P, Agarwal P, Kanawaria S et al (2015) Plant-based synthesis of silver nanoparticles and their characterization. *Nanotechnology and plant sciences*. Springer International Publishing, Cham, pp 271–288
- Pelissari FM, Andrade-Mahecha MM, Sobral PJdoA, Menegalli FC (2013) Optimization of process conditions for the production of films based on the flour from plantain bananas (*Musa paradisiaca*). *LWT Food Sci Technol* 52:1–11. <https://doi.org/10.1016/j.lwt.2013.01.011>
- Pelissari FM, Andrade-Mahecha MM, Sobral PJdoA, Menegalli FC (2017) Nanocomposites based on banana starch reinforced with cellulose nanofibers isolated from banana peels. *J Colloid Interface Sci* 505:154–167. <https://doi.org/10.1016/j.jcis.2017.05.106>
- Prachayawarakorn J, Chaiwatyothin S, Mueangta S, Hanchana A (2013) Effect of jute and kapok fibers on properties of thermoplastic cassava starch composites. *Mater des* 47:309–315. <https://doi.org/10.1016/j.matdes.2012.12.012>
- Qi M, Jiang L, Song J, Li L, Xu M, Li Y, Ma C, Chen S, Li H (2024) Enhancing cassava beer quality: extrusion-induced modification of cassava starch structure boosts fermentable sugar content in wort. *Int J Biol Macromol* 278:134895. <https://doi.org/10.1016/j.ijbiomac.2024.134895>
- Ribeiro Sanches MA, Camelo-Silva C, Tussolini L et al (2021) Development, characterization and optimization of biopolymers films based on starch and flour from jabuticaba (*Myrciaria cauliflora*) peel. *Food Chem* 343:128430. <https://doi.org/10.1016/j.foodchem.2020.128430>
- Robles-García MÁ, Del-Toro-Sánchez CL, Márquez-Ríos E et al (2018) Nanofibers of cellulose bagasse from Agave tequilana Weber var. azul by electrospinning: preparation and characterization. *Carbohydr Polym* 192:69–74. <https://doi.org/10.1016/j.carbpol.2018.03.058>
- Siqueira G, Bras J, Dufresne A (2010) Cellulosic bionanocomposites: a review of preparation, properties and

- applications. *Polymers* 2:728–765. <https://doi.org/10.3390/polym2040728>
- Soni R, Asoh T-A, Hsu Y-I et al (2020) Effect of starch retrogradation on wet strength and durability of cellulose nanofiber reinforced starch film. *Polym Degrad Stab* 177:109165. <https://doi.org/10.1016/j.polymdegradstab.2020.109165>
- Soofi M, Alizadeh A, Hamishehkar H et al (2021) Preparation of nanobiocomposite film based on lemon waste containing cellulose nanofiber and savory essential oil: a new biodegradable active packaging system. *Int J Biol Macromol* 169:352–361. <https://doi.org/10.1016/j.ijbiomac.2020.12.114>
- Souza AL, Teixeira LMC, Freitas-Marques MB et al (2025) Influence of acid hydrolysis on properties of cellulose nanofibers from wolf fruit peels (*Solanum lycocarpum* A. St.-Hill). *Biomass Convers Biorefin* 15:18963–18975. <https://doi.org/10.1007/s13399-024-06402-7>
- Sutrisno E, Tanpichai S, Phiriyawirut M, Chuangchote S (2020) Investigation of cellulose nanofibers (CNFs) from *Macaranga hypoleuca* pulp in chemical treatment phase. *Mater Today Proc* 23:674–680. <https://doi.org/10.1016/j.matpr.2019.12.257>
- Tibolla H, Czaikoski A, Pelissari FM et al (2020) Starch-based nanocomposites with cellulose nanofibers obtained from chemical and mechanical treatments. *Int J Biol Macromol* 161:132–146. <https://doi.org/10.1016/j.ijbiomac.2020.05.194>
- Travalini AP, Lamsal B, Magalhães WLE, Demiate IM (2019a) Cassava starch films reinforced with lignocellulose nanofibers from cassava bagasse. *Int J Biol Macromol* 139:1151–1161. <https://doi.org/10.1016/j.ijbiomac.2019.08.115>
- Wang W, Zhang Y, Ye R, Ni Y (2015) Physical crosslinkings of edible collagen casing. *Int J Biol Macromol* 81:920–925. <https://doi.org/10.1016/j.ijbiomac.2015.09.032>
- Yang M, Zhang X, Guan S et al (2020) Preparation of lignin containing cellulose nanofibers and its application in PVA nanocomposite films. *Int J Biol Macromol* 158:1259–1267. <https://doi.org/10.1016/j.ijbiomac.2020.05.044>
- Zhang Z, Liu Q, Zhao R, Li J, Hu H, Richel A (2025) Modulation of potato starch gelatinization, rheological properties, and mechanical properties: The role of potato soluble dietary fiber with different molecular weights. *Int J Biol Macromol* 323:147211. <https://doi.org/10.1016/j.ijbiomac.2025.147211>
- Zhao Y, Huerta RR, Saldaña MDA (2019) Use of subcritical water technology to develop cassava starch/chitosan/gallic acid bioactive films reinforced with cellulose nanofibers from canola straw. *J Supercrit Fluids* 148:55–65. <https://doi.org/10.1016/j.supflu.2019.02.022>

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.