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Low-starch potato peel for the development of starch-based composites obtained by thermocompression moulding

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In this study, the effect of incorporating low-starch potato peel, whether thermally treated or not, into starch-based films on their functional properties was evaluated. The films, obtained by melt blending and thermo-compression moulding, were characterised in terms of barrier, thermal and optical properties together with the microstructure, FTIR analysis and antioxidant activity. The potato peel particles subjected to the thermal pre-treatment exhibited a more homogeneous size distribution and increased phenolic content. The incorporation of potato peel into the starch matrix led to films that were mechanically more reinforced as compared to the neat starch films (up to 233% and up to 448% higher tensile strength and elastic modulus, respectively) with improved water vapor barrier capacity (up to 30%). In general, these improvements were slightly greater in films incorporating the thermally pre-treated potato peel, likely because these films presented a better integration of the residue components into the polymeric matrix. Furthermore, the composite films presented strong light barrier capacity and exhibited notable antioxidant activity, which could be of interest to limit oxidative processes in oxidation-sensitive foods. Thus, these results showed that it is possible to obtain films with up to 50 g/100 g starch (or 27 g/100 g film) of low-starch potato peel with enhanced properties at a lower cost.

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Sustainability spotlight

Potato peel is a major by-product of potato product manufacturing with an interesting composition to be used in the development of biodegradable materials for food packaging with antioxidant properties. This alternative may help to reduce the environmental impact of petroleum-based plastics, while valorising this low-cost agro-industrial waste. The aim of this study is to evaluate the effect of low-starch potato peel containing bioactive compounds on the properties of thermo-processed starch-based films obtained by melt blending and compression moulding. This study supports SDGs 12, 13 and 14 by developing an eco-friendly packaging material for food preservation and it is aligned with the circular economy concept.

1. Introduction

In recent years, the search for biobased biodegradable materials, as a sustainable alternative for food packaging, has intensified due to the growing need to mitigate the environmental impact of petroleum-based plastics. However, the transition to biodegradable packaging continues to face challenges such as limited scalability, high production costs, technological constraints, and the need for adequate waste management systems and supporting infrastructure.[1] Thus, economic sustainability is one of the key factors to be taken into account to ensure that biodegradable materials can effectively compete in the global market. In this regard, starch is a good candidate for the development of biobased biodegradable materials for food packaging applications[2] as it is an abundant, biodegradable and low-cost polysaccharide that can be easily converted into a thermoplastic material with the aid of plasticizers. Starch-based films have good optical, organoleptic and gas barrier properties;[3] however, they present drawbacks compared to other synthetic-based polymers, primarily due to their poor moisture resistance and mechanical resistance.[4] These limitations can be reduced by using different strategies such as the use of reinforcing natural fibres.[5] In this context, the use of lignocellulosic wastes from agro-industrial activities may represent an interesting alternative to obtain starch-based packaging materials with improved properties for food preservation purposes, while revalorizing these residues. Usually, these lignocellulosic fibre materials are chemically treated, purified or compatibilized, in order to avoid their aggregation and to achieve proper adhesion between the matrix and the filler, thus ensuring their reinforcement effect.[6–8]

Potato peel is a major by-product of potato product manufacturing, ranging from 15% to 40% of the original flesh

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weight, depending on the peeling process.[9] This agro-industrial waste presents an interesting composition to be used as a reinforcing agent in starch-based films, while also providing the films with potential antioxidant activity. It contains carbohydrates (72–80% dry basis), including starch, cellulose, hemicellulose, pectin, and minor components such as free sugars (2.5–7.5%), proteins (10–17%), lipids (0.5–2%), ashes (7–15%) and different phenolic compounds with high antioxidant capacity.[10–13] Numerous studies have been found in the literature about films containing PoP components, such as starch, cellulose or phenols extracted from PoP, including biodegradation studies.[14,15] Nevertheless, only a few studies have focused on the use of entire PoP. Likewise, the studies carried out with entire PoP used high-starch content PoP (around 37–70%) to obtain films by a solvent casting method.[16–18] No previous studies have applied typical scalable thermoplastic processing methods, which are more widespread at the industrial level, to obtain starch-based composites.[19,20]

The composition of potato peel is greatly affected by the peeling process that allows for incorporating different ratios of the flesh components, such as starch. When using low-starch potato peel resulting from highly efficient abrasion industrial peeling, not enough starch is present in the product to produce films. This fact limits the film-forming capacity of the material, making it necessary to blend it with other polymers to obtain potato peel-based films. Several authors have reported that thermal pre-treatment of starch-containing residue may improve its compatibility with the polymer matrix. This hypothesis is based on the promotion of structural modifications by heat treatment that improve interfacial adhesion through the partial or total loss of crystallinity of the starch granules present in the residue.[21,22]

Therefore, the aim of this study is to evaluate the effect of low-starch potato peel containing bioactive compounds on the properties of thermo-processed starch-based films obtained by melt blending and compression moulding. Likewise, the influence of the thermal pre-treatment of the potato peel on its reinforcing properties was also analysed. Thus, films incorporating up to 50% potato peel (g potato peel/100 polymer), whether untreated (PoP) or thermally treated (PoP-T), were characterised in terms of their microstructure, and their barrier, mechanical, optical and thermal properties. The Fourier-transform infrared spectra (FTIR), total phenol content (TPC) and antioxidant activity of the films were also evaluated.

2. Materials and methods

2.1. Materials

Potato peel (var. *Mona Lisa*) was provided by an industrial company dedicated to the abrasive peeling of vegetables (Reproducciones Inoxidables, Alzira, Spain). Native potato starch (puriss. p.a.) was purchased from Sigma (Hamburg, Germany) and 99% pure glycerol was obtained from Panreac (*Castellar del Valles*, Spain). Magnesium nitrate ($Mg(NO_3)_2$) (98.0–102.0%) (Labkem, Barcelona, Spain), pentoxide (P_2O_5) (99%) (Sigma, Hamburg, Germany) and sodium chloride (NaCl) (≥99.5%) (Labkem, Barcelona, Spain) were used for sample conditioning. Folin reagent (2 M with respect to the acid) and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were provided by Sigma (Hamburg, Germany) and methanol (≥99.8%) by Labkem (Barcelona, Spain).

2.2. Potato peel powder preparation

Potato peel was dried for 96 h at 60 °C in a convection air oven (JP Selecta, Barcelona, Spain), until constant weight was reached, ground using a grinding machine (Thermomix TM6, Vorwerk, Wuppertal, Germany) at 10 200 rpm for 4 min and sieved (<250 μm) to obtain untreated potato peel (PoP). The thermal pre-treatment of the residue was carried out by mixing PoP with distilled water in a 0.03 : 1 (w/w) ratio and then heating the mixture in a water bath at 95 °C for 30 min under constant agitation. Subsequently, the suspension was dried at 60 °C until constant weight to obtain thermally treated potato peel (PoP-T).

2.3. Potato peel characterization

Both PoP and PoP-T were characterized in terms of their particle size distribution, microstructure, FTIR spectra, thermal stability, total phenolic content and antioxidant activity. The proximate composition was assessed for PoP, as no differences are expected after the thermal pre-treatment. To evaluate the effect of the grinding and sieving steps, the particle size distribution and the microstructure of the raw potato peel were also characterized.

The proximate composition was analysed by the corresponding Association of Official Agricultural Chemists (AOAC) methods.[23] Moisture (AOAC 934.06), ash (AOAC 923.03), protein (AOAC 920.152, using a conversion factor of 6.25), fat (AOAC 983.23) and fibre (AOAC 978.10) contents of the samples were determined in triplicate. For the determination of total starch content, a Megazyme K-TSTA was used, following the AOAC 996.11 method[23] and measurements were carried out in triplicate.

The particle size distribution was obtained using a laser diffraction particle size analyser (Mastersizer® 2000, Malvern Instruments Ltd, Malvern, UK) equipped with a dry dispersion unit (Scirocco 2000). The particle size distribution curves were obtained in triplicate using Mie theory and particle refraction and absorption indices of 1.52 and 0.1, respectively. The mean particle sizes were analysed using the De Brouckere mean diameter ($D[4,3]$).

The microstructure was analysed by using a high-resolution field emission scanning electron microscope (GeminiSEM 500, Zeiss, Oxford Instruments, UK). To this aim, samples were mounted on support stubs, gold-coated and observed using a 3 kV accelerating voltage.

The thermal stability was evaluated in duplicate by thermogravimetric analysis (TGA) (TGA 1 Stare System analyser, Mettler-Toledo, Urtenen-schönbühl, Switzerland). Around 5 mg of each sample previously conditioned at 0% relative humidity (RH) for 10 days at 25 °C were placed in alumina pans and heated from 25 to 700 °C at 10 °C min^{-1} in a nitrogen environment (10 mL min^{-1}). From the TGA and their first derivative (DTGA) curves, the initial degradation temperature (T_{onset}), the

temperature at the maximum degradation rate (T_{peak}) and the residual mass at 700 °C were obtained.

The Fourier-transform infrared (FTIR) spectra were obtained in duplicate, by means of a FTIR spectrometer equipped with an attenuated total reflectance sampling module (Agilent Cary 630, Agilent Technologies Inc., Santa Clara, CA, USA) in the wavelength range of 4000–400 cm^{-1}, using a resolution of 2 cm^{-1} and 32 scans.

The total phenolic content (TPC) and the antioxidant capacity (EC_{50}) determinations were performed in triplicate. The extraction of the phenolic compounds from the sample was carried out using a solvent (80/20 methanol/water): sample ratio of 1 : 10 (w/v) for 20 h at room temperature under constant agitation (1000 rpm) in complete darkness. The Folin-Ciocalteu method was used to measure the TPC in the methanolic extracts filtered with a 22-micron syringe (Scharlab, Barcelona, Spain), using a UV-visible spectrophotometer (Evolution 201 Thermo Scientific, Waltham, USA) at 725 nm. The results were expressed in milligrams of gallic acid equivalent (GAE) per gram of dry sample using a gallic acid standard curve. The DPPH radical scavenging method was used to assess the sample antioxidant capacity, following the methodology described by Martin-Perez *et al.*[24] This antioxidant activity was expressed as the EC_{50} value, defined as the sample mass required to decrease the initial DPPH concentration by 50%.

2.4. Film preparation

Both neat thermoplastic starch (TPS) films, which were used as controls, and composites were obtained by melt-blending and thermocompression moulding. 75% RH pre-conditioned starch and glycerol (ratio 1 : 0.3) were blended with different untreated or thermally treated potato peel proportions (0, 10, 20, 30, 40 and 50% with respect to the total starch mass), previously pre-conditioned at 75% RH. These RH values were selected based on preliminary tests to obtain a homogeneous blend. The eleven formulations were named considering the amount of untreated (PoP) or thermally treated (PoP-T) potato peel incorporated. The melt blending process was conducted in an internal mixer (HAAKE™ PolyLab™ QC, Thermo Fisher Scientific, Osterode Am Harz, Germany) for 10 min at 160 °C and 50 rpm. The resulting blends were then cold ground and conditioned for 10 days at 25 °C and 53% RH. Thereafter, 4 g of the milled pellets were placed onto Teflon sheets, pre-heated at 160 °C for 2 min in a hot-plate hydraulic press (Model LP2, Labtech Engineering, Samutprakarn, Thailand), and then heated at 160 °C for 2 min at 50 bar, followed by 6 min at 100 bar, and finally cooled down for 3 min. The obtained films were equilibrated for 10 days at 25 °C at 53% or 0% RH using respectively $Mg(NO_3)_2$ or P_2O_5 oversaturated salt solutions, until further analysis.

2.5. Film characterization

The cross-sectional microstructure of the films was characterized using a high-resolution field emission scanning electron microscope (GeminiSEM 500, Zeiss, Oxford Instruments, UK). Pre-conditioned film samples at 0% RH were cryo-fractured in liquid nitrogen, mounted on aluminium stubs, and sputter-coated with a thin layer of gold prior to observation at 3 kV accelerating voltage.

The equilibrium water content of the films was determined gravimetrically in triplicate in a vacuum oven (VaciotemT, Selecta SA, Barcelona, Spain) at 105 °C for 48 h.

The mechanical properties of the films were determined using a texture analyser (TA.XT plus, Stable Micro Systems, Godalming, UK) following the American Society for Testing and Materials (ASTM) D882 standard method.[25] Film samples (2.5 × 10 cm), pre-conditioned at 53% RH, were stretched at a cross-head speed of 12.5 mm min^{-1} and room temperature until failure, using A-TG tensile grips set 50 mm apart at the start of the test. For each formulation, eight replicates were analysed. Stress–strain curves were obtained from the recorded force–displacement data, from which tensile strength (TS), elastic modulus (EM), and elongation at break (ε) were calculated. Film thickness was measured with a digital micrometre (COMECTA, Palmer, Spain; accuracy: 0.001 mm) at six random positions per sample.

Water vapor permeability (WVP) and oxygen permeability (OP) were determined at 25 °C in triplicate. WVP was measured under a RH gradient of 53–100% using the gravimetric method described in ASTM E96-95,[26] following the procedure reported by Martin-Perez *et al.*[24] OP was evaluated according to ASTM F1927 (ref. 27) with an oxygen permeation analyser (model 8108e, Systech Illinois, Lisle, USA) at 25 °C and 53% RH.

The optical properties of the films were evaluated in triplicate by recording their reflection spectra in the 400–700 nm range using a spectro-colorimeter (CM-3600d, Minolta Co., Tokyo, Japan) against black and white backgrounds, according Kubelka–Munk theory, from which the internal transmittance (T_i) and the infinite reflectance spectra were calculated.[28] From the infinite reflectance spectra, the CIELAB colour coordinates (L^*, a^*, b^*) were determined under a D65 illuminant and a 10° standard observer, which were used to calculate the chroma (C^*_{ab}) and hue angle (h^*_{ab}) values, together with the total colour difference (ΔE^*) between each composite film and the TPS control films. Furthermore, the % of transmittance in the UV-visible spectrum (200–700 nm) of the films was measured in triplicate using a UV-visible spectrophotometer (Evolution 201 Thermo Scientific, Waltham, USA).

TGA analysis and FTIR spectra of the pre-conditioned films (0% RH for 10 days at 25 °C) were performed as described in Section 2.3. Differential scanning calorimetry (DSC) was performed using a DSC instrument (Stare System, Mettler-Toledo Inc., Urtenen-Schönbühl, Switzerland). Film samples (5–10 mg), previously conditioned at 0% RH for 10 days at 25 °C, were sealed in aluminium pans and subjected to a heating–cooling–heating cycle: first heating from 25 °C to 160 °C at 10 °C min^{-1}, cooling to 25 °C, and reheating to 160 °C at the same rate. An empty sealed pan was used as a reference. All measurements were carried out in duplicate.

The TPC values of the composite films were determined by quantifying the phenolic compounds released by the films into the media (80 : 20 methanol : water, using a solvent : film ratio

of 50 : 2 (v/w)) described in Section 2.3, together with their corresponding antioxidant capacity.

2.6. Statistical analysis

The statistical analyses were carried out using analysis of variance (ANOVA) with Statgraphics Centurion XIX-X64 software (Statgraphics Technologies, Inc., The Plains, Virginia, USA). Significant differences were determined by Fisher's least significant difference (LSD) test at a 95% confidence level ($p < 0.05$).

3. Results and discussion

3.1. Characterization of potato peel

Potato peel (PoP and PoP-T) had a complex composition, characterised by a high total fibre content (57 ± 2%, dry basis (db)), proteins (8.3 ± 0.3%, db), low fat (1.78 ± 0.06%, db), moisture (3.3 ± 0.2%, db), starch (4.26 ± 0.10%, db) and remarkable ash content (24.8 ± 0.2% db). While most of these values agreed with those found in previous studies,[9,29–31] the starch content was lower than the values reported by other authors. This difference is probably due to several factors affecting the peel composition such as the peeling method, potato variety and degree of maturity.[17,18,32] On the other hand, the relatively high ash content likely reveals a certain degree of contamination from soil adhering to the potato peel.[33] Concerning the phenolic content, the TPC value for untreated potato peel (PoP) was 3.3 ± 0.2 mg GAE per g PoP, in agreement with those reported by other authors (0.5–3.59 mg GAE per g PoP db), depending on the solvent and extraction method used.[11,34–36] According to the literature, the potato peel contains more phenolic compounds than potato flesh (up to ten times higher),[9] with chlorogenic acid as its main phenolic compound, which can constitute up to 90% of the TPC. Other phenolic acids that can be found are caffeic, ferulic, gallic and protocatechuic acids.[35,37] The antioxidant activity of PoP, evaluated using the EC_{50} value, was around 49.7 ± 1.4 mg PoP per mg DPPH. The thermal pre-treatment of the PoP increased its total phenol content (8.55 ± 0.14 mg GAE

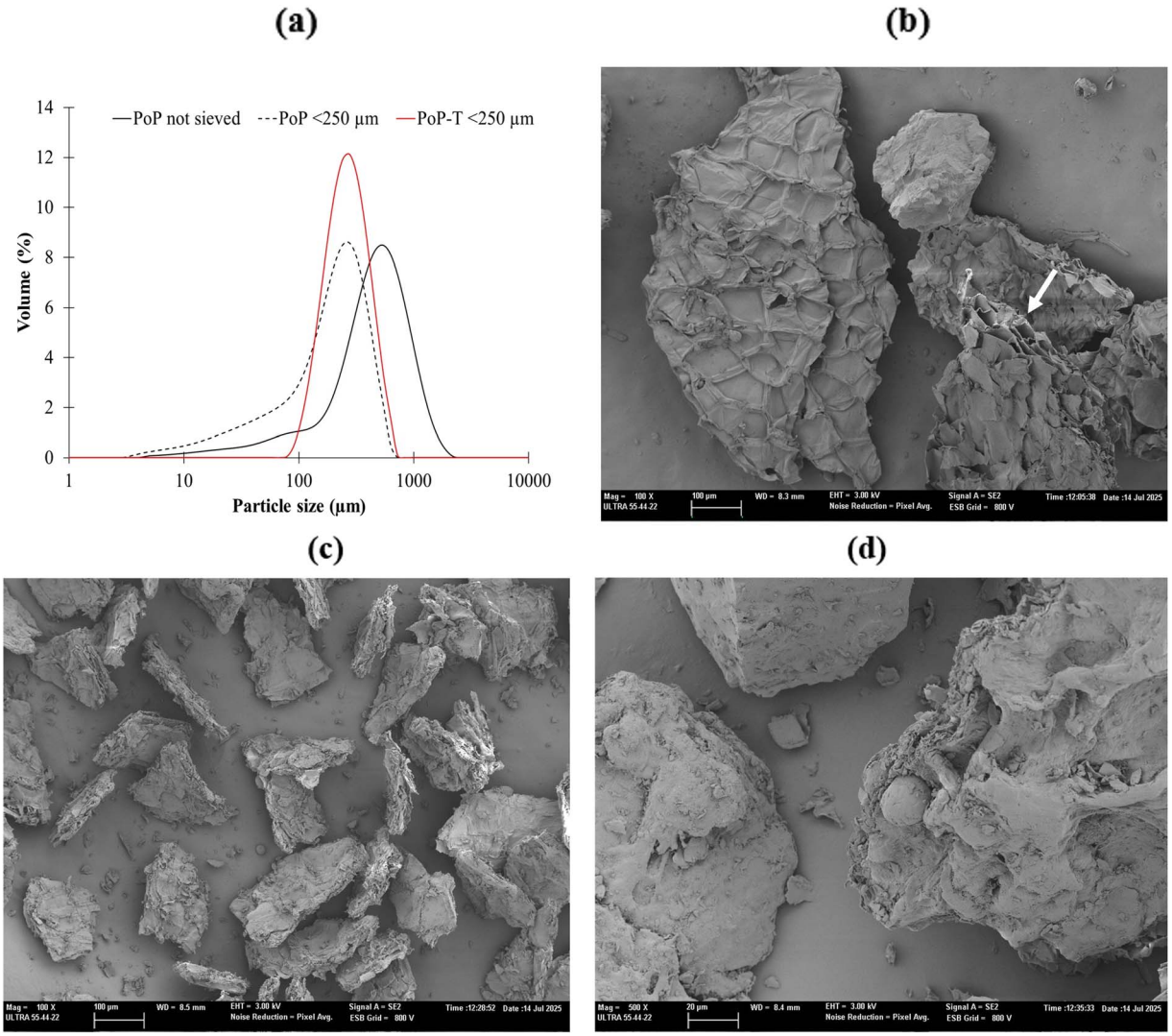

Fig. 1 Particle size distribution curves of raw, untreated (PoP) and thermally treated potato peel (PoP-T) (a) and FESEM micrographs (100× magnification) of raw potato peel (b) and PoP samples (c and d).

per g PoP-T) and, coherently, its antioxidant activity (22.5 ± 0.7 mg PoP-T per mg DPPH). This enhancement could be attributed to the thermal pre-treatment (90 °C for 30 min), which promoted a higher release of phenolic compounds.[38]

The particle size of the vegetal residue incorporated in the composites plays an important role in their functional properties, as polymeric matrices interrupted by large particles can have negative effects on the homogeneous appearance of the films and on their mechanical and barrier properties.[30,39] In general, the smaller the particles, the higher the interfacial area of the filler and therefore, the less these properties are affected.[30] Fig. 1 shows the particle size distribution curves of raw potato peel, PoP and PoP-T samples, and the field emission scanning electron microscopy (FESEM) micrographs for raw potato peel and PoP only, as no remarkable differences were found between the microstructures of PoP and PoP-T samples. As expected, the initial particle size of raw potato peel was drastically reduced after the milling and sieving processes, which shifted the size distribution curves towards smaller particle sizes. Thus, the fine PoP and PoP-T fractions exhibited monomodal size distributions, with particle sizes below 700 μm. In the PoP-T sample, the finest particles present in the raw and PoP samples, mainly consisting of starch granules and small cellular fragments, completely disappeared due to the thermal treatment, thus leading to a more homogeneous particle size distribution. The De Brouckere mean diameter ($D[4,3]$) was 482 ± 12 μm, 195 ± 2 μm and 306 ± 16 μm for non-sieved, PoP and PoP-T, respectively. Given that the PoP-T particles presented a significantly greater particle size in comparison with PoP ($p < 0.05$), these results suggested that the thermal pre-treatment promoted the agglomeration of the potato peel particles.

Microstructural analysis of the raw potato peel revealed compact and regular polygonal structures, typical of the potato epidermis.[40] Additionally, multi-layered cell structures were observed in some cross-sectional fragments (the arrow in Fig. 1b), corresponding to the cuticle, epidermis, and epithelial cells, in agreement with the histological features previously reported for potato peel tissues.[40] The ground and sieved fractions exhibited smaller particles, where starch granules adhered to the vegetal tissue were visible only in the untreated potato peel (PoP) (Fig. 1d).

The thermal stability of potato peel samples was analysed through TGA and its derivative (DGTA) curves. These results are presented in Fig. 2 and Table 1 only for the PoP sample, as the pre-treatment did not induce significant differences in the thermal stability of the samples ($p > 0.05$). As can be observed in Fig. 2, at least three thermal events could be identified in the potato peel sample. In the first step, small mass losses (about 6%) related to the bound water and degradation of low

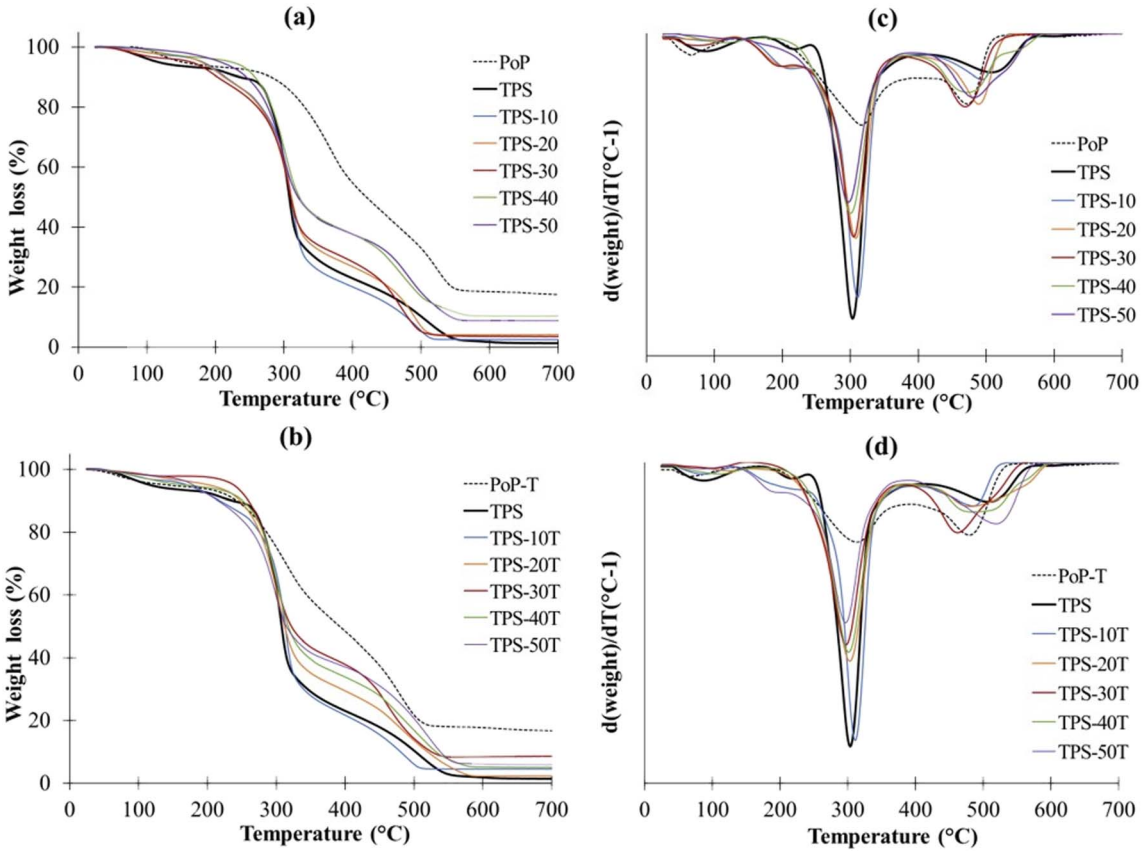

Fig. 2 TGA (a and b) and DTGA (c and d) curves of potato peel (PoP and PoP-T), control (TPS) and composite films incorporating different concentrations of untreated and thermally treated potato peel.

Table 1 Thermal degradation parameters of the potato peel, control (TPS) and composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel (mean values ± standard deviation)[a]

	1st event [40–240 °C]		2nd event [240–400 °C]		3rd event [400–700 °C]		
Sample	T_{onset} (°C)	T_{peak} (°C)	T_{onset} (°C)	T_{peak1} (°C)	T_{peak2} (°C)	T_{endset} (°C)	Residual mass at 700 °C (%)
Potato peel	42 ± 3^{bc}	68.1 ± 0.6^{h}	137.4 ± 0.5^{e}	316.5 ± 0.7^{a}	475 ± 5^{efg}	531 ± 4^{e}	17.4 ± 0.3^{a}
TPS	49 ± 8^{ab}	103 ± 3^{bcd}	149 ± 2^{cd}	306.3 ± 0.5^{c}	537 ± 10^{a}	581 ± 2^{bc}	1.4 ± 0.2^{e}
TPS-10	40.6 ± 0.6^{c}	97 ± 2^{cde}	144.2 ± 0.2^{de}	310.8 ± 1.2^{b}	494.8 ± 1.3^{cd}	547 ± 4^{de}	2.4 ± 0.2^{de}
TPS-20	49.8 ± 1.3^{ab}	95 ± 8^{de}	136 ± 2^{e}	307.2 ± 0.7^{c}	481 ± 11^{defg}	553 ± 11^{de}	4.2 ± 0.3^{cd}
TPS-30	43 ± 2^{bc}	76 ± 11^{gh}	138.5 ± 0.7^{e}	305.09 ± 0.12^{d}	470.5 ± 0.7^{fg}	549 ± 2^{de}	3.6 ± 0.3^{cd}
TPS-40	48 ± 3^{abc}	111 ± 3^{ab}	170 ± 13^{a}	300.7 ± 1.2^{e}	477 ± 14^{efg}	560 ± 37^{cd}	10 ± 2^{b}
TPS-50	50.31 ± 1.02^{ab}	109 ± 6^{ab}	141 ± 6^{de}	297.4 ± 0.6^{f}	486 ± 6^{def}	563 ± 5^{cd}	9 ± 2^{b}
TPS-10T	49 ± 3^{abc}	89 ± 5^{ef}	139.0 ± 1.4^{e}	310.9 ± 0.4^{b}	487 ± 3^{def}	551.1 ± 0.5^{de}	5 ± 3^{cd}
TPS-20T	46.1 ± 1.2^{abc}	118 ± 8^{a}	156 ± 3^{bc}	301.7 ± 0.7^{e}	490 ± 6^{de}	605.1 ± 1.2^{a}	3.7 ± 1.3^{cde}
TPS-30T	53 ± 4^{a}	104 ± 3^{bcd}	162 ± 5^{ab}	297.0 ± 0.1^{f}	464 ± 4^{g}	565 ± 8^{cd}	5.3 ± 1.2^{cd}
TPS-40T	44.9 ± 0.5^{abc}	107 ± 2^{bc}	157 ± 6^{bc}	301.1 ± 0.6^{e}	512 ± 11^{bc}	586 ± 16^{abc}	4.99 ± 0.06^{c}
TPS-50T	48 ± 6^{abc}	82 ± 2^{fg}	136 ± 5^{e}	296.5 ± 0.7^{f}	525 ± 11^{ab}	601 ± 12^{ab}	5.9 ± 0.9^{cd}

[a] Different letters in each column indicate significant differences ($p < 0.05$) among samples.

molecular mass volatile compounds are observed.[40] A second wide degradation event starting at 137.5 °C, corresponding to the overlapped degradation of polymeric constituents of the plant residue, can be attributed to the decomposition of hemicellulose (150–350 °C), cellulose (290–360 °C), starch (300 °C), and a fraction of the total lignin (160–900 °C). In this second event, the main mass losses occurred (about 75%). The third event, with a temperature at which the maximum degradation rate occurred of 474 °C (T_{peak}) (Table 1), is related to the decomposition of the residual lignin and the products from the fragmentation of the organic structures previously thermodegraded.[41–43]

FTIR measurements in the range of 400–4000 cm^{-1} were performed for the PoP and PoP-T samples. Nevertheless, in Fig. 4a only the FTIR spectrum of PoP particles is shown, as no differences were found due to the applied thermal treatment. The FTIR spectrum of PoP, which agreed with those found by other authors,[44,45] revealed a band between 3000 and 3500 cm^{-1}, attributed to the presence of free and hydrogen bonded –OH group stretching vibrations bands. The C–H stretching vibration showed a peak at 2918 cm^{-1} and a typical band at 1735 cm^{-1} due to the presence of lipids. The peak at 1632 cm^{-1} is ascribed to the existence of free or bonded water molecules, whereas the peak around 1017 cm^{-1} is attributed to the presence of C–O–C vibrations of pyranose sugar rings (from carbohydrates).

3.2. Characterization of composite films

3.2.1. Film morphology. Microstructural observations are valuable to better understand the properties of the composite films.[2] The cryo-fractured cross-section FESEM micrographs of the composite films with different concentrations of potato peel (0, 10, 30 and 50%), treated or not, are shown in Fig. 3. The TPS films exhibited the typical homogeneous matrix of the thermoplasticised starch film, as reported by other authors.[28,46] The presence of a heterogeneously fractured layer near the film surface reveals the progress of starch crystallization in this area due to its greater molecular mobility associated with water vapour diffusion.[39]

The incorporation of potato peel into the starch-based films resulted in a slightly more heterogeneous matrix, where the presence of fibre particles was evident, especially at high PoP concentrations. In films incorporating PoP, the fibres appeared superimposed on the starch matrix, and some gaps at the fibre–matrix interface were observed (*i.e.*, magnification in TPS-50). In contrast, the PoP-T particles were embedded and fully integrated in the starch matrix, making their visualization more difficult. These observations suggest high interfacial adhesion between the PoP-T particles and the starch phase. This is likely due to their greater phenolic content, which may promote the establishment of further hydrogen bonds with the hydroxyl groups of the polymer chains. The affinity and dispersion of the residue particles in the starch polymer matrix will determine their functional properties, especially those related to molecular mobility and diffusion, such as mechanical and barrier properties. Not only the phenolic compounds but also the cellulosic content of these low-starch PoP particles could contribute to benefiting the starch matrix/filler interactions due to the similar polysaccharide structure.[47] In this sense, Balakrishnan *et al.*[47] showed that the presence of nanocellulose fibers favoured the formation of constrained regions among the starch polymer chains that restricted their mobility, which led to more rigid films with higher water barrier capacity.

3.2.2. Thermal behaviour. Fig. 2 shows the thermal degradation curves determined by thermal gravimetric analysis (TGA and their first derivatives, DTGA) of the different films conditioned at 0% RH. The onset and endset temperatures, the temperature at the maximum degradation rate (T_{peak} in DTGA curves), and the residual mass are summarized in Table 1.

As can be observed, the TPS films exhibited the typical degradation behaviour of glycerol-plasticized starch, with three main thermal events as previously reported.[28,48–50] The first mass loss step (around 6%), between 40 and 150 °C, can be attributed to the loss of bonded water. The second degradation step,

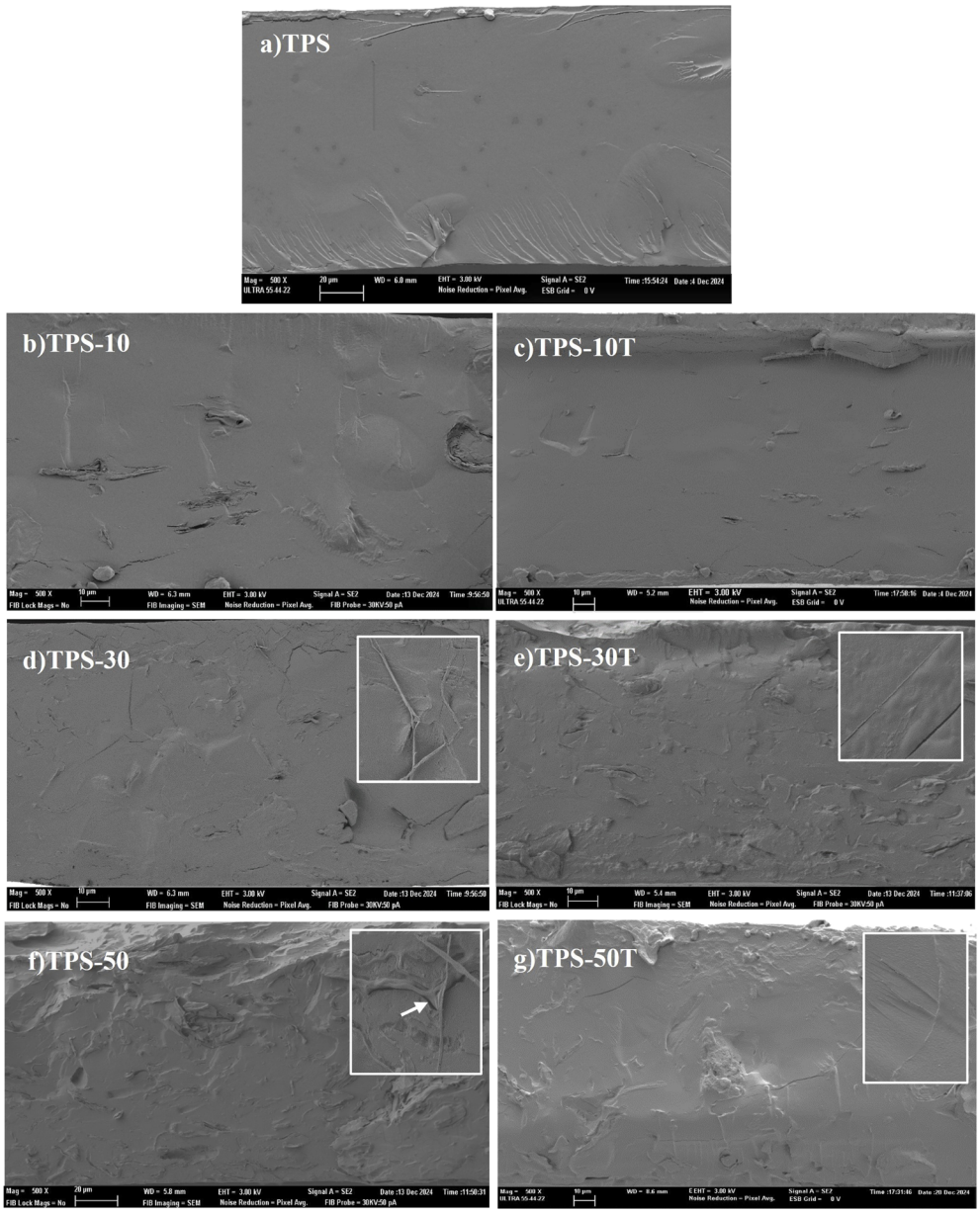

Fig. 3 FESEM micrographs (500× magnification) of the cross-sections of the thermoplastic starch film (TPS) (a) and those incorporating 10, 30 and 50% of untreated (b, d and f) and thermally treated (c, e and g) potato peel. The images embedded in each section (5000× magnification) show an enlarged detail of the section of the film.

between 150 and 340 °C, in which major mass losses occurred (around 70%), corresponded to the degradation of glycerol and starch through chain decomposition and depolymerization. The last degradation step, between 400 and 700 °C, can be attributed to the degradation of secondary products from the earlier stages.

The composite films displayed similar thermo-degradation patterns to the control films as the degradation peaks of the potato peel components overlapped. Overall, the thermal pre-treatment of the potato peel particles did not significantly ($p > 0.05$) affect the thermal stability of the starch-based films. The mass losses in the first event, approximately 3% for all composites, were lower than in control films. The major weight losses, between 140 and 340 °C, decreased with the increment in the potato peel content, in agreement with the greater thermostability of their main components (such as cellulose and lignin). Thus, this weight loss was around 70% for TPS-10 and TPS-10T films, whereas this value was approximately 55% for TPS-50 and TPS-50T films. In general, the temperature at which the maximum degradation rate occurred decreased (T_{peak}) when the potato peel content rose. This could be associated with the partial hydrolysis of the polymer containing potato peel, rich in phenolic acids and other hydrolysis-promoting compounds, during the thermoprocessing of the films, in agreement with the FTIR results shown below. Menzel[51] reported a similar trend in studies conducted with starch films incorporating phenolic

Table 2 Glass transition temperatures of the TPS and composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel, in the first (T_{g1}) and second heating scans (T_{g2}) (mean values ± standard deviation)[a]

Sample	Tg_1 (°C)	Tg_2 (°C)
TPS	78 ± 6[a]	80 ± 2[a]
TPS-10	66.7 ± 1.4[b]	74.1 ± 0.2[b]
TPS-20	77 ± 3[a]	84 ± 2[a]
TPS-30	59.9 ± 0.9[c]	61 ± 3[e]
TPS-40	63 ± 3[b]	63.0 ± 0.9[de]
TPS-50	51.9 ± 0.8[d]	66.6 ± 0.6[cd]
TPS-10T	63.4 ± 0.3[b]	68 ± 5[c]
TPS-20T	n.d	n.d
TPS-30T	65.3 ± 0.4[b]	62.2 ± 0.3[cd]
TPS-40T	74 ± 3[a]	75 ± 3[b]
TPS-50T	63 ± 3[b]	66.4 ± 0.1[cd]

[a] Different letters in each column indicate significant differences ($p < 0.05$) among samples.

extracts from rice straw. As expected, the residual mass values significantly increased when the potato peel in the films rose in the composite films. In general, these obtained residual mass values agreed with those theoretically predicted taking into account the mass fraction of the film components.

The glass transition temperatures (T_g) of the TPS and composite films, obtained in the first and second heating scans, are presented in Table 2. The T_g midpoint values of TPS films were lower than those obtained by other authors in studies carried out with less plasticised potato starch films. For instance, Moreno *et al.*[52] and Pooja *et al.*[53] reported T_g values of 138 and 125.9 °C, respectively, for potato starch films with glycerol ratios of 1 : 0.20 and 1 : 0.25. These greater T_g values are attributable to the lower plasticizer content used, which decreases polymer chain mobility. In general, the incorporation of PoP or PoP-T into TPS films significantly decreased ($p < 0.05$) the T_g values as the residue content rose. This plasticising effect could be attributed to: (1) the presence of low molecular weight compounds in potato peel (*i.e.*, lipids), which increased the free volume of the polymer chains and promoted their molecular mobility and (2) the presence of compounds in the potato peel, such as phenolic acids, which promoted the thermal degradation of the starch chains. Menzel *et al.*[46] also reported the plasticising effect of the phenolic-rich rice straw aqueous extracts on thermo-processed starch films, as a result of the reduction in the molecular weight of starch during the heat-shear treatment, melt blending and compression moulding process. The T_g values obtained in the second heating scan (T_{g2}) were greater than those observed in the first scan, which is

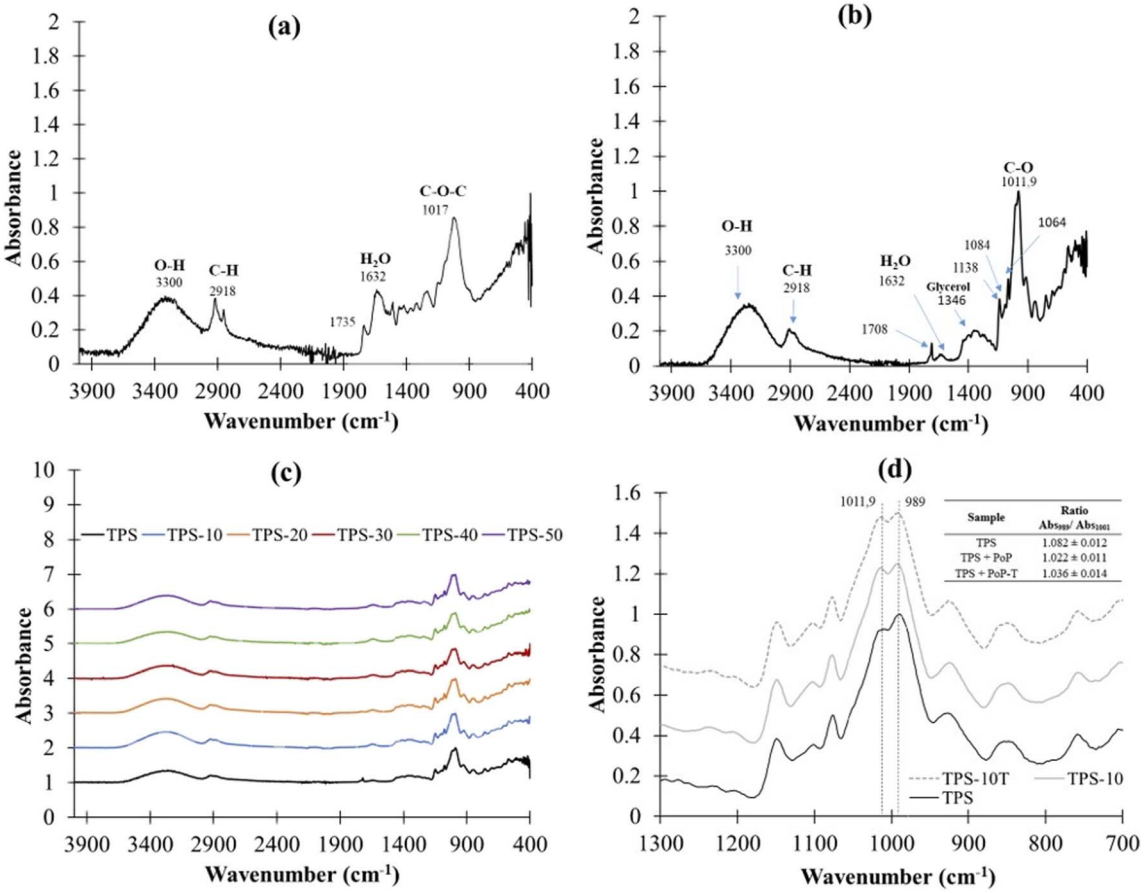

Fig. 4 FTIR spectra of potato peel (PoP) (a), control (TPS) (b), composite films incorporating different concentrations of PoP (c) and control and films incorporating 10% PoP (TPS-10) and 10% PoP-T (TPS-10T) in the 1300–700 cm^{-1} range (d).

Table 3 Total phenolic content (TPC) and antioxidant capacity (EC_{50} parameter) of composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel (mean values ± standard deviation[a]

Sample	TPC[1] (mg GAE per g dry film)	TPC[2] (mg GAE per g potato peel)	EC_{50} (mg potato peel per mg DPPH)
TPS-10	0.68 ± 0.02^{j}	9.5 ± 0.3^{g}	15.4 ± 0.3^{d}
TPS-20	1.74 ± 0.06^{g}	13.1 ± 0.4^{a}	14.3 ± 0.7^{f}
TPS-30	2.12 ± 0.05^{f}	11.3 ± 0.3^{e}	18.8 ± 0.6^{a}
TPS-40	2.50 ± 0.03^{d}	10.64 ± 0.11^{f}	14.4 ± 0.2^{ef}
TPS-50	3.02 ± 0.06^{b}	10.9 ± 0.2^{f}	13.2 ± 0.4^{g}
TPS-10T	0.91 ± 0.03^{i}	12.7 ± 0.4^{ab}	12.5 ± 0.2^{g}
TPS-20T	1.63 ± 0.08^{h}	12.2 ± 0.6^{cd}	17.6 ± 0.5^{b}
TPS-30T	2.23 ± 0.06^{e}	11.9 ± 0.3^{d}	16.6 ± 0.5^{c}
TPS-40T	2.91 ± 0.04^{c}	12.4 ± 0.2^{bc}	15.2 ± 0.4^{de}
TPS-50T	3.4 ± 0.2^{a}	12.3 ± 0.8^{bc}	17.1 ± 0.6^{bc}

[a] Different letters in each column indicate significant differences ($p < 0.05$) among formulations.

consistent with the loss of residual water content in the samples during the first heating. No significant differences were found in the T_g values of the films due to the thermal pre-treatment applied to the residue.

3.2.3. FTIR characterization. FTIR measurements in the range of 400–4000 cm^{-1} were performed for the control TPS films and the different composites (Fig. 4). In Fig. 4c, only the FTIR spectra of films incorporating PoP are shown as similar patterns were found in the PoP-T loaded formulations. In Fig. 4d, differences in the spectra in the 1300–700 cm^{-1} range of the control and TPS films incorporating 10% PoP and 10% PoP-T are also displayed.

In the TPS films, starch, glycerol and bound water contain –OH groups with stretching vibrational modes, which are responsible for the broad peak at 3300 cm^{-1} that dominates the starch spectra associated with intermolecular hydrogen bonds.[54,55] The peak at 2918 cm^{-1} was associated with the aliphatic saturated C–H stretching vibration of CH_2 groups. Additionally, an absorption band at 1708 cm^{-1} was observed, corresponding to carbonyl groups (C═O) attributed to a starch decomposition process during its thermoprocessing, which produces carbonyl groups such as aldehydes, ketones and carboxylic acids.[56,57] The peak at 1632 cm^{-1} is attributed to the O–H bending vibrations from absorbed and free water, whereas the peak at 1346 cm^{-1} is attributed to glycerol molecules.[58,59] The bands at 1138 and 1064 cm^{-1} are characteristic of TPS, corresponding to the C–O bonds of starch C–O–H groups.[60] Finally, the doublet around 989 cm^{-1} is typical of starch as a consequence of the presence of ordered structures and amorphous regions.[56,61]

As can be observed in Fig. 4c, similar FTIR spectra to those of the TPS films were found in the composite films, with potato peel characteristic bands overlapped with those of starch. In Fig. 4d, the typical doublet related to the presence of less and more ordered starch structures (around 1011.9 and 989 cm^{-1}, respectively) was clearly observed in all the samples. Nevertheless, the absorption band around 1011.9 cm^{-1} appeared more noticeable in the composites, which has been attributed to the presence of less ordered starch structures.[62] Thus, these results suggest that the formation of ordered starch structures was more inhibited in the composites than in the TPS films. The absorbance ratio of these two bands (Abs989 : Abs1011.9) has also been proven to positively correlate with a greater structural organization of starch.[61,62] This ratio was significantly lower in the composite films ($p < 0.05$), regardless of the potato peel concentration (table in Fig. 4d). This suggests that the presence of potato peel particles affected the structural arrangement of the starch chains, decreasing the interchain hydrogen bonds. On the one hand, the dispersed particles could interrupt the polymer chain association. On the other hand, the potential interactions of the released low molecular compounds from the particles, such as phenols, which can form hydrogen bonds with the polymer hydroxyls, could also affect the interchain hydrogen bonds. Likely, the partial hydrolysis of the polymer chains when potato peel is present, as deduced from TGA, could also influence the interchain association and structural arrangement.

3.2.4. Total phenolic content and antioxidant capacity. Table 3 shows the total phenolic content (TPC) and the antioxidant capacity (EC_{50} parameter) of the different composite films. The TPC values of the composite films, expressed as mg GAE per g film (TPC^1), significantly increased ($p < 0.05$) with the increase in the potato peel content. When the TPC of the films was referred to as mg GAE per g potato peel (TPC^2), the values remained almost constant (mean value: 11.1 ± 1.3 mg GAE per g PoP and 12.3 ± 0.3 mg GAE per g PoP-T) but significantly higher than the ones obtained for the residues (3.3–8.5 mg GAE per g residue). This could be explained by the neoformation of Maillard compounds with antioxidant activity during the film thermoprocessing at high temperatures, such as melanoidins and 5-hydroxymethylfurfural (HFM),[63,64] as previously observed by other authors working with thermoplastic starch films incorporating rice straw extracts.[42] As expected, the TPC content and the antiradical capacity of the PoP-T films were slightly greater ($p < 0.05$) than those with untreated potato peel (PoP).

The composite films exhibited lower EC_{50} values (mean value 15 ± 2 mg PoP per mg DPPH) than the ones observed in the residue (22.5–49.7 mg residue per mg DPPH), thus suggesting a greater antiradical activity surely because of the additional antioxidant activity of the neoformed compounds. The results

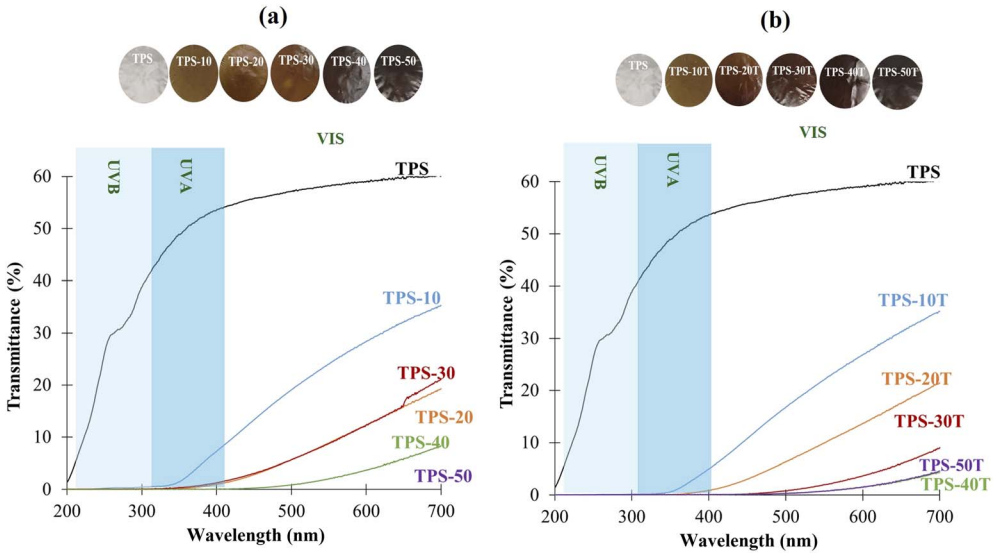

Fig. 5 Appearance and internal transmittance curves of control (TPS) and composite films incorporating different concentrations of untreated (a) and thermally treated (b) potato peel.

are in line with those reported by other studies incorporating active extracts from sunflower seed husks into thermoplastic starch matrices.[46] No clear trend in the EC_{50} values was observed due to the application of the thermal pre-treatment to the potato peel. The incorporation of these phenolic compounds with antioxidant capacity into films in combination with their UV light protection and oxygen barrier properties endowed these composite films with potential antioxidant activity that could be of interest to limit oxidative processes in oxidation-sensitive foodstuffs rich in unsaturated lipids or vitamins such as fruit juice, sunflower oil, nuts, fish and meat products, among others.

3.2.5. Optical properties. The optical properties of the films are directly related to the film microstructure and are affected by the surface and internal heterogeneity.[39] Fig. 5 shows the internal transmittance spectra (T_i) in the 200 to 700 nm range for each formulation and Table 4 summarizes the optical properties of the different films, in terms of luminosity (L^*), hue angle (h^*_{ab}) and chroma (C^*_{ab}),, together with the total colour difference with respect to the TPS films (ΔE^*) and film appearance. The TPS films were colourless and exhibited high transmittance values in agreement with their high degree of homogeneity and transparency. In contrast, composite films became opaquer (lower T_i values), with a reddish-brown, darker and more saturated colour compared to control films. These changes could be attributed to the differences in the refractive index of the polymer matrix due to the filler incorporation, which promotes light scattering and opacity in the films, to the intrinsic colour of the potato peel components and to the progress of Maillard reactions or caramelization of sugars present in the potato peel during the film thermoprocessing. The thermal pre-treatment of the potato peel led to films that are less transparent, darker and more reddish ($p < 0.05$) in comparison with the films incorporating PoP, as a consequence of the more intense heat treatment received. The described changes, evaluated using the colour difference values, were significantly greater ($p < 0.05$) when the mass fraction of potato peel in the film increased. Similar results were found for thermoplastic starch films with lignocellulosic extracts from rice and coffee husks[7] or rice straw,[28] where opacity and darkness increased with the residue incorporation.

As can be observed in Fig. 5, all the composite films exhibited strong light absorption at wavelengths below 550 nm, blocking up to 100% of light compared to the control film, thus acting as an effective light barrier. This effect was marked in films with higher phenolic content, achieved either by increasing the potato peel content or by using the thermally treated residue (PoP-T). This agrees with the strong absorbance of aromatic structures and functional groups present in the polyphenolic compounds linked to the vegetal lignocellulosic fibres and proteins.[65–68]

Table 4 Values of luminosity (L^*), hue angle (h^*_{ab}), chroma (C^*_{ab}) and total colour difference (ΔE^*) for the control (TPS) and composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel (mean values ± standard deviation)[a]

Sample	L^*	C^*_{ab}	h^*_{ab}	ΔE^*
TPS	95.4 ± 0.9^{a}	6.8 ± 0.4^{f}	88.2 ± 1.2^{a}	—
TPS-10	60 ± 2^{b}	19.1 ± 0.4^{a}	$72.4 \pm .6^{b}$	37.7 ± 1.6^{g}
TPS-20	48.4 ± 0.6^{d}	13.6 ± 1.2^{c}	64.4 ± 1.4^{c}	47.7 ± 0.5^{e}
TPS-30	43.0 ± 1.5^{f}	8 ± 2^{e}	52 ± 5^{d}	52.6 ± 1.4^{d}
TPS-40	40.6 ± 1.2^{gh}	4.0 ± 0.6^{h}	47 ± 2^{g}	54.99 ± 1.12^{bc}
TPS-50	40.2 ± 0.6^{ghi}	2.9 ± 0.6^{i}	49.8 ± 1.5^{efg}	56.5 ± 1.0^{a}
TPS-10T	57 ± 2^{c}	17.1 ± 0.5^{b}	73.3 ± 0.8^{b}	39.5 ± 1.8^{f}
TPS-20T	45 ± 2^{e}	10 ± 2^{d}	58 ± 4^{d}	51.3 ± 1.9^{d}
TPS-30T	41.1 ± 0.7^{g}	5.3 ± 0.7^{g}	48.5 ± 1.5^{fg}	54.4 ± 0.8^{c}
TPS-40T	37.3 ± 0.8^{hi}	2.8 ± 0.3^{i}	49 ± 2^{fg}	56.2 ± 0.5^{ab}
TPS-50T	39.2 ± 1.0^{i}	1.8 ± 0.4^{j}	50 ± 7^{ef}	55.5 ± 0.5^{abc}

[a] Different letters in each column indicate significant differences ($p < 0.05$) among formulations.

Table 5 Water content (x_w), thickness (t), oxygen permeability (OP) and water vapor permeability (WVP) of the control (TPS) and composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel (mean values ± standard deviation)[a]

Sample	x_w (g water/100 g film)	t (mm)	OP × 10^{14} (cm^3 m^{-1} d^{-1} Pa^{-1})	WVP × 10^{11} (g Pa^{-1} s^{-1} m^{-1})
TPS	8.6 ± 0.6^{ab}	0.181 ± 0.015^{b}	8.4 ± 1.4^{f}	256 ± 14^{c}
TPS-10	8.3 ± 0.3^{bc}	0.13 ± 0.05^{f}	20.8 ± 0.6^{b}	303 ± 7^{ab}
TPS-20	9.22 ± 0.14^{a}	0.157 ± 0.015^{def}	30.8 ± 1.3^{a}	326 ± 12^{a}
TPS-30	6.72 ± 0.06^{ef}	0.154 ± 0.008^{ef}	15.0 ± 1.4^{cd}	236 ± 7^{cde}
TPS-40	6.11 ± 0.14^{fg}	0.167 ± 0.02^{cde}	16.84 ± 0.12^{c}	238 ± 5^{cd}
TPS-50	5.4 ± 0.2^{gh}	0.18 ± 0.02^{bc}	14.6 ± 1.4^{de}	180 ± 17^{f}
TPS-10T	7.6 ± 0.3^{cd}	0.147 ± 0.009^{f}	12.82 ± 0.05^{de}	285 ± 18^{b}
TPS-20T	6.93 ± 1.11^{de}	0.149 ± 0.001^{f}	8.0 ± 0.2^{f}	239 ± 11^{cd}
TPS-30T	7.4 ± 0.3^{de}	0.180 ± 0.002^{bc}	16.1 ± 0.2^{c}	232 ± 18^{cde}
TPS-40T	5.3 ± 0.3^{h}	0.1693 ± 0.0106^{bcd}	11.9 ± 1.2^{e}	211 ± 26^{e}
TPS-50T	5.78 ± 0.07^{gh}	0.206 ± 0.006^{a}	6.94 ± 1.6^{f}	219 ± 15^{de}

[a] Different letters in each column indicate significant differences ($p < 0.05$) among formulations.

Preventing the transmission of UV radiation, in combination with the appropriate oxygen barrier and antioxidant properties of the film, represents an advantage for its application as a packaging material for food sensitive to oxidation reactions. Moll *et al.*[69] also analysed and reported the protective effect against oxidation of brown-coloured materials rich in antioxidant substances derived from raw straw.

3.2.6. Water content, thickness and barrier properties. The equilibrium water content (x_w), thickness (t), water vapour permeability (WVP) and oxygen permeability (OP) values of the films are shown in Table 5. The obtained values for the neat starch films are similar to those observed by other authors working with thermoplastic potato starch.[7,28,46,52] In general, the incorporation of potato peel (PoP or PoP-T) led to a significant reduction in the film's water content, consistent with the TGA results, and in their thickness. The presence of high fibre content in the potato peel, constituted by closely packed cellulose chains through interchain hydrogen bonds, and the diffusion of phenolic compounds from the vegetal tissue into the polymeric matrix could modify its water binding capacity through the formation of hydrogen bonds with the hydroxyl groups of the polymer chains, blocking these active points for water binding.[70]

On the other hand, the higher flowability of the composite blend during the thermocompression step may be responsible for the lower thickness of the composite films.[71] Nevertheless, this flowability was compromised at high potato peel contents (50%), leading to films with thickness values similar to the ones observed for the control films.

The effect of potato peel incorporation on the WVP and OP values depended on the filler concentration and pre-treatment. Low potato peel contents (up to 20% for PoP and up to 10% for PoP-T) increased the WVP values of the films ($p < 0.05$), whereas the opposite was observed when the filler content rose. Thus, WVP values decreased up to 41% in the composites with 50% potato peel as compared to the control, regardless of the filler pre-treatment. Similarly, the OP values notably increased ($p < 0.05$) with the potato peel addition, but this effect was less marked with higher PoP incorporation. Gomez-Contreras *et al.*[72] showed that the incorporation of beer bagasse and the increment in the filler : polymer ratio worsened the gas barrier capacity of the starch-based films. This was mainly attributed to the filler aggregation and the hydrophilic nature of the beer bagasse that promoted the transport of water molecules.

The barrier properties of the films are affected by several factors such as the effective tortuosity factor for the diffusion of both water vapor and oxygen molecules through the film, the degree of filler dispersion within the polymeric matrix and the interfacial adhesion forces between components, among others.[8,70] Thus, high filler contents could promote the tortuosity factor associated with the potato peel particle distribution along the polymer matrix, which would limit the diffusion rate of gas molecules (water and oxygen) through the films. Moreover, the lower water content of these films (with high potato peel content) also contributed to a reduced plasticisation of the polymer matrix, further decreasing the WVP values. In general, PoP-T loaded films presented significantly higher oxygen barrier capacity ($p < 0.05$) than those containing PoP. The diffusion of phenolic compounds from the phenolic-richest potato peel particles (PoP-T) within the TPS matrix may positively affect the oxygen barrier property of the polymer, likely

Table 6 Values of elastic modulus (EM), tensile strength (TS) and elongation at break (ε) for the control (TPS) and composite films incorporating different concentrations of untreated (PoP) and thermally treated (PoP-T) potato peel (mean values ± standard deviation)[a]

Sample	EM (MPa)	TS (MPa)	ε (%)
TPS	85 ± 26^{d}	3.7 ± 0.8^{h}	40 ± 5^{a}
TPS-10	113 ± 15^{cd}	6.69 ± 1.06^{cde}	18 ± 2^{b}
TPS-20	109 ± 23^{cd}	4.4 ± 0.5^{gh}	12 ± 3^{d}
TPS-30	282 ± 48^{b}	6.5 ± 0.8^{de}	6 ± 2^{ef}
TPS-40	287 ± 71^{b}	7.16 ± 1.06^{cd}	7 ± 2^{e}
TPS-50	466 ± 93^{a}	8.9 ± 0.2^{b}	3.2 ± 1.4^{fg}
TPS-10T	117 ± 20^{cd}	5.9 ± 1.3^{ef}	15 ± 3^{bc}
TPS-20T	169 ± 45^{c}	5.0 ± 0.9^{fg}	11 ± 3^{d}
TPS-30T	146 ± 32^{cd}	6.8 ± 0.4^{cde}	10 ± 4^{cd}
TPS-40T	366 ± 91^{b}	8.0 ± 0.5^{bc}	6 ± 3^{efg}
TPS-50T	429 ± 91^{a}	13.2 ± 1.9^{a}	2.8 ± 0.8^{g}

[a] Different letters in each column indicate significant differences ($p < 0.05$) among formulations.

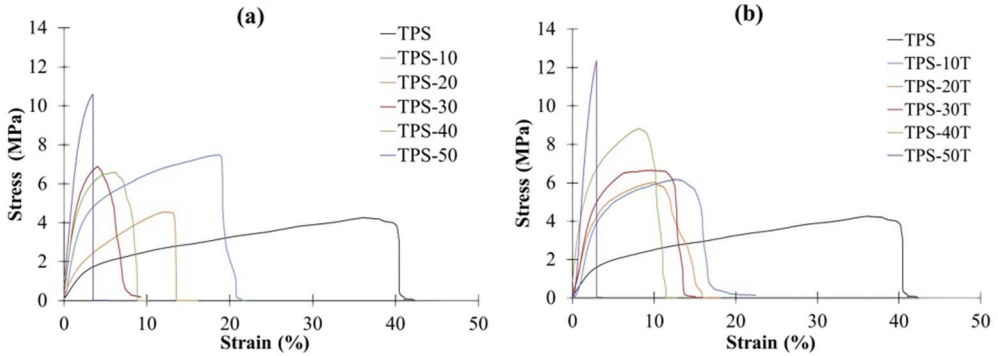

Fig. 6 Typical stress–strain curves for control (TPS) and composite films incorporating different concentrations of untreated (a) and thermally treated (b) potato peel.

due to their oxygen scavenger activity, as previously described by other authors for antioxidant phenolic acids.[73] Among all the samples, the TPS-50 films exhibited the best water barrier properties, while TPS-50T films maintained the good oxygen performance of the pure starch-based films ($p > 0.05$). Therefore, the use of potato peel at high concentrations as a filler in starch-based films represents an adequate strategy to improve their water barrier capacity, while preserving the oxygen permeability values. The preservation of oxygen barrier capacity, together with protection against UV light and antioxidant activity, could contribute to preserving the organoleptic and nutritional quality of oxidation sensitive food, as commented on before. On the other hand, appropriate water vapour barrier properties are a key characteristic for foods that are sensitive to water gain or loss, such as dry or crunchy products.

3.2.7. Mechanical properties. Table 6 summarizes the mechanical parameters obtained for the control and the potato peel-loaded films, and Fig. 6 shows the typical stress–strain curves for the different formulations.

The mechanical values for the TPS films were similar to those reported by other authors working with potato thermoplastic starch.[17,74,75] Nevertheless, Guo *et al.*[76] reported lower tensile strength (1.5 MPa) and a higher percentage of elongation values (80%) for plasticised potato starch films obtained by casting. In the casting process, the slow drying process allows polymer chains to gradually unfold and orient themselves, leading to a more ordered three-dimensional network, which enhances chain slippage during stretching and promotes film extensibility.[77]

As can be observed in Fig. 6, the addition of potato peel (PoP and PoP-T) provoked significant changes ($p < 0.05$) in the mechanical performance of the starch films, increasing the resistance to break (TS values) and the stiffness (EM) while decreasing their stretchability (ε). These effects were more noticeable as the potato peel content increased, leading to a significant reinforcement effect ($p < 0.05$). The greatest reinforcing effect was observed in films with 40–50% potato peel (93–257% higher TS and 238–448% higher EM), although these films became less extensible. The film's elastic modulus is mainly related to the intrinsic properties of the components, the fibre Young's modulus being much higher (from 5.5 up to 150 GPa depending on the type of fibre)[78] compared to TPS, in agreement with its highly crystalline cellulose content. The contribution of the crystallinity of cellulose to increasing the rigidity, elastic modulus and tensile strength and reducing the film's water vapor permeability has been previously observed by other authors.[79]

The present findings are promising, as the direct addition of lignocellulosic residues into polymeric matrices often leads to deterioration of mechanical properties, as previously observed with other lignocellulose waste such as banana fibres, tiger nut residue, or brewer's spent grain in TPS films.[22,58,80] Typically, such reinforcement behaviour is obtained using bleached micro- or nanocellulose fibres, owing to the high aspect ratio of the particles and crystallinity and to the starch-cellulose bonding ability.[8,28] For example, Collazo-Bligadri *et al.*[41] reported that the incorporation of 10% cellulose fibres from coffee husks and rice husks led to increases of 30% and 46% in TS and 260% and 231% in EM and a 75% reduction in elasticity, compared to the thermoplastic starch control film. Thus, the incorporation of potato peel into the starch film may have achieved a reinforcement effect comparable to that of cellulose, without the need to isolate and purify the fibrous component from the residue prior to film processing. The presence of both starch and a substantial proportion of cellulose-rich fibre in the potato peel residue ($57 \pm 2\%$, db)[81] likely promoted high compatibility and strong interfacial adhesion between the blended components.

On the other hand, the reduction in the film extensibility is associated with the limited polymer chain mobility caused by the fibres and the microstructural discontinuities within the polymer matrix. Likewise, a progressive reduction in the glycerol mass fraction in the films with increasing percentage of potato peel may imply a less plasticised and more rigid matrix.[82] In general, the thermal pre-treatment of the residue did not significantly affect the mechanical parameters of the obtained films ($p > 0.05$).

4. Conclusions

The results obtained revealed the potential of potato peel to obtain economically more affordable starch-based films by incorporating low-starch potato peel up to 50 g/100 g starch (or

27 g/100 g film) into the composite blend. Among all the tested formulations, the composite films incorporating the highest potato peel ratio exhibited significantly improved rigidity, mechanical resistance and water vapour barrier properties as compared to TPS films, although they were less stretchable. The phenolic content of the residue, thermally pre-treated or not, endowed the films with strong light barrier capacity and certain antioxidant potential that could be of interest to limit oxidative processes in oxidation-sensitive foods. The thermal pre-treatment of the potato peel favoured the integration of the potato peel components into the polymeric matrix and slightly affected the properties of the films, promoting higher antioxidant activity and enhanced light blocking capacity due to its greater phenolic content. Thus, these low-cost composites could contribute to delaying oxidative reactions in sensitive food products, while contributing to the transition towards a more sustainable circular economy. Future work will focus on real-food application studies to effectively assess the potential of these films to preserve foodstuffs sensitive to oxidative reactions and on optimizing formulation parameters and assessing long-term stability and industrial applicability of these films.

Author contributions

Chelo González-Martínez and Maria Eugenia Martín-Esparza – conceptualization, Chelo González-Martínez and Maria Eugenia Martín-Esparza – methodology, Marta Santos-Iparraguirre – formal analysis, Chelo González-Martínez, Maria Eugenia Martín-Esparza and Marta Santos-Iparraguirre – data curation, Chelo González-Martínez and Marta Santos-Iparraguirre – writing—original draft, Chelo González-Martínez and Maria Eugenia Martín-Esparza – writing—review and editing, Chelo González-Martínez and Amparo Chiralt – supervision, and Chelo González-Martínez and Amparo Chiralt – funding acquisition. All authors have read and agreed to the published version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

Data availability

Research data will be available in our Institutional Repository RiuNeT: https://riunet.upv.es/handle/10251/231818.

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