

Article

Valorization of Green Banana Peel Waste into Starch–Glycerol Biodegradable Films: Optimization and Physicochemical Characterization [†]

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Abstract

The growing demand for sustainable alternatives to conventional plastic packaging has promoted the development of biodegradable materials derived from agro-industrial residues. In this study, starch extracted from green banana peel waste was used to produce biodegradable starch–glycerol films for food-packaging applications. Nine film formulations (PPM1–WP6) were prepared using starch obtained through different extraction approaches, including parenchyma-only and whole-peel processing, with and without sodium metabisulfite treatment. The extracted starches were characterized through proximate composition analysis, Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and differential scanning calorimetry (DSC). The resulting films were evaluated for structural, thermal, mechanical, and barrier properties using FTIR, X-ray diffraction (XRD), SEM, dynamic mechanical analysis (DMA), water vapor permeability (WVP), and water solubility (WS). The films exhibited characteristic starch functional groups, semicrystalline structures, thermal degradation temperatures ranging from 283 to 302 °C, and viscoelastic behavior associated with plasticized polymeric matrices. Increasing glycerol concentration improved film flexibility, whereas sodium metabisulfite treatment enhanced mechanical strength. Films prepared from whole-peel starch showed higher WVP and WS values than those obtained from parenchyma starch. A randomized factorial design was used to optimize film performance for maximum tensile strength and minimum water solubility. Among the evaluated formulations, PPM2 (4% starch with sodium metabisulfite treatment) was identified as the optimal formulation and was statistically validated. These findings demonstrate the potential of green banana peel waste as a sustainable source of starch for developing biodegradable films with promising applications in food packaging.

Keywords: banana peel valorization; biodegradable films; glycerol plasticization; starch extraction; sustainable food packaging; water vapor permeability



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1. Introduction

Plastics are widely used in the food-packaging industry for their versatility, durability, low production costs, and favorable physicochemical properties [1]. Conventional packaging materials are predominantly manufactured from petroleum-derived polymers such as polyethylene terephthalate (PET), low-density polyethylene (LDPE), and polystyrene (PS) [2]. Despite their widespread use, plastic waste has become a major environmental concern due to its persistence and limited degradation under natural conditions [3]. Over time, plastic debris can fragment into microplastics (MPs) and nanoplastics (NPs), which are increasingly detected in aquatic and terrestrial ecosystems, where they may negatively affect environmental quality and ecological functions [4]. Consequently, the environmental impact of plastic pollution has attracted global attention and reinforced the need to develop sustainable alternatives in accordance with the United Nations Sustainable Development Goals (SDGs) [5].

In response to this challenge, biodegradable materials obtained from renewable resources and agro-industrial residues have emerged as promising alternatives to conventional plastics [6]. Several studies have explored the use of agricultural by-products and natural polymers for the production of biodegradable packaging materials, including potato peel powder [7], gelatin–papaya composites [8], polylactic acid [9], chitosan [10], and nanocellulose–lignin nanoparticle composites [11]. Although these materials have demonstrated potential for packaging applications, many still have limitations in mechanical performance, flexibility, barrier properties, or production costs. Therefore, the search for low-cost, biodegradable materials derived from abundant agricultural residues remains an active area of research.

Among the available agro-industrial residues, banana peels are an attractive raw material for the development of biodegradable materials [12]. The banana (*Musa acuminata*) is one of the most widely cultivated fruits worldwide, produced in approximately 130 countries. India was the leading producer in 2019, with an annual production of about 30.4 million tonnes [13]. However, large-scale banana production generates substantial amounts of underutilized waste, particularly peels, which are commonly discarded despite containing valuable biopolymers [14]. One promising strategy for valorizing this residue is to extract starch to manufacture biodegradable films [15].

Starch is a naturally occurring polysaccharide mainly composed of amylose, a linear polymer of α -(1→4)-linked glucose units, and amylopectin, a highly branched polymer containing both α -(1→4) and α -(1→6) glycosidic linkages [16]. Due to its film-forming ability, biodegradability, renewability, and wide availability, starch has been extensively investigated as a raw material for sustainable packaging systems [17]. Furthermore, starch-based materials exhibit good oxygen-barrier properties and relatively low retrogradation rates, thereby improving dimensional stability in packaging applications [18]. Nevertheless, the highly hydrophilic nature of starch limits its resistance to moisture and water vapor, often resulting in reduced mechanical performance and limited applicability under humid conditions [19].

To overcome these limitations, plasticizers are commonly incorporated into starch matrices to improve flexibility and processability. Among them, glycerol is one of the most widely used due to its low molecular weight, high boiling point, and ability to form hydrogen-bond interactions with starch chains [20]. These interactions reduce intermolecular forces within the polymeric network, increase chain mobility, and improve film flexibility. Previous studies have demonstrated that glycerol incorporation can enhance the mechanical properties and biodegradability of starch-based materials [21,22]. Additionally, chemical pretreatments and modification strategies have been explored to improve the structural integrity and functional performance of starch-derived materials [23]. Although starch–glycerol films derived from

green banana peel have been reported previously, the influence of starch extraction methodology and sodium metabisulfite pretreatment on the physicochemical, thermal, mechanical, and functional properties of the resulting films remains underexplored. Furthermore, comparative studies evaluating starch from banana parenchyma and whole-peel residues under different extraction conditions remain limited. Therefore, identifying optimized formulations that simultaneously exhibit favorable mechanical properties and controlled water solubility remains a challenge for food-packaging applications.

Accordingly, this study aimed to develop and characterize biodegradable starch–glycerol films produced from green banana peel waste using different starch extraction methodologies, including sodium metabisulfite pretreatment. The extracted starches and the resulting films were characterized through physicochemical, morphological, thermal, structural, mechanical, and barrier analyses. In addition, a factorial experimental design was employed to identify the formulation with the most suitable combination of mechanical strength and water solubility for potential application as a sustainable food-packaging material.

2. Materials and Methods

2.1. Green Banana Samples

Green banana peels (*Musa acuminata*) were collected from a local market in Barranquilla, Colombia (10°57'00.4" N 74°47'40.1" W), in the Atlántico Department, and from a local market in Santiago de Cali, Colombia (3°21'45.6" N 76°31'53.9" W), in the Valle del Cauca Department. Glycerin and sodium metabisulfite were purchased from Doquímica de la Costa (Barranquilla, Colombia). Citric acid powder was purchased from Juliao Tienda, located on Paseo Bolívar (Barranquilla, Colombia), and sodium hypochlorite from Tiendas Ara (Barranquilla, Colombia). No further purification of the chemicals was performed.

2.2. Extraction of Starch from Green Banana Peels

For the peels collected from the Atlántico department, starch extraction was carried out using the methodology reported by [24], with some modifications. Additionally, a wet treatment (with and without metabisulfite) was performed, involving the physical separation of components such as proteins and fibers through particle-size reduction, milling, washing, and sedimentation in an aqueous medium. Briefly, the samples were washed with 0.02% *w/v* sodium hypochlorite and 5% *w/v* citric acid solutions. The parenchyma was separated by discarding the fibrous part of the peel, then oven-dried (42 L Oster Digital Oven with French Door Fryer, WI, USA) for 6 h at 65 °C. Once dry, the samples were treated with a 0.5% *w/v* sodium metabisulfite solution and then oven-dried at 65 °C for 6 h. Subsequently, the particle size was reduced by moistening the sample and grinding it with an immersion blender (MIX IMUSA Hand Blender, Colombia) and then allowing it to stand for 4 h in a container of distilled water. The supernatant was then removed, and the sample was filtered through a mesh screen. It was then dried again under the same conditions. The particle size was further reduced by pulverizing the sample with an electric pulverizer (BI-DTOOL, Shanghai, China). Finally, the resulting starch was sieved using Endecott sieves (London, UK) of different diameters (425, 180, and 150 µm). The resulting powder was stored in a Ziploc bag at room temperature.

Second Methodology for Starch Extraction

For the peels collected in the Valle del Cauca department, the methodology reported by [25] was applied in the laboratory, with modifications. The entire green banana peel was used, including both the fibrous and parenchyma layers. The peels were chopped to reduce their size and then blended in an Oster blender (Racine, WI, USA), and the mixture was filtered. The sedimented product was oven-dried (Oster, WI, USA) at 40 °C for 8 min

and subsequently ground (IKA 2900001 A 11, Staufen, Germany). Finally, the powdered product was stored in Ziploc bags at room temperature.

2.3. Preparation of Banana Starch/Glycerin Films

The preparation of the films followed the methodology reported by [26], with modifications. Nine different formulations were prepared by varying the amounts of starch and glycerin (Table 1). For starch extracted solely from the treated parenchyma, with and without sodium metabisulfite, a mixture of starch and glycerin at 20% *w/w* (relative to starch) was prepared in 100 mL of water; for starch extracted from the whole peel, mixtures of starch with glycerin at 15%, 20%, and 25% *w/w* (relative to starch) were prepared. The mixtures were then heated in an oven with constant stirring at 85 °C for 30 min and poured onto non-stick aluminum trays. The samples were left to dry for 12 h at room temperature and for 24 h in an oven at 60 °C. Finally, the solid slides were removed and stored in Ziploc bags at room temperature.

Table 1. Formulations used to obtain starch films from green banana peels and glycerin.

Sample	PPM1	PP1	PPM2	PP2	PPM3	PP3	WP4	WP5	WP6
Starch banana (g)	3	3	4	4	5	5	4	4	4
Glycerin (g)	0.6	0.6	0.8	0.8	1	1	0.6	0.8	1
Wt. %	20	20	20	20	20	20	15	20	25
Metabisulfite treatment	Yes	Not	Yes	Not	Yes	Not	Not	Not	Not
Peel Part Used	Parenchyma	Parenchyma	Parenchyma	Parenchyma	Parenchyma	Parenchyma	Whole peel	Whole peel	Whole peel

2.4. Analysis of Starch from Green Banana Peels

2.4.1. Proximate Starch Analysis

The proximate composition of the extracted starch was evaluated (% moisture, fat, ash, protein, and crude fiber). Moisture content was determined using AOAC method 925.10; fat content, according to AOAC method 920.39; ash content, according to AOAC method 923.03; protein percentage, according to the Kjeldahl method AOAC 981.10; and crude fiber content, according to AOAC method 962.09. Total carbohydrate content was determined indirectly using Equation (1):

$$\%CT = 100 - (\%H + \%G + \%C + \%F + \%Pc) \quad (1)$$

where %CT = percentage of total carbohydrate; %H = percentage of moisture; %G = percentage of fats; %C = percentage of ash; %F = percentage of crude fiber; %Pc = percentage of protein.

2.4.2. Characterization of Functional Groups

Fourier-transform infrared (FTIR) spectroscopy analysis was performed using a Spectrum Two FT-IR spectrometer (PerkinElmer Inc., Waltham, MA, USA) to determine functional groups. Spectra were obtained by averaging 32 scans at a resolution of 4 cm^{−1} over the range 400 to 4000 cm^{−1}, using an ATR accessory.

2.4.3. Morphological Analysis

Scanning electron microscopy (SEM) was used to analyze the surface morphology of starch. For starch with and without metabisulfite, the SU8000 Scanning Electron Microscope (Hitachi, Krefeld, Germany) was used with an accelerating voltage of 20 kV; the samples

were coated with a gold bath to improve conductivity. For whole-peel starch, the ZEISS EVO10 microscope was used with an accelerating voltage of 5 kV.

2.4.4. Thermogravimetric Analysis

Thermogravimetric analysis was used to evaluate the thermal stability of the compounds. For this purpose, a TGA 2 Star System (METTLER TOLEDO) was used in a nitrogen atmosphere at a flow rate of 50 mL/min and a temperature ramp of 10 °C/min over the range of 30 to 800 ± 2 °C.

2.4.5. Differential Scanning Calorimetry Analysis

Differential scanning calorimetry (DSC) was used to study the thermal behavior of starch during heating. It was performed using a DSC2A-00181 system (TA Instruments, New Castle, DE, USA). Briefly, 5.4 mg of the sample was placed in a metal capsule. A heating cycle from −25 °C to 250 °C was performed with a ramp rate of 10 °C/min and a nitrogen flow rate of 50 mL/min. This was followed by a 5 min isotherm, a cooling cycle to −20 °C, and a subsequent heating cycle identical to the first.

2.5. Analysis of Banana Starch/Glycerin Films

2.5.1. Characterization of Functional Groups

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2.5.4. X-Ray Diffraction Analysis

X-ray diffraction (XRD) was performed on the Bruker D8 Advance diffractometer (Bruker, MA, USA) with Cu Kα1 (1.540598 Å) and Kα2 (1.544426 Å) radiation in a 2θ range of 5° to 80°. The crystallinity index of the films was quantitatively calculated based on the ratio of the crystalline area to the total area of the diffractogram, following the procedure of [27], using Equation (2) and the Origin 2025 software (OriginLab Corporation, Northampton, MA, USA).

$$\text{Crystallinity index, IC (\%)} = \left(\frac{\sum \text{area of the crystalline peaks}}{\sum \text{area of crystalline and amorphous peaks}} \right) \times 100\% \quad (2)$$

2.5.5. Dynamic-Mechanical Analysis

Strips approximately 2.2 mm wide and 15 mm long were cut from the films for dynamic mechanical analysis. Samples were analyzed in tensile mode using a METTLER-TOLEDO

DMA/SDTA861e (Greifensee, Switzerland). The storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) of the strips were measured at a frequency of 1 Hz, in a temperature range of $-100\text{ }^{\circ}\text{C}$ to $65\text{ }^{\circ}\text{C}$, and with a heating rate of $2\text{ }^{\circ}\text{C}$ [28].

2.5.6. Mechanical Properties

A universal SHIMADZU EZ-LZ test machine (Shimadzu, Tokyo, Japan) with a 500 N load cell was used for the tensile test in accordance with ASTM D882. The samples were tested with a 100 mm gap between the jaws at a speed of 50 mm/min. At least 10 samples per formulation were used. The length and width of the tested samples were 100 mm and 20 mm, respectively.

2.5.7. Contact Angle

The surface properties of the films were determined by measuring the contact angle of a water droplet. The water contact angle (WCA) was measured at $25\text{ }^{\circ}\text{C}$ using a KSV CAM200 tensiometer (KSV Instruments, Helsinki, Finland). The contact angle is defined as the angle between the film surface and the tangent line at the point of contact of the water droplet with the surface. For each synthesized film, at least 10 measurements were taken, and the average and standard deviation were calculated.

2.5.8. Water Vapor Permeability

Water vapor permeability (WVP) was determined using the ASTM E96/E96M-24a desiccant method [29]. The diameter and thickness of the tested samples were $80 \pm 0.2\text{ mm}$ and $0.14 \pm 0.04\text{ mm}$, respectively. The samples were affixed to the mouth of a permeation cell with silica gel. The cells were stored in an airtight container at 75% RH and $25\text{ }^{\circ}\text{C}$. The slope of the permeation cell mass change over time was obtained using linear regression. The WVP (g mm/kPa h m^2) was calculated using Equation (3):

$$WVP = \left[\frac{W V T R}{P R \times H} \right] \times l \quad (3)$$

where WVTR is the water vapor transmission rate, obtained from the ratio between the water gained as a function of time (g/h) and the permeation area of the sample (m^2); P is the saturation vapor pressure of water (kPa); HR is the relative humidity in the desiccator; and l is the average thickness of the sample (m).

2.5.9. Water Solubility

Water solubility (WS) was determined as described by [30]. Briefly, $2\text{ cm} \times 2\text{ cm}$ films were dehydrated at $105\text{ }^{\circ}\text{C}$ for 24 h, and their weight (W_i) was recorded. The dried films were then immersed in 30 mL of distilled water for 24 h. Finally, the samples were filtered and dehydrated at $105\text{ }^{\circ}\text{C}$ for 24 h to obtain their final dry weight (W_f), allowing the calculation of water solubility, WS (%), using Equation (4):

$$WS = \left[\frac{W_i - W_f}{W_i} \right] \times 100\% \quad (4)$$

2.6. Experimental Design and Optimization

A completely randomized factorial experimental design was implemented with two factors, namely starch concentration (3, 4, and 5%) and the presence or absence of sodium metabisulfite, yielding six treatments corresponding to formulations PPM1 through PP3, as shown in Table 1. The optimization process was performed on these six treatments, based on variables that had significant effects on mechanical responses (maximized) and solubility properties (minimized). The desirability function (D : global; d : individual) was used to

convert the functions to a 0–1 scale, combine them using the geometric mean, and optimize the overall metrics. The experimental design and optimization were performed using Minitab software (2019). Subsequently, the optimal formulation without metabisulfite was used to evaluate the effects of glycerol concentrations (15, 20, and 25%) on the properties of starch films extracted from whole-fruit peels. These treatments correspond to formulations WP4–WP6 described in Table 1.

Statistical Analysis

The Shapiro–Wilk normality test and Levene’s test for homogeneity of variances were performed to analyze the data distribution. Once the normality of the data was verified, an analysis of variance (ANOVA) was performed using Minitab software (2019) to determine the effects of the factors and their interactions on the response variables (mechanical properties, WVP, WS) in the factorial experimental design. A one-to-one ANOVA and a post hoc ANOVA with Fisher’s exact test were performed for the treatments. A significance level of 5% was used in all analyses.

3. Results and Discussion

3.1. Proximate Analysis of Starch from Starch Husks

Bananas, in their green ripening stage, are a good source of starch. A wet milling process is ideal for starch isolation due to its low impurity content, and various extraction methods can be used, including alkaline and non-alkaline methods [31]. Starch is primarily composed of carbohydrates; however, its composition also includes non-starch components such as lipids, proteins, and ash [32].

The proximate analysis of starch using different methodologies is presented in Table 2. The variation in chemical composition between the two starch sources (Valle del Cauca and Atlántico) is because tropical countries like Colombia have diverse banana varieties, resulting from different microclimates, soil types, and harvest times, which can lead to varying degrees of maturity [33]. Furthermore, when working with waste, multiple varieties of green bananas may be present. The results showed that the starch extracted solely from the parenchyma (with and without metabisulfite) had higher moisture contents (12.41% and 12.19%, respectively). The change in moisture content can be attributed to differences in extraction methodologies, temperature, drying time, and storage conditions [34]. However, the moisture content of the three extracted green banana starches allows for their storage at room temperature and their stability against fungal and microbial infections [35], given that the maximum recommended moisture content for safe storage is 13% (NTC 926; NTC 6066).

Table 2. Proximate analysis of starch extracted from green banana peels.

Parameters	Starch Without Metabisulfite (Parenchyma Only)	Starch with Metabisulfite (Parenchyma Only)	Starch (Whole Peel)
Humidity (%)	12.19	12.41	8.01
Protein (%)	3.50	3.14	5.76
Fats (%)	1.60	0.90	9.96
Ash (%)	3.96	1.10	7.11
Crude Fiber (%)	1.59	1.94	2.06
Total Carbohydrates (%)	77.16	80.51	67.1

In starch extracted from the whole green banana peel, the protein (5.76%), ash (7.11%), fat (9.96%), and crude fiber (2.06%) contents were higher than in starch extracted from the parenchyma. The high protein content in the starch could be associated with the presence of amino acids such as leucine, valine, phenylalanine, and threonine in the banana peel [36].

The high ash content, on the other hand, is due to the concentration of sap present in the green banana peel, which promotes better adhesion between the minerals and the starch granules [37], as well as the concentration of potassium, magnesium, sodium, and calcium [38]. An increase in mineral content was observed with the addition of peel to the starch flour; a similar result was obtained [39] with 7.05% ash, indicating a high level of soil contamination. Furthermore, the high fat content is due to the presence of a greater amount of lipids and fat-soluble compounds, including polyunsaturated fatty acids (primarily linoleic and linolenic acids) [40]. Ref. [41] studied a fat content of $0.66 \pm 0.03\%$ for banana flour (*Musa paradisiaca* L.) in Mexico, and [42] investigated a fat content between $0.60 \pm 0.02\%$ and $0.54 \pm 0.03\%$ for pulp flour (*Musa* spp.) in four cultivars of Kapas, Kepok, Ambon, and Nangka in Indonesia. The results depend on the fruit variety, the part of the fruit used, the geographical area, and the process used to obtain starch flours. The fiber content, composed of non-starch polysaccharides such as hemicellulose and pectin present in starch [43], increased to a value between 1.59% and 1.945% and up to 2.06% with the preservation of the fruit peel. This is corroborated by a study conducted by [44] on flour from unpeeled green bananas of different varieties, obtaining fiber content values of $6.29 \pm 0.08\%$, $7.25 \pm 0.08\%$, and $8.44 \pm 0.31\%$, indicating that the presence of the peel contributes to higher levels of ash and phytosterols. On the other hand, total carbohydrate values decreased when the peel was incorporated and increased when the fruit parenchyma was used for extraction. [45] found a carbohydrate percentage of $82.53 \pm 0.05\%$, consistent with that obtained in this study (80.51%). They also reported that the starch content, including amylose and amylopectin, increased after washing with sodium metabisulfite, demonstrating the inhibitory capacity of this compound on enzymatic activity and preserving the starch by preventing its fractionation. The starch treated with metabisulfite, obtained from the parenchyma of green bananas, showed low levels of each chemical component (proteins, fats, ash, and crude fiber) and high total carbohydrate levels, unlike the other two extracted starch types, indicating purer, higher-quality starch. Extraction by immersion in a sodium metabisulfite solution has been reported to improve starch yield and quality [46,47].

3.2. FT-IR Spectroscopic Analysis of Green Banana Starch

Figure 1 shows the FT-IR characterization of green banana starch, extracted from the peel parenchyma with and without metabisulfite, and of starch from the whole peel. The FTIR spectrum showed broad bands at 3309 cm^{-1} , 3323 cm^{-1} , and 3329 cm^{-1} , attributed to the stretching of the hydroxyl (OH) groups in the carbohydrate structure. However, differences in their intensity may be due to the extraction method used; starch treated with metabisulfite (PSM) increases water absorption, which contributes to the formation of hydrogen bonds, as corroborated by the moisture content shown in Table 2. The bands at 2922 cm^{-1} and 2926 cm^{-1} correspond to CH_2 stretching and are characteristic of the starch structure. The absorption bands observed at 1612 cm^{-1} , 1637 cm^{-1} , and 1639 cm^{-1} are related to the stretching of the $\text{C}=\text{O}$ bonds present in the amide group of proteins [48]. For WPS starch, this results in a more intense band due to the nature of the whole-peel residue, which shows a higher protein percentage in Table 2. The absorption band at 1151 cm^{-1} is related to the stretching of the CO bond of the starch COH group [49]. On the other hand, the absorption bands around 991 cm^{-1} and 995 cm^{-1} are associated with the COC glycosidic bonds [50]; likewise, the bands at 921 cm^{-1} and 925 cm^{-1} are attributed to the saccharide structure [51]. Due to the extraction method and the portion of the husk used (PMS), these bands are more intense due to the high total carbohydrate content.

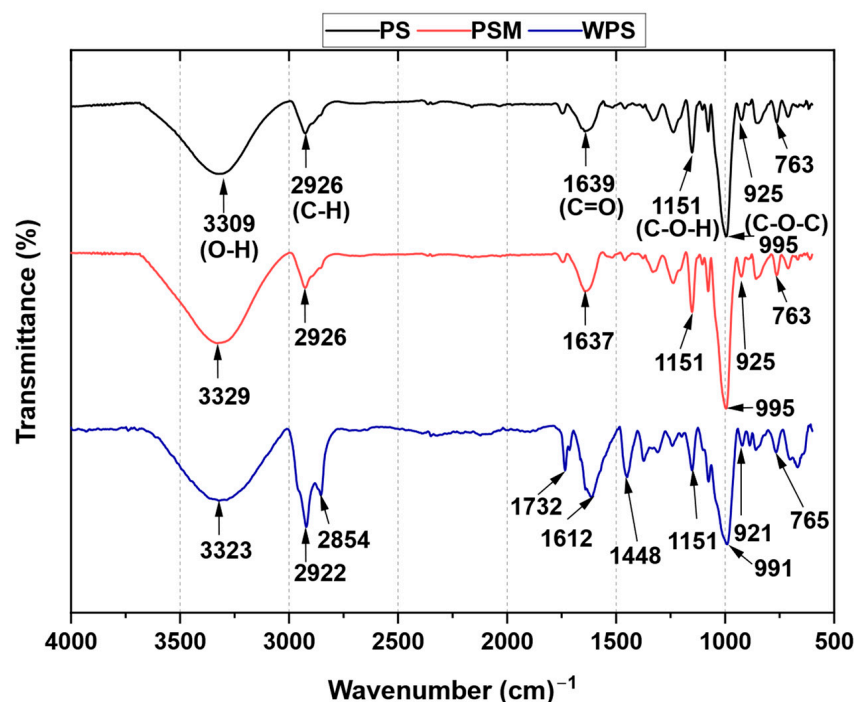


Figure 1. FT-IR spectrum of green banana parenchyma starch without metabisulfite treatment (PS), green banana parenchyma starch with metabisulfite treatment (PSM), and green banana whole peel starch (WPS).

Finally, the absorption bands at 763 cm^{-1} and 765 cm^{-1} are attributed to aromatic groups associated with the phenolic components of the samples [52]. Therefore, all bands were retained between the starch treated without metabisulfite and that treated with metabisulfite in this analysis. However, for starch extracted with its husk intact, a distinct band is observed at 2854 cm^{-1} . A study by [53] identified bands at 2915 cm^{-1} and 2845 cm^{-1} , attributed to symmetric and asymmetric CH stretching in cellulose; likewise, the band at 1448 cm^{-1} is associated with asymmetric CH stretching in methyl esters in pectins. We also observe a band with greater intensity at 1732 cm^{-1} , which may be associated with the C=O stretching of a minor carbonyl group, such as those present in fatty acids [54]. These results are consistent with the proximate analysis, which revealed a higher presence of crude fiber and fats in the starch of the whole peel.

3.3. Thermogravimetric Analysis of Green Banana Starch

The thermogravimetric (TG) analysis of banana starch with and without metabisulfite, and of starch with the whole peel, is shown in Figure 2. The thermogram showed mass losses around 100 °C , attributable to the evaporation of water adsorbed by the starches. The next decomposition phase of starch extracted from the parenchyma (with and without treatment) occurs between 200 and 350 °C , which is associated with the breaking of bonds in the glucose ring of the starch's amylose–amylopectin polymeric ratio [55]. For starch extracted from the whole peel, this stage occurs between approximately 180 °C and 480 °C , attributable to the degradation of the amylose–amylopectin ratio in the starch, followed by the breakdown of the glycosidic bonds in the hemicellulose, cellulose, and lignin present in the whole peel samples [56,57].

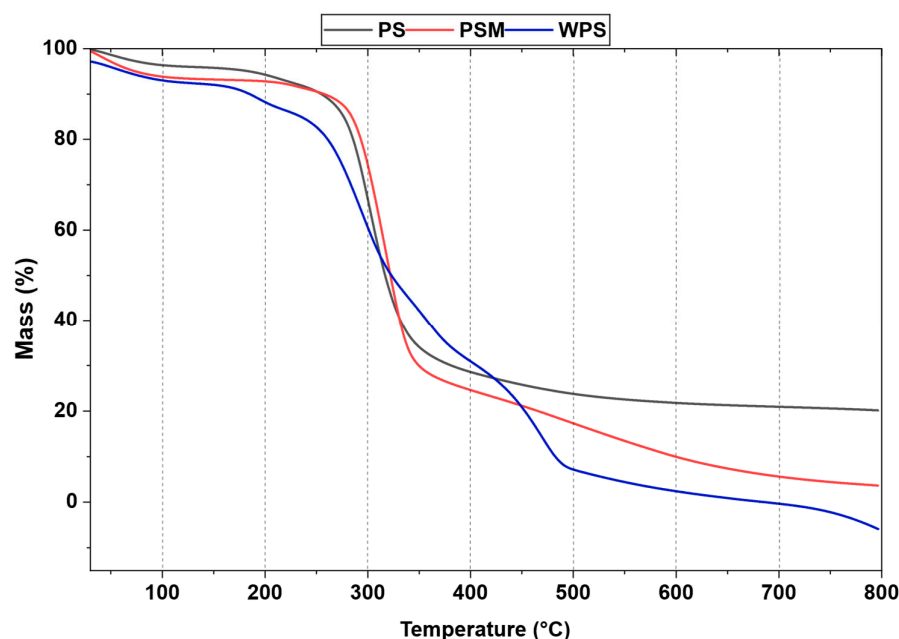


Figure 2. TGA thermograms of green banana parenchyma starch without metabisulfite treatment (PS), green banana parenchyma starch with metabisulfite treatment (PSM), and green banana whole peel starch (WPS).

On the other hand, Figure 3 shows the derived weight-loss (DTG) curve, which indicates the thermal degradation temperatures (T_{dmax}) for parenchyma and whole-peel starches. The peaks at 48 °C correspond to the evaporation of water adsorbed by the starch in the first degradation zone observed in the TGA [58]. The peaks corresponding to the depolymerization of the amylose–amylopectin polymer chains shown in the second TGA degradation zone for parenchyma starches are at 302 °C and 318 °C, showing that banana parenchyma starch can be subjected to various storage conditions without notable changes in its stability at temperatures below 226 °C [59].

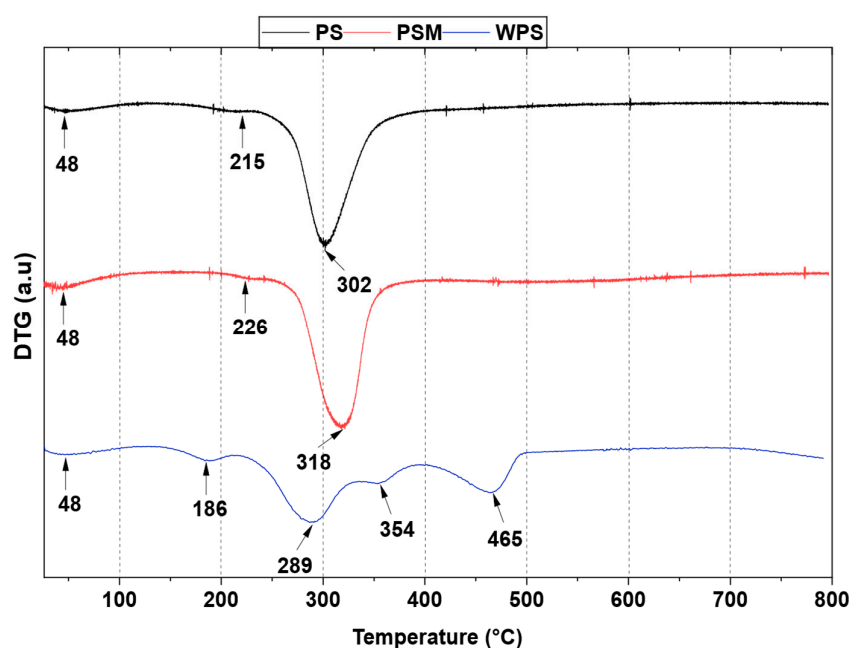


Figure 3. DTG thermograms of green banana parenchyma starch without metabisulfite treatment (PS), green banana parenchyma starch with metabisulfite treatment (PSM), and green banana whole peel starch (WPS).

In contrast, the behavior observed for the whole peel starch showed a peak at 186 °C, attributable to the degradation of hemicellulose compounds that are more thermally unstable and that influence the thermal shift of the peak to 286 °C, corresponding to the depolymerization of amylose–amylopectin, since hemicellulose degradation also occurs at temperatures ranging approximately from 210 °C to 248 °C [60]. In the study of [61], the authors demonstrated that the decomposition of lignocellulosic components primarily occurs at temperatures between 340 °C and 380 °C. Similarly, [62] showed that the decomposition of cellulose in raw biomass occurred between 329.8 °C and 353.3 °C. This behavior is associated with the peak observed at 354 °C, corresponding to the degradation of cellulose present in the whole peel, and correlates with the 2854 cm^{−1} band in the FT-IR spectrum assigned to the C-H vibrations of this polysaccharide. Finally, the peak at 465 °C may be related to the degradation of aromatic structures, such as residual lignin, which can decompose over a wide temperature range and whose presence is correlated with the 3763 cm^{−1} and 765 cm^{−1} bands in the FT-IR spectrum, which are attributed to aromatic groups associated with phenolic components.

3.4. Differential Scanning Calorimetry from Green Banana Starch

Differential scanning calorimetry (DSC) was used to study the thermal transitions of samples upon heating or cooling. In this regard, the thermogram of banana starch (Figure 4) showed endothermic peaks at 91 °C, 85 °C, and 71 °C, with no treatment, with metabisulfite treatment, and with the whole banana peel, respectively. This peak is related to the loss of water adsorbed by the starch, which corresponds to the gelatinization temperature (T_p), where the bonds between the starch polymer structures (amylose and amylopectin) begin to break [63]. These findings were similar to those obtained in [64], which used native green banana flour. The endothermic transition of the starch samples is influenced by chain length, hydrogen bonding in the crystalline region, granule morphology, and the various orientations of the starch molecules. These properties influence their swelling capacity, including the amylose–amylopectin and amylopectin–lipid complexes [65,66].

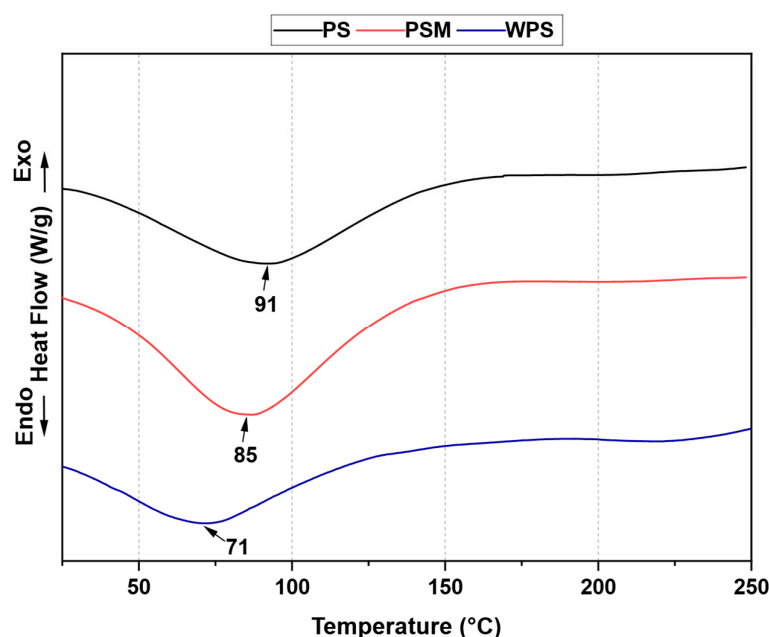


Figure 4. Differential scanning calorimetry of green banana parenchyma starch without metabisulfite treatment (PS), green banana parenchyma starch with metabisulfite treatment (PSM), and green banana whole peel starch (WPS).

Pretreatment with sodium metabisulfite may have altered the granular structure of the starch, thereby decreasing T_p , as larger starch granules can absorb more water and swell more readily [67]. In fact, a study on epichlorohydrin-crosslinked resistant starch reported this effect [68]. Water and heat promote the breaking of intermolecular bonds in the starch polymer, facilitating the action of water molecules as plasticizers. T_p can increase at lower moisture content, as indicated by proximate analysis of starch without metabisulfite compared to starch with metabisulfite [69]. For whole-peel starch, we observed a shift in T_p at low temperatures. This is due to the presence of non-starch compounds from the fibrous part of the shell, which result in more thermally unstable starch granules, thereby reducing the energy absorbed by the starch [70].

3.5. Scanning Electron Microscopy (SEM) of Green Banana Starch

Scanning electron microscopy (SEM) analysis allows observation of the compound's morphology, structural integrity, shape, and size. Morphological analysis of starch extracted from the parenchyma of green banana peel, with and without metabisulfite pretreatment, reveals granules of variable shapes and sizes. The micrographs presented in Figure 5 reveal elongated, flattened, and irregular oval structures, with sizes ranging from 10.7 μm to 33.7 μm for starch with metabisulfite (Figure 5A,D), 11.5 μm to 33.4 μm for starch without metabisulfite (Figure 5B,E), and 13.8 μm to 34.2 μm for starch extracted from the whole peel (Figure 5C,F) [71]. Particle sizes were close to those reported previously by [72] with equatorial lengths between 29.3 μm and 48.53 μm and transverse lengths between 16.6 μm and 40.55 μm , indicating a predominantly ellipsoidal structure in the granules. These morphologies of starch flour extracted from banana peel are primarily related to the botanical source and the specific characteristics of the starch type, as well as the amount of amylose and amylopectin present [73]. The structural characteristics of the starch also depend on the presence of α -glucans, lipid composition, and protein content [74]. The irregular structures present in the starch granules (Figure 5D–F) can be attributed to the amyloplast membranes that enclose the starch granules within the banana fruit cell [75]. The large size of the starch granules indicates slow hydrolysis by digestive enzymes, with banana starch being a type 2 starch resistant to digestion [76]. The granules obtained without metabisulfite pretreatment are less agglomerated and more easily distinguishable. In the starch sample where all the peel waste was used, granules are observed attached to fibrous and protein structures due to their high content, as reported in the respective proximate analysis [77].

3.6. FT-IR Spectroscopic Analysis of Green Banana Starch/Glycerin Films

Figure 6 shows the FT-IR characterization of the green banana starch/glycerin films (PPM1-WP6). All films showed a strong, broad band corresponding to the OH stretching. This is due to the characteristic signal of native green banana starch, corresponding to the hydroxyl stretching [78], a vibration also present in glycerin [79]. For PP2 and PP3, this band is more pronounced, with a shift to higher vibrational frequencies of 3310 cm^{-1} and 3315 Hz cm^{-1} ; this may be due to the starch/glycerin mass ratio, generating a noticeable change because the highest amounts of starch and glycerin were used for both formulations. The bands between 2927 and 2931 cm^{-1} across all samples demonstrate CH stretching, which is attributed to the CH_2 groups of starch, as well as the proportions of amylose and amylopectin in green banana flour [80]. The absorption bands between 1642 cm^{-1} correspond to the $\text{C}=\text{O}$ bond typical of amides, which are present in the residual proteins of the flours [81].

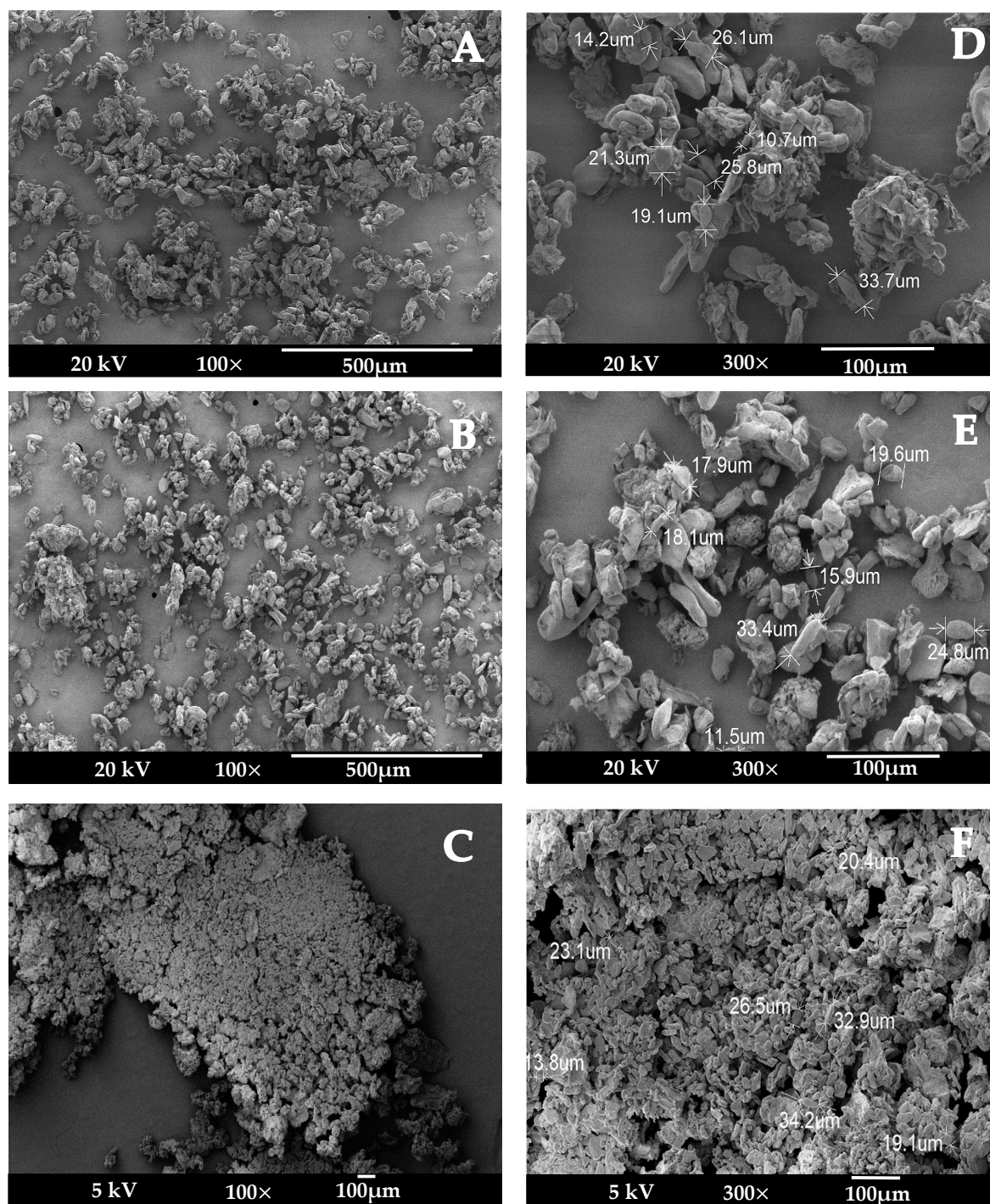


Figure 5. Morphological analysis by scanning electron microscopy (SEM) of green banana starch flour: (A) (500 μm) starch with metabisulfite; (B) (500 μm) starch without metabisulfite; (C) (100 μm) whole-peel starch; (D) (100 μm) starch with metabisulfite; (E) (100 μm) starch without metabisulfite; and (F) (100 μm) whole-peel starch.

Another characteristic band of starch is the C–O–C group vibration, observed near 999 cm^{-1} [83]. This band is present in all formulations, shifting slightly to 1003 cm^{-1} . For glycerin, absorption corresponding to C–C and C–O bond vibrations is observed in the spectral region between 800 and 150 cm^{-1} [84]. The bands at 1148 and 1153 cm^{-1} were mainly attributed to the C–O stretching of C–O–H in starch [85]. It is observed that the films synthesized with metabisulfite-treated starch (PPM1, PPM2, and PPM3) maintain the same bands and functional groups as the films synthesized with non-metabisulfite-treated

starch, as confirmed by previous studies [86]. However, in PPM1, PPM2, and PPM3, the bands are less intense. This can be attributed to the hydroxyl groups in the starch structure, which may act as nucleophiles, interacting with the electrophilic region at the sulfur center of sodium metabisulfite [87].

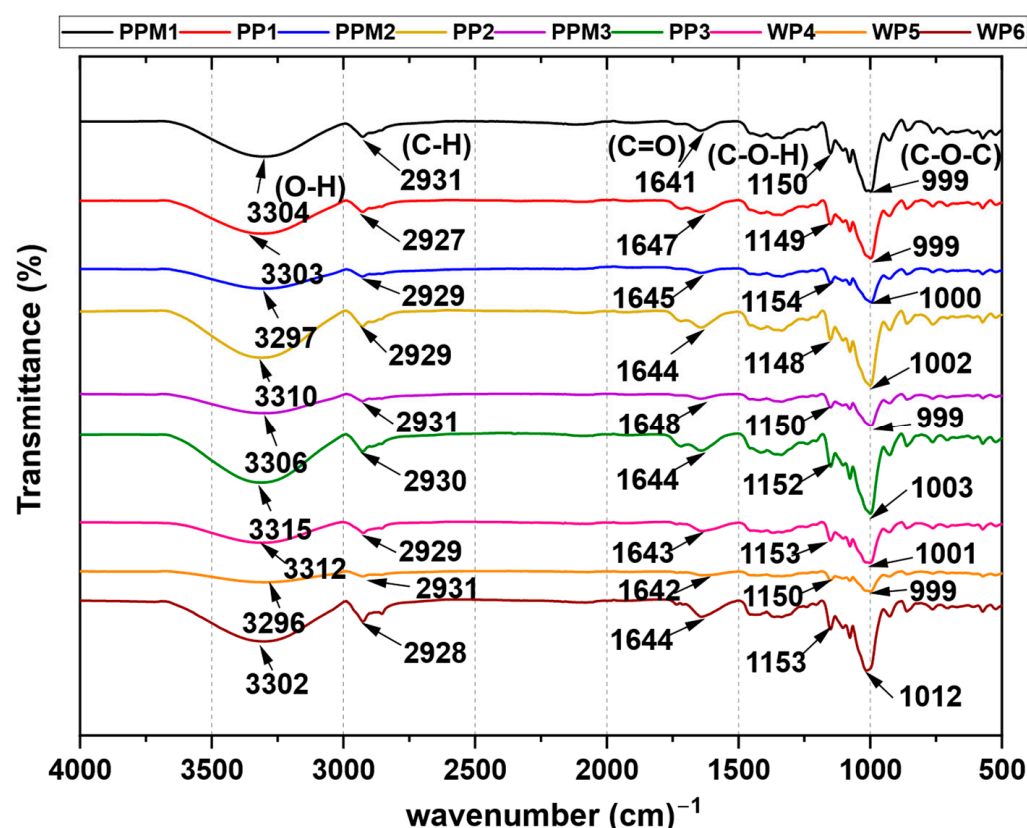


Figure 6. FT-IR spectra of starch and glycerin films. PPM1 (starch/glycerin 3/0.6 g bisulfite treatment); PP1 (starch/glycerin 3/0.6 g); PPM2 (starch/glycerin 4/0.8 g bisulfite treatment); PP2 (starch/glycerin 4/0.8 g); PPM3 (starch/glycerin 5/1 g bisulfite treatment); PP3 (starch/glycerin 5/1 g); WP4 (starch/glycerin 4/0.6 g); WP5 (starch/glycerin 4/0.8 g); WP6 (starch/glycerin 4/1 g). Reproduced from [82], licensed under CC BY 4.0.

3.7. Scanning Electron Microscopy (SEM) of Green Banana Starch/Glycerin Films

Figure 7 shows scanning electron micrographs of the cross-section of the green banana starch/glycerin films. For formulations PPM1–PP3, the plasticizer concentration was kept constant by weight, whereas for WP4–WP6, it varied among 15%, 20%, and 25%. The films exhibited relatively homogeneous structures, with some defects attributable to the dispersion of glycerin between the polymer chains [88]. In solution, starch forms an ordered or semi-ordered network due to interactions among polymer chains; however, when it interacts with glycerin, these intermolecular interactions decrease, as will be seen later in the XRD analysis, increasing the mobility of the polymer chains. The drop-casting technique used to prepare the film has limitations in producing highly homogeneous films, which explains the irregularities observed in the micrographs and in their thickness [89]. In addition, the varying shapes and sizes of the starch granules shown in Figure 5 are reflected in films with rough surfaces, and the cross-cutting method (fracturing the frozen film in liquid nitrogen) may have introduced some of the observed defects. Finally, although no noticeable pores were observed, this roughness may be attributed to a less compact internal structure, as corroborated by the X-ray patterns in Figure 8, which indicate reduced crystallinity.

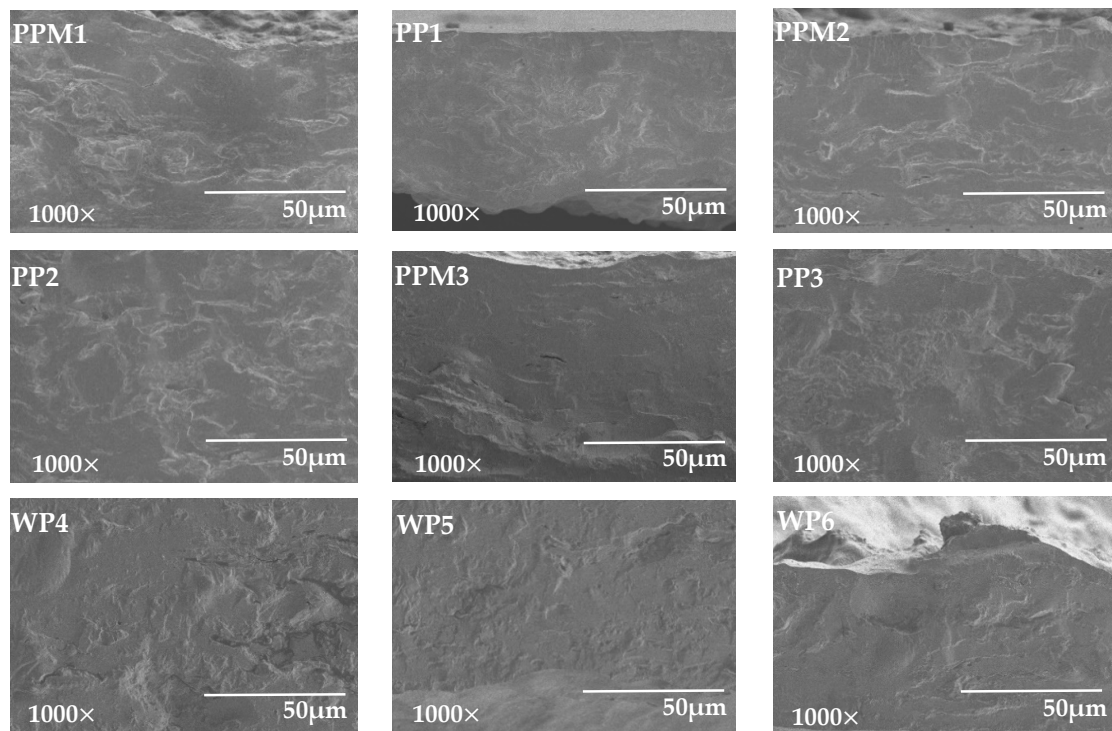


Figure 7. SEM micrographs of starch and glycerin films. Reproduced from [82] licensed under CC BY 4.0.

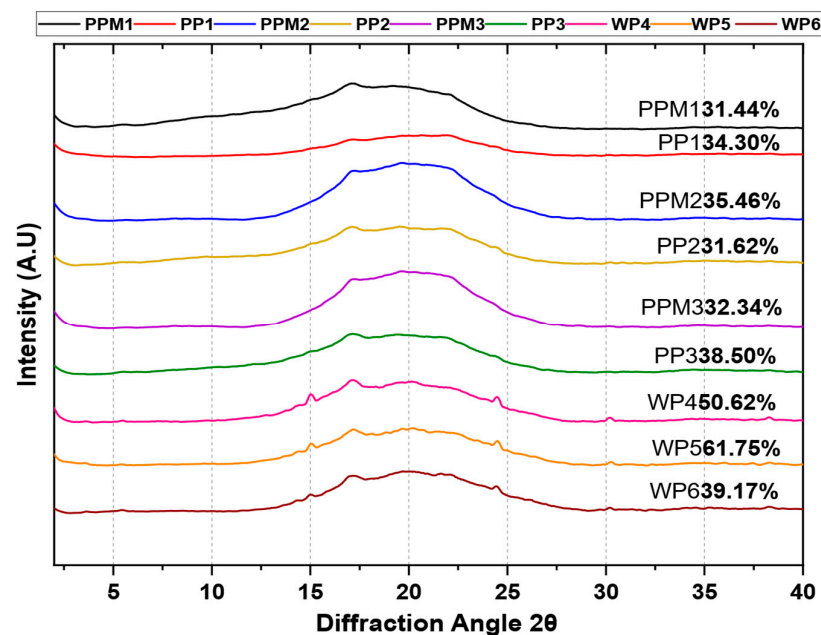


Figure 8. XRD spectra of starch and glycerin films. Reproduced from [82], licensed under CC BY 4.0.

3.8. X-Ray Diffraction (XRD) of Green Banana/Glycerin Films

X-ray diffraction spectra show a semicrystalline structure with amorphous regions in the starch/glycerin films. In Figure 8, the modified starch films exhibit C-type crystallinity, equivalent to a mixture of A- and B-type polymorphs that depend on the botanical origin of the banana starch [90]. Some peaks detected in the films with $2\theta = 15^\circ$, 17° , 19° , 22° and 24° are present in C-type starches, suggesting this mixture [91]. The C-type crystalline structure presents a combination of A- and B-type crystallites, with hexagonal unit cells (type B) surrounded by monocyclic unit cells (type A) that provide greater packing in the structure to form the starch granule, constituting a polymorphic

structure [92]. A study of native banana starch films and those oxidized with glycerol confirms the coexistence of A- and B-type polymorphs in their crystallinity, as evidenced by peaks $2\theta = 17.6^\circ$, 19° , and 26° [93]. For WP4 to WP6, a peak is observed at $2\theta = 30^\circ$, corresponding to a crystalline reflection of the cellulose [94].

For PPM1 to PP3, no significant difference is observed in the X-ray diffraction spectra, despite variations in the amounts of starch and glycerin. For films prepared with starch treated with metabisulfite, the diffraction spectra are less intense, possibly due to interactions between the nucleophilic centers of the starch (hydroxyl groups) and the electrophilic sulfur centers of the metabisulfite, as previously mentioned. The amorphous regions of the starch granules are formed by the branching points of amylopectin and amylose [95]. The diffractograms of WP4 to WP6 show greater ordering of the starch molecules when all the banana peel waste is used for starch extraction. As shown in the proximate analysis, this is attributed to the presence of other components, such as carbohydrates, lipids, and polyunsaturated fatty acids (especially linoleic and α -linolenic acids present in the peel), which can promote a more ordered arrangement and, therefore, greater crystallinity [96]. The amylose–amylopectin ratio, molecular mass, degree of branching, and the length of the external amylopectin chains can also influence their crystalline structures [97].

Furthermore, the degree of crystallinity (shown in Figure 8) of the lamellae was calculated using Equation (1). It was observed that for lamellae with starch content of 4 g, the treatment appeared to favor the crystalline region, which consists of the amylopectin double helices [98]. For WP4–WP6, where the whole peel was used, the crystallinity index increased compared to that of the parenchyma. It was also observed that with increasing amounts of plasticizer, the crystallinity index decreased. This is because the addition of glycerol can decrease the degree of crystallinity without altering the X-ray pattern of the starch [99]. Possibly, the glycerol added to the mixture decreased the ability of the chains to form crystals and their mobility. In addition, the hydroxyl group of glycerol forms strong hydrogen bonds with the starch chains, which decreases the interaction (intra- and intermolecular) between them [100]. WP6 showed the highest IC, indicating that, in moderate amounts, glycerin can facilitate the mobility and organization of starch and cellulose chains present in the shell.

3.9. Thermogravimetric Analysis of Green Banana Starch/Glycerin Films

Thermogravimetric analysis allows evaluation of material degradation and determination of weight changes by assessing the material's resistance to weight changes over time when the sample is subjected to a controlled temperature increase [101]. This analysis of the films reveals the temperature conditions at which the material begins to decompose. In the thermogram (Figure 9), the films exhibit the first weight loss between 80 and 130 °C, attributed to the loss of volatile molecules from the samples, including water, glycerol, and alcohol groups [102]. The second weight loss, occurring between 210 and 350 °C, corresponds to the structural degradation of the starch films due to the depolymerization of the polysaccharide chain [101], resulting in an approximate weight loss of 60%, the greatest among the films. Likewise, the third stage of mass loss, between 350 °C and 700 °C, can be attributed to the complete decomposition of the material, with the release of carbon compounds and gases (CO_2 , CO, and H_2O).

The TG derivative represents the mean decomposition temperature and allows us to identify how quickly a material decomposes [101]. Figure 10 shows the weight loss derivative (DTG) curve, where the peak corresponds to the thermal degradation temperature (T_{dmax}). The thermogram shows the T_{dmax} values for the formulations, which range from 283 °C to 302 °C. A pronounced degradation peak is evident, mainly attributable to the decomposition and depolymerization of the starch's organic components, observed in the

second stage of thermal degradation in TGA (Figure 9). No pronounced peaks are observed in the dehydration zones of the films, as the material has not yet undergone the greatest weight loss. In the thermal degradation behaviors where metabisulfite was used (PPM1, PPM2, and PPM3), temperatures above 300 °C were observed, which can be attributed to the interactions between metabisulfite and starch, as discussed in previous studies [103].

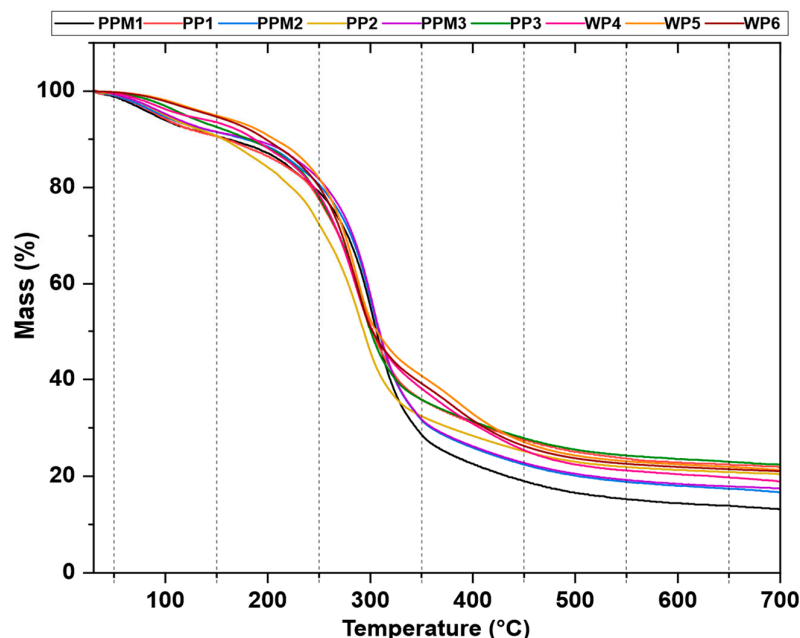


Figure 9. Thermograms (TGA) of starch and glycerin films. Reproduced from [82], licensed under CC BY 4.0.

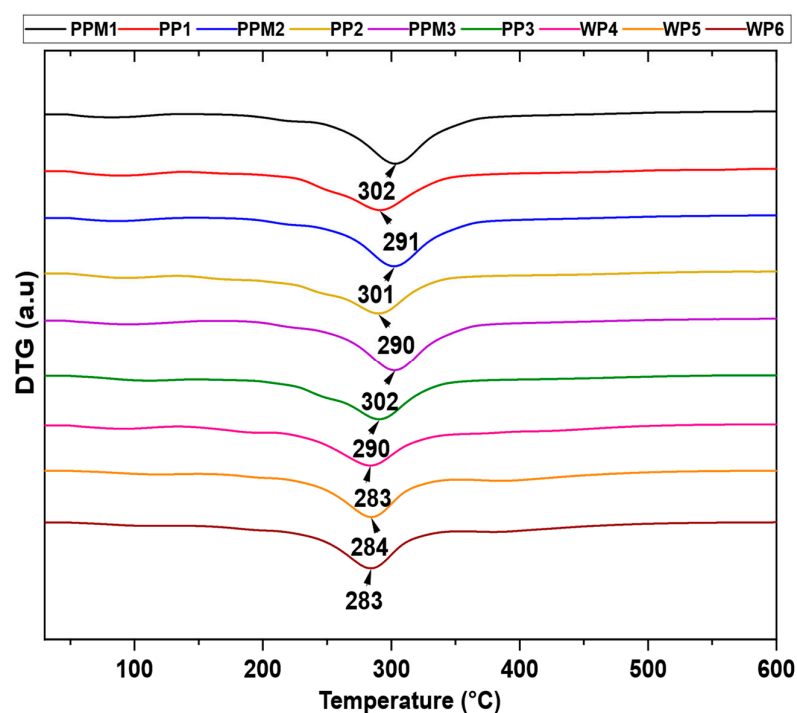


Figure 10. Thermograms (DTG) of starch and glycerin films. Reproduced from [82], licensed under CC BY 4.0.

3.10. Dynamic Mechanical Analysis (DMA) of Green Banana Starch/Glycerin Films

The storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$) curves were used to determine the stiffness and viscoelastic properties of each of the developed films. The storage modulus (E') indicates the elastic behavior of the material within a temperature range; this behavior is perceived as the material's capacity to store energy for future use, providing information about its stiffness under cyclic stress [104,105]. As shown in Figure 11, in the range of -100 °C to 60 °C, the E' of the films decreases as the temperature increases. As the temperature rises, a sharp drop in the curve is observed in the lower temperature range, indicating that the material transitions from a glassy to a rubbery state [106]. The E' modulus was high in almost all formulations with varying starch content (PPM1-PP3), indicating that a high starch content in the film may restrict movement within the matrix and impart stiffness properties [107]. PP2 (the film without pretreatment) exhibited the highest E' , indicating greater stiffness compared to the other formulations; however, at 25 °C, it is more flexible than the others, as later confirmed by the Young's modulus tensile test. For PPM2 and PPM3, E' decreased with metabisulfite pretreatment. However, this relationship does not hold as the temperature increases; that is, in the plastic region, the films exhibit different behaviors at different temperatures. For PPM1, E' increased with pretreatment and remained constant; it can be said that a low starch content treated with metabisulfite improves stress transfer and adhesion with the plasticizer, resulting in a stiffer film [108].

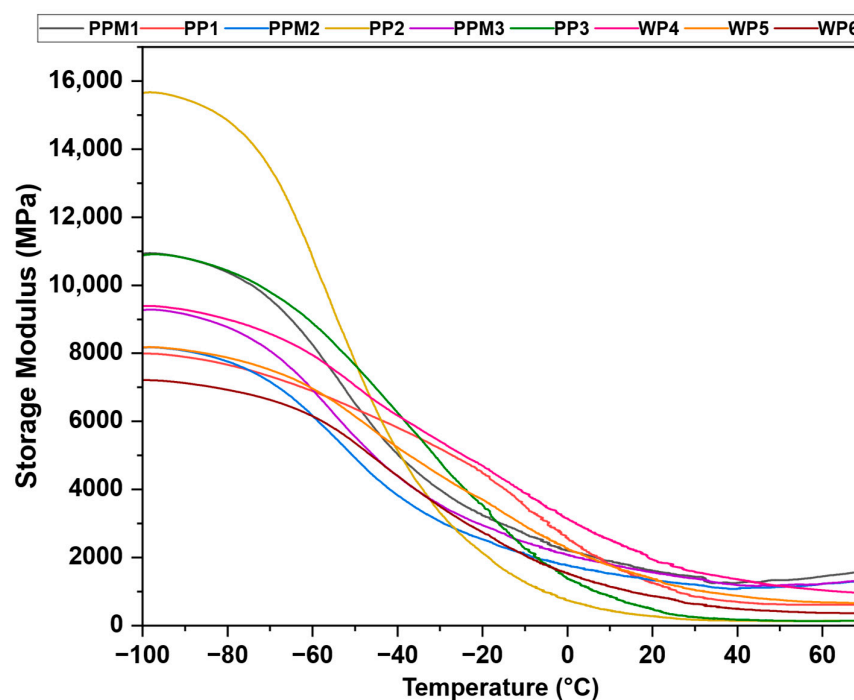


Figure 11. Storage modulus vs. temperature of starch and glycerin films. Reproduced from [82] licensed under CC BY 4.0.

Typically, incorporating a material into the polymer matrix facilitates the application of mechanical stress, as this stress is transferred to the reinforcement and enhances filler dispersion and material compatibility when combined [109]. A study using bagasse fiber as a reinforcing material in starch increased the E' value and improved its mechanical properties [110]. Films with varying plasticizer concentrations (WP4-WP6) exhibited low storage modulus, demonstrating a low degree of stiffness. This indicates that increasing the amount of plasticizer causes it to interpose between the polymer chains, weakening

intramolecular interactions and increasing mobility. This results in a less rigid material, as seen in WP6, with a lower E' value in the formulations.

Another study [111] described how the softening of the material is due to the addition of plasticizing molecules, which increases its ductility and improves its flexibility. The loss modulus (E'') curve shown in Figure 12 is a viscous response of the material and is perceived as the energy dissipated under cyclically applied deformation [112]. The peaks observed in E'' indicate chain mobility due to the highly amorphous state, primarily of amylopectin, and partial mechanical degradation [113]. E'' depends on the amount of starch, the amount of plasticizer, and the use of metabisulfite pretreatment as the temperature increases; it is evident that PP2 presented the highest E'' compared to other formulations [114]. They reported that the high starch nanoparticle content, increased glycerol content, and a gelatinization time of 60 min improved the films' loss modulus.

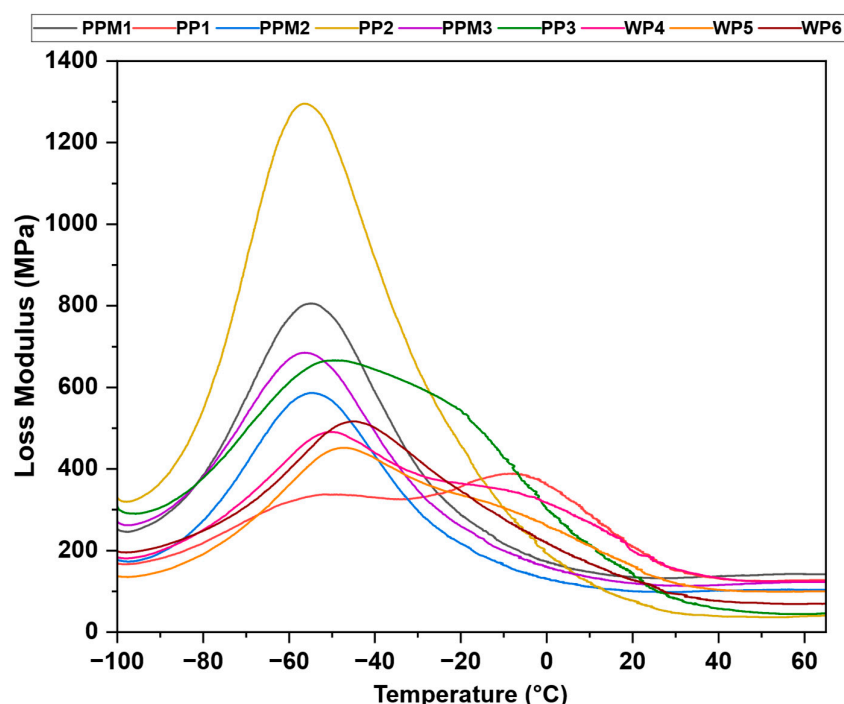


Figure 12. DMA of loss modulus vs. temperature of starch and glycerin films. Reproduced from [82] licensed under CC BY 4.0.

In formulations where the amount of glycerin was varied (WP4–WP6), a shift in the peaks associated with the glass transition temperature (T_g) was observed, and at higher temperatures, the peaks became flat. Studies using glycerol as a plasticizer have shown that the flat peaks are related to the evaporation of water molecules rather than relaxation transitions, indicating that a material with glycerin has a higher moisture content, generating thermal instability in the material [115]. A further increase in temperature will not change the elastic modulus, but the material could begin to thermally degrade, approaching the first degradation zone observed in the TGA.

In the glassy regime, elastic behavior with limited mechanical energy dissipation is observed. Upon passing through a glass transition (T_g), long-range cooperative macromolecular movements are activated, and mechanical energy dissipation increases [116]. In the films obtained, this transition occurs at approximately -60°C , which corresponds to the T_g of glycerol. This behavior is typical of plasticized biopolymeric systems such as those developed by [117], who identified a glass transition temperature of approximately -63°C , attributing it to the partial miscibility between starch and glycerol. Other stud-

ies have reported a T_g for glycerol between $-50\text{ }^{\circ}\text{C}$ and $-60\text{ }^{\circ}\text{C}$, reflecting the level of interactions between the plasticizer and the polysaccharides [118].

The damping factor ($\tan \delta$) shown in Figure 13 relates to elastic ($E' > E''$) and ($E'' < E'$) behavior. These results show mechanical relaxations associated with the glass transition temperature (T_g); this macromolecular mobility occurs between $-60\text{ }^{\circ}\text{C}$ and $-40\text{ }^{\circ}\text{C}$ for starch [119]. For the parenchyma starch formulations with metabisulfite (PPM1, PPM2, and PPM3), more clearly defined mechanical relaxation peaks are observed, suggesting that metabisulfite pretreatment during starch extraction improves compatibility between the polymer and the plasticizer matrix. A high peak indicates greater mobility of the polymer chains and greater miscibility between the polymer and the plasticizer [120,121]; in turn, this is related to the elongation at break in the mechanical tests. As for the peaks of the parenchyma starch films without metabisulfite (PP1, PP2, and PP3), they differ in behavior; it is possible that the material is not predominantly homogeneous; that is, there is a plasticized phase as the dominant matrix and also another more crystalline or less plasticized phase that softens at higher temperatures [122].

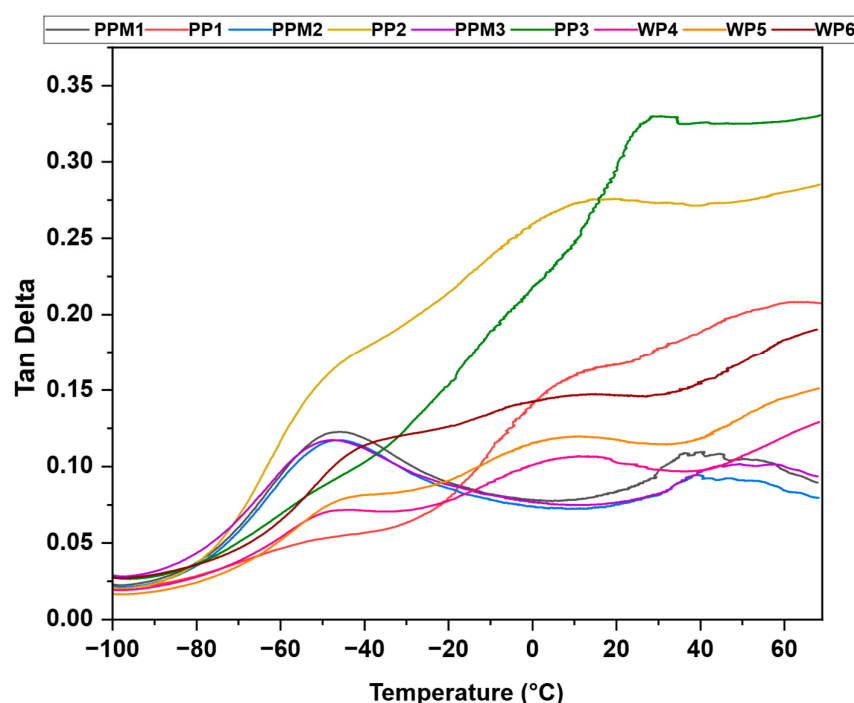


Figure 13. DMA of tan delta vs. temperature of starch and glycerin films. Reproduced from [82], licensed under CC BY 4.0.

3.11. Mechanical Properties of Green Banana Starch/Glycerin Films (Tensile Tests)

Table 3 shows the Young's modulus, maximum tensile strength, and breaking strain values for the green banana peel starch and glycerin films. Statistical analysis indicated that both individual factors and their interactions significantly affected ($p < 0.05$) the mechanical properties of the samples, except for Young's modulus, whose interaction was not significant ($p > 0.05$). Analysis of variance revealed that the presence of metabisulfite enhanced the mechanical strength of the films, resulting in higher Young's modulus and maximum tensile strength values. This may be related to interactions between the hydroxyl groups of starch and sodium metabisulfite, which could favor the formation of a film structure, thereby improving mechanical strength. In a study using thermoplastic starch and sodium metabisulfite, it was reported that the latter improved the mechanical strength of the films at low starch concentrations, where better dispersion of the starch chains was

achieved [123]. This effect was also observed in treatments PPM1, PPM2, and PPM3, with PPM1, which had a lower starch concentration, showing greater mechanical resistance.

Table 3. Mechanical properties of starch films obtained from green banana peels and glycerin.

Formulation	Young's Modulus (MPa)	Maximum Stress (MPa)	Tensile Breakage (%)
PPM1	673.79 ± 92.18	6.33 ± 1.04	2.82 ± 1.36
PP1	315.51 ± 158.6	4.47 ± 1.46	8.06 ± 2.85
PPM2	480.73 ± 78.50	10.37 ± 0.76	3.50 ± 0.36
PP2	44.66 ± 12.60	3.23 ± 0.12	22.64 ± 5.38
PPM3	633.52 ± 199.1	10.03 ± 0.81	4.29 ± 0.95
PP3	112.94 ± 6340	4.53 ± 0.23	21.59 ± 1.90
WP4	248.62 ± 39.49	3.38 ± 0.24	8.42 ± 0.54
WP5	123.23 ± 12.85	2.30 ± 0.38	14.42 ± 1.75
WP6	43.81 ± 12.50	1.26 ± 0.24	21.78 ± 0.85

On the other hand, the variation in glycerol concentration in treatments WP4, WP5, and WP6 significantly affected ($p < 0.05$) the mechanical properties of the films. The flexibility of the films increased with increasing glycerol content, as shown in Table 3. Conversely, stiffness decreased, as observed in the Young's modulus and maximum tensile strength values. Glycerol affects the mechanical properties of the film by acting as a plasticizer, reducing the intramolecular bonds between the polymer chains [124]. For formulations WP4, WP5, and WP6, the stiffness of the films decreased significantly with increasing glycerol content, as shown in previous studies [125]. The Young's modulus was in the range of 43.807 ± 12.50 MPa for WP6 and 248.62 ± 39.49 MPa for WP4. This is due to the compatibility of glycerol with starch, which facilitated the formation of new hydrogen bonds and played a role in the packing of amylose within the starch matrix [126]. Furthermore, the Young's modulus increased when using starch extracted from the whole peel instead of starch extracted only from the parenchyma. Comparing treatments PP2 and WP5, a 176% increase in modulus and a 36% reduction in maximum strain were observed, suggesting an increase in mechanical strength. This could be related to the addition of whole-peel fiber, which likely reinforces the matrix, as reported in other studies using chestnut shell fiber [127].

3.12. Water Vapor Permeability (WVP) of Green Banana Starch/Glycerin Films

Water vapor permeability (WVP) values are presented in Table 4. Starch concentration and the presence of metabisulfite did not significantly affect the WVP values ($p > 0.05$) or the interaction. This indicates that formulations PPM1 to PP3 did not exhibit significant variation with respect to the factors studied, as shown in Table 4. Formulations WP4 to WP6, however, showed significant differences ($p < 0.05$) due to variation in glycerol concentration. It was observed that increasing the glycerol content increased the water vapor permeability of the films. This may be related to an increase in active sites for hydrogen bonding on the film surface due to increased plasticizer content, which favors the transfer of water vapor molecules through these sites. This is due to the polyhydroxyl nature of glycerol [128]. The formulations using whole-peel starch (WP4, WP5, and WP6) exhibited the highest values among the formulations made with parenchyma starch. This may be because untreated fibers are typically highly hygroscopic, which facilitates the diffusion of water vapor through the films. In the food industry, low water vapor permeability is expected to reduce or prevent moisture transmission [129]. The WVP values of the films ranged from 0.139 to 3.411 WVP (g mm/kPa h m²). The values obtained for the films in this study

are higher than those for conventional materials, such as low-density polyethylene, which range from 0.003 to 0.0035 WVP (g mm/kPa hm²) [130].

Table 4. Water vapor permeability, water solubility, and contact angle of starch from green banana peels and glycerin.

Formulation	WVP (g mm/kPa hm ²)	Water Solubility (%)	Contact Angle (°)	
			Left	Right
PPM1	0.197 ± 0.011	9.07 ± 0.243	84.14 ± 5.89	84.97 ± 5.85
PP1	0.169 ± 0.026	9.08 ± 0.313	66.98 ± 1.98	63.56 ± 4.78
PPM2	0.139 ± 0.006	7.51 ± 0.364	73.21 ± 8.88	71.19 ± 11.32
PP2	0.167 ± 0.013	8.37 ± 0.301	41.20 ± 1.50	40.32 ± 3.177
PPM3	0.161 ± 0.038	8.28 ± 0.614	50.55 ± 6.32	49.35 ± 4.95
PP3	0.175 ± 0.010	8.16 ± 0.058	41.40 ± 6.04	42.00 ± 5.20
WP4	2.454 ± 0.081	9.31 ± 0.393	43.22 ± 1.63	40.94 ± 2.47
WP5	3.122 ± 0.108	10.46 ± 0.465	41.11 ± 4.31	40.61 ± 4.26
WP6	3.411 ± 0.084	11.27 ± 0.274	41.61 ± 4.77	38.84 ± 8.74

3.13. Water Solubility (WS) of Green Banana Starch/Glycerin Films

The water solubility (WS) values are presented in Table 4. The analysis of variance indicated that the starch concentration significantly affected the water solubility of the films ($p < 0.05$). In contrast, the presence of metabisulfite and the interaction between the two factors did not significantly affect said solubility ($p > 0.05$).

The solubility of the films ranged from $7.51 \pm 0.364\%$ to $9.08 \pm 0.313\%$ across the factorial design treatments. Formulation PPM2 exhibited the lowest solubility, while PP1 showed the highest. This behavior may be due to the fact that the bond between sulfite and hydroxyl groups introduces sulfur atoms into the starch chains [131]. This improves the hydrophobicity of the matrices. Therefore, a starch concentration of 4% is most favorable for the formation of these chains, which decreases their solubility. This same phenomenon was observed in the WVP formulation, where PPM2 presented the lowest value. Regarding the films made with whole-peel starch, the ANOVA showed that the glycerol concentration significantly affected the films' solubility ($p < 0.05$). Similar to what was observed in WVP, the samples from treatments WP4, WP5, and WP6 showed higher solubility, likely due to the presence of cellulose and lignin, which promoted hydrogen bonding with water. Natural cellulose is characterized by its high polarity due to the abundance of hydroxyl groups [132]. An increasing trend was observed with increasing glycerol concentration due to increased hydrogen bond formation. This is reflected as the percentage of glycerol increases, favoring the bioplastic's binding to water [130]. Increasing the plasticizer concentration modifies the polymer's molecular network and decreases the interaction density of the starch network, leading to increased water solubility [133]. Other studies have also reported increased film solubility with increased film solubility with increasing plasticizer concentration, as the plasticizer positively affects water solubility due to its hydrophilic properties [134]. Glycerol has linear chains, which favor its insertion between polymer–polymer chains.

3.14. Contact Angle of Green Banana Starch/Glycerin Films

The contact angles (CA) measured on the films are shown in Table 4. This property indicates the wettability of the film surface, evaluating its ability to attract or repel water [135]. Angles less than 90° were observed, indicating that the films exhibited hydrophilic surfaces with increasing surface polarity. This was possibly due to the addition of the plasticizer, which decreased the contact angle due to its hygroscopic nature and the formation of hydrogen bonds within the film matrix. In the parenchyma starch films, formulations PPM1, PP1, and PPM2 showed larger contact angles than the others, indicating decreased

surface hydrophilicity. As the contact angle increases, the potential interaction between the water droplet and the film decreases. For films made for water, interactions between polymer molecules can be reduced, making them more soluble due to their hydrophilic nature, as evidenced by low contact angle values [136,137]. It was reported that the low contact angle was due to the addition of the plasticizer glycerin, which improves flexibility by decreasing the contact angle. Therefore, the films' affinity for water increases with the number of hydroxyl groups. Likewise, these films exhibit higher WVP and CA values, which are related to water vapor molecules that can be more easily adsorbed on the surface and to the content of highly hygroscopic fiber, suggesting that there could be more polar sites on the surface of these films [138].

3.15. Optimization of the Formulation of Green Banana Starch/Glycerin Films

To determine the optimal formulation of green banana peel starch films with glycerin using an extraction methodology and a factorial design, the objective was to maximize mechanical properties while minimizing water solubility and water vapor permeability. Maximizing tensile strength was considered a key objective, as the mechanical property values of a conventional low-density polyethylene (LDPE) film, which reports a tensile strength of 8 MPa, were used as a reference [139]. Given the use of these films for food packaging, low water solubility and water vapor permeability values are necessary, as the film will come into direct contact with food. To optimize formulations PPM1 through PP3 based on solubility and maximum stress, the response variables with significant effects from both factors (starch concentration and the presence or absence of metabisulfite) were prioritized. In formulations WP4 through WP6, where the amount of glycerin was varied, all variables showed significant effects. Table 5 presents the optimization criteria for each response variable.

Table 5. Optimization criteria for the factorial design for green banana starch films.

	Response Variable	Lower	Upper	Target
Parenchyma (PPM1-PP3)	Maximum stress (MPa)	3.23	10.37	Maximize
	Water solubility (%)	7.51	9.07	Minimize
Whole Peel (WP4-WP6)	Young's modulus (MPa)	43.80	248.6	Maximize
	Maximum stress (MPa)	1.26	3.38	Maximize
	Max strain (%)	8.42	21.78	Minimize
	Water solubility (%)	9.31	11.27	Minimize
	WVP (g mm/kPa hm ²)	2.454	3.411	Minimize

The optimized solution for films with starch extracted from the parenchyma was the formulation with 4% starch and 20% glycerin, treated with metabisulfite (PPM2). The optimized solution for films with starch extracted from the whole peel was the formulation with 4% starch and 15% glycerin (WP4). The overall desirability coefficients were 0.8685 (87% prediction accuracy) and 0.9041 (90% prediction accuracy), respectively. The optimal conditions for the dependent variables were validated to ensure the fit of the obtained models. Experiments were performed in quintuplicate to compare the experimental results with the predicted values of the response variables (Table 6). For films PPM1 to PP3, the predicted values for the maximum stress and WS variables were 10.37 MPa and 7.50%, respectively, with a predictability of $0.83 < d < 0.89$. For films WP4 to WP6, the predicted values for Young's modulus, maximum stress, tensile rupture, WS, and WVP were 248.62 MPa, 3.38 MPa, 8.42%, 9.31%, and 58.90 g mm/kPa hm², respectively, with a predictability of $0.84 < d < 0.96$.

Table 6. Optimal solution for film formulation.

Formulation	Response Variable	Predicted Response	Validated Response	RMSE
Parenchyma PPM1-PP3	Maximum stress (MPa)	10.36	10.37 ± 0.7612	0.6216
	Water solubility (%)	7.509	7.0803 ± 0.1480	0.4457
Whole Peel WP4-WP6	Young's modulus (MPa)	248.62	278.17 ± 19.7	33.650
	Maximum stress (MPa)	3.38	3.81 ± 0.33	0.5143
	Max strain (%)	8.42	8.49 ± 0.84	0.6919
	Water solubility (%)	9.31	9.53 ± 0.32	0.3472
	WVP (g mm/kPa dm ²)	58.90	59.88 ± 1.03	1.2952

4. Conclusions

Green banana starch extracted from the peel and treated with sodium metabisulfite showed great potential for producing biodegradable films from food waste, contributing to the recovery of agroindustrial waste and the circular economy. In this study, green banana starch and glycerin films with good mechanical properties were obtained. The starch extracted from the entire peel waste exhibited a higher protein and ash content. FT-IR analysis of the three starch types, from the parenchyma (with and without sodium metabisulfite) and from the whole peel, confirmed the presence of their functional groups, such as the hydroxyl and ether groups characteristic of the glycosidic bond. Furthermore, the presence of hydroxyl and methyl groups from the starch and glycerin was observed in the resulting films, displaced by chemical interactions between their functional groups. In the DSC analysis of the starches, the gelatinization temperatures were 91 °C, 85 °C, and 71 °C for untreated starch, starch treated with metabisulfite, and starch from the whole peel, respectively, due to the effect of metabisulfite on the granular structure, as evidenced by their SEM micrographs. Thermogravimetric analysis of the starches showed thermal degradation temperatures ranging from 289 °C to 318 °C, indicating depolymerization of their polysaccharide chains. For the films obtained using parenchyma starch, a degradation temperature above 300 °C was observed; this behavior may be attributed to sodium metabisulfite treatment during starch extraction, which may have influenced the material's structural characteristics. The SEM analysis showed relatively homogeneous films, with irregularities due to the drop-casting method and manual fracturing for cross-sectional observation. XRD analysis of the films revealed a semicrystalline structure, with amorphous regions attributed to starch granules and exhibiting C-type crystallinity. Dynamic mechanical analysis of the films showed glass transition temperatures (T_g) between −60 °C and −40 °C; it is presumed that the viscoelastic behavior is influenced by the density of the polymer network, the added agents, and the mobility afforded by low-molecular-weight plasticizers. Furthermore, the presence of sodium metabisulfite and the low starch content were found to enhance the mechanical strength of the films. At the same time, water vapor pressure (WVP) was affected by variations in glycerin concentration. The water solubility of the films decreased in the presence of sodium metabisulfite in parenchyma-containing films and increased with increasing plasticizer concentration. The optimized and statistically validated formulation was PPM2, with 4% starch, sodium metabisulfite treatment, and 20% glycerin, prepared only from the fruit parenchyma. Using the complete waste from green banana peels, the optimized and statistically validated formulation was WP4, with 4% starch and 15% glycerin. However, PPM2 exhibited a film with greater matrix homogeneity, demonstrating better mechanical and barrier properties. Regarding the extraction methodology, starch extracted from the fruit parenchyma had a higher total carbohydrate content and lower fiber content, yielding cleaner starch with fewer impurities.

However, the limitations of this study highlight the need for further research before its application in the food-packaging industry can be supported. For example, researchers should study additional barrier properties such as oxygen permeability, carbon dioxide permeability, optical properties, and film biodegradability under standardized conditions; conduct storage tests with dry foods; and perform overall and specific migration tests of starch and glycerol components into foods to demonstrate that the developed film is safe for direct contact with food. It is also important to note that starches were obtained from samples collected from different regions using extraction methods specific to each plant matrix. Therefore, we recommend that future studies use residues from the same location and crop variety and follow a standardized extraction protocol to isolate the effect of each variable. Finally, this study was conducted on a laboratory scale, and further research is needed to evaluate its industrial scalability, production costs, feasibility, and regulatory compliance for single-use applications involving dry foods before its commercial implementation can be considered.

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Data Availability Statement: The data supporting the results of this study are available in the article. The FTIR spectra of starch and glycerin films, as well as their TGA, DTG, and XRD data, SEM micrographs, and dynamic mechanical analysis (DMA) figures for green plantain starch and glycerin films (storage modulus, loss modulus, and tan delta), were previously presented in a preliminary poster at the 5th International Electronic Conference on Food (Session: New Preservation and Packaging Technologies) [82]. This manuscript substantially expands upon these preliminary results through additional characterization of the films, including mechanical properties, WPV, WS, and CA; characterization of the starches; optimization of the green banana starch and glycerin film formulations; data analysis; and interpretation. All reused material is properly cited and complies with the terms of the original publication's license (reproduced from [82] under a CC BY 4.0 license).

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