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ORIGINAL PAPER

Development of Novel Pectin-Based Films from Coffee Waste: Mucilage and Pulp

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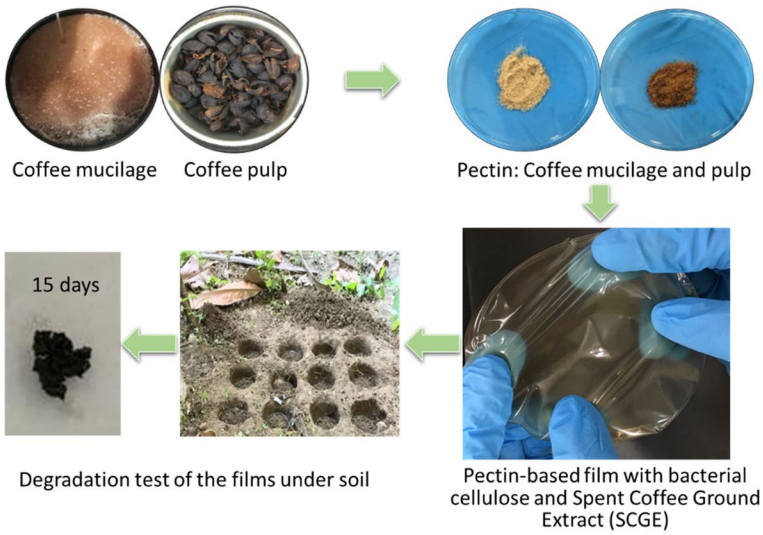
Abstract

Coffee is the world's second-most preferred beverage and a highly commercialized agri-food product. During its production, a significant volume of post-harvest and processing waste is produced. In this study, post-harvest coffee waste was processed to develop a novel pectin-based film reinforced with bacterial cellulose obtained by the fermentation from coffee mucilage and coffee oil extract of spent coffee grounds (SCG). Pectin from coffee waste (coffee mucilage and coffee pulp) was isolated by citric acid hydrolysis. The addition of bacterial cellulose and coffee oil (SCGE) to the formulation of the pectin-based films improved the physicomechanical properties, flexibility, and functionality (tensile strength 3.4 MPa, elongation at break 24.5%, resistance of water solubility 40%, and spontaneous degradation during 16 days in soil under uncontrolled conditions). The reinforced films based on pectin from coffee pulp showed better properties when compared to pectin-based films from coffee mucilage (tensile strength of 2.2 MPa and water solubility of 58.5%); this effect is attributed to the morphological characteristics and the presence of pores in the polymeric matrix of pectin-based films from coffee mucilage. According to the results, these composite films hold great promise as substitutes, for example, for single-use plastics while giving value to underexplored coffee residues.

Graphical Abstract



Keywords Cellulose · Coffee residues · Films · Lipophilic extracts · Pectin · Spent coffee grounds

Extended author information available on the last page of the article

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Introduction

In 2022, the National Federation of Coffee Growers [1] estimated that 11.1 million 60 kg bags of green coffee were produced in Colombia. During post-harvest operations, coffee cherries undergo a transformation process in which significant amounts of waste (such as mucilage, pulp, parchment, and silver skin) are generated. The amount of mucilage and pulp resulting from coffee processing is estimated to be 768 kg ha/year and 2.258 kg ha/year, respectively [2]. Green or unripe cherries are separated to obtain coffee parchment by wet processing. After that, the exocarp and mesocarp are removed from the fruit by mechanical pulping (husks and pulp). On average, the pulp accounts for 29% of the total fruit dry weight [3]. Coffee pulp is a by-product containing tannins (1.80–8.56%), reducing sugars (12.4%), chlorogenic acids (2.6%), and total pectic substances (6.5%), as well as proteins and minerals [4]. Because it contains bioactive and antioxidant compounds (such as flavonoids, proanthocyanidins, chlorogenic acid, caffeine, and fatty acids) it has drawn particular interest [5, 6]. However, despite its high potential, few bioactive compound extraction processes are used in coffee production areas.

After pulping, which is characterized as a sieve method to remove unwanted beans or beans that were not completely pulped, coffee mucilage is obtained. Following pulping, the beans are separated from the pulp by mechanical treatment or spontaneous fermentation. The mucilage, a layer that is roughly 2 mm thick and is strongly adhered to the endocarp of the cherry due to the presence of a gelling agent, represents 14.8% of the total fresh fruit weight. It is important because it contains sugars (6.2–7.4%), lipids (0.12%), proteins (0.9%), carbohydrates (7.5–9.8%), ashes (0.43%), and pectic substances (0.6–2.0%) [4]. After the mucilage is processed, a final washing step is performed to eliminate any remaining mucilage attached to the grain. This step requires a large amount of water, approximately 40 L/kg. Seen in this light, coffee mucilage, which has a high BOD (approx. 30,000 mg/L), is accountable for 26% of effluent pollution. Likewise, processing steps such as threshing—in which the parchment or endocarp is removed with the partial detachment of the silver skin—roasting, grinding, and beverage preparation leave the spent coffee ground as an additional residue. Due to their production volume and chemical composition, coffee residues have been proposed as raw materials for agricultural purposes (e.g., soil amendment and animal feed) and as substrates for fermentation processes. In addition, they can be used as raw materials for the thermochemical conversion process to produce biochar due to their lignocellulosic nature.

Although there are many options for waste management, pectin extraction is one of the most popular because pectin is a commercial compound used in both the food and non-food industries due to its gelling and thickening properties [7]. Pectins are polysaccharides present in the primary cell walls, and they structurally contain 1,4 α-D-galactosiluronic acid (GalpA) residues. They are classified according to their Degree of Esterification (DE), which is the percentage of carboxyl groups esterified with methanol. Pectins with a DE > 50% are known as High-Methoxyl (HM) pectins and those with a DE < 50% are known as Low-Methoxyl (LM) pectins. Pectin can be extracted by ultrasound-assisted, enzyme-assisted, microwave-assisted, pulsed electric field, high-pressure processing, and conventional chemical (acid hydrolysis) methods using sulfuric acid, nitric acid, or organic acids like citric acid under controlled conditions (temperature, time, and pH) [8]. The yields, the DE, the environmental impact, the costs, and the requirements for scaling-up all influence the extraction process choice. Pectin is mainly used in the food sector due to its properties as a gelling, texturizing, emulsifying, and stabilizing agent. As an additive, it has been employed as a fat or sugar substitute in food matrices, given its associated functionality, as it contains polar and non-polar regions. Its application, however, is conditioned to its degree of methoxylation and particle size.

The use of pectin as a potential substitute for polymers derived from non-renewable sources in the creation of biodegradable films is currently expected to meet high expectations. For instance, single-use plastics made from synthetic polymers have been replaced with pectins extracted from renewable sources such as mango, cantaloupe, and pineapple peels. Since pectin-based films have a hydrophilic character, i.e., they exhibit low stability when they come into contact with a liquid phase, the use of a plasticizing agent is required. In addition, their functionality depends on their mechanical and barrier properties; therefore, to increase their physicomechanical resistance, it is necessary to incorporate reinforcing agents as polysaccharides, such as cellulose [9] and lipophilic compounds [10].

Considering this, together with the inadequate final disposal of agro-industrial and plastic waste, it is imperative to explore novel and workable solutions to these problems. From an eco-friendly perspective and using by-products obtained from the coffee industry, this study aims to develop biobased films made from pectin extracted from coffee mucilage and pulp and reinforced with bacterial cellulose and the lipophilic extract of coffee grounds. The structural and physicomechanical properties of the developed films will, indeed, determine their final application in the packaging industry.

Materials and Methods

Preparation of Raw Materials

Arabica coffee mucilage and pulp were both collected from a coffee farm in Jardín, Antioquia (Colombia). The coffee mucilage was extracted mechanically during the processing of the fresh coffee fruit. Once collected, it was filtered to remove particulate matter. Subsequently, it was dried at 80 °C for 5 h in a forced convection oven (Memmert) to concentrate solids. The coffee pulp was dried at 40 °C for 24 h and then crushed in a coffee grinder (KRUPS GX4100) and sieved through an 80-mesh screen.

Pectin Extraction

Pectin was extracted using the methodology proposed by Valdespino et al. [11] with some modifications. The coffee mucilage and pulp were suspended in acidulated water (1 M citric acid) at pH 1.8 in a 2:1 v/v and 7 mL/g under the following operating conditions: 80 °C and 45 min for the mucilage and 90 °C and 50 min for the pulp (both at 500 rpm). Then, the solution was centrifuged (HERMLE Z 366 K, Germany) for 15 min at 25 °C and 7,500 rpm to remove solids. The supernatant (1:1 v/v) was mixed with 96% ethyl alcohol at 4 °C in order to extract pectin. After mixing and resting for 15 min, the mixture was centrifuged at 4 °C and 7,500 rpm for 10 min. The obtained pectin was mixed with 96% ethyl alcohol in a 1:1 w/w ratio and centrifuged at 25 °C and 7,500 rpm for 10 min to remove impurities. Finally, the pectin was dried at 30 °C for 24 h, and, after grinding, it was sieved through an 80-mesh screen. The operating parameters (hydrolysis temperature and time) were evaluated using a 2^2 experimental design (Statgraphics Centurion XVI.I - Stat Point, Inc., USA), with a response variable corresponding to pectin yield, which was calculated using Eq. (1).

$$\text{Yield}(\%) = \frac{\text{Extracted pectin(g)}}{\text{Mucilage/pulp sample(g)}} \times 100 \tag{1}$$

Equivalent Weight, Free Acidity, and Methoxyl Content

Dry pectin (0.1 g) was placed in a 250 mL flask, and then 1 mL of ethanol was added to it. Following that, 0.2 g of NaCl, 20 mL of distilled water, and 3 drops of phenol red were added under constant stirring until the pectin was completely dissolved. The mixture was titrated with 0.1 N of NaOH until a color change was observed (pH 7.5) [12]. The equivalent weight and free acidity were calculated using Eqs. (2) and (3), respectively.

$$\text{Equivalent weight (mg/meq)} = \frac{\text{Weight of pectin (mg)}}{\text{mL NaOH} \times \text{concentration of NaOH(N)}} \tag{2}$$

$$\text{Free acidity (meq/g)} = \frac{\text{mL NaOH} \times \text{concentration of NaOH(N)}}{\text{weight of pectin(g)}} \tag{3}$$

The methoxyl content was determined using the titrated solution employed in the equivalent weight test. Five mL of NaOH were added to 0.25 N, and after a 30-min rest at room temperature, 5 mL of HCl were added to 0.25 N. Finally, the solution was titrated with 0.1 N of NaOH until it turned magenta. The methoxyl content was determined using Eq. (4).

$$\text{Methoxyl content (\%)} = \frac{\text{concentration of NaOH(N)} \times \text{volume of NaOH(mL)} \times 3.1}{\text{weight of pectin(g)}} \tag{4}$$

Esterification Degree Analysis by Fourier-Transform Infrared Spectroscopy

Fourier-Transform Infrared (FTIR) spectra were recorded using the potassium bromide disk method on a Shimadzu IRTracer-100 equipment (Perkin Elmer, USA) at a resolution of 4/cm and throughout a spectral range of 500 to 4000/cm in transmittance mode. As suggested by Muhammad [13], the DE of pectin was determined based on the ratio of the peak area of esterified (AGCE) and non-esterified carboxyl groups (AGCNE), as shown in Eq. (5).

$$\text{DE}(\%) = \frac{\text{AGCE}}{\text{AGCE} + \text{AGCNE}} \times 100 \tag{5}$$

Pectin Analysis by High-Performance Liquid Chromatography

100 mg of pectin extracted from coffee mucilage and pulp was hydrolyzed in a 1:30 (w/v) ratio with 80 U of pectinase at pH 4 (0.1 M citrate buffer) and 30 °C while being continuously stirred at 200 rpm for 2 h using a UNIMAX 1010 stirrer (Heidolph). The hydrolyzed samples were kept for 24 h at 4 °C before being filtered through a 0.25-µm syringe filter and taken to vials with a sample-to-type I water ratio of 1:10 for the analysis of galacturonic acid, arabinose, glucose, and xylose. Such analysis was performed using a high-performance liquid chromatograph (Shimadzu CBM-20 A W) equipped with a Refractive Index Detector (RID) using an Aminex HPX-87 C column (300 mm x 7.8 mm x

9 µm; Bio-Rad) and type I water as the mobile phase at a rate of 0.6 mL/min in isocratic mode. The column operated at a temperature of 65 °C, an injection volume of 20 µL, and a total run time of 15 min. The target components were quantified using calibration curves with D (+) galacturonic acid (Sigma-Aldrich 73960) and a sugar kit (Sigma-Aldrich).

Formulation of Films

Preliminary tests revealed that films without additives demonstrated poor performance, characterized by brittleness and cracking. Consequently, incorporating additives such as glycerol and Tween 20 was deemed essential. Glycerol, a widely used plasticizer, enhances film flexibility by dispersing within polymer chains, reducing intermolecular attractions. Following methodologies from Sood [14] and Gaona-Sánchez [15], with modifications, initial tests using 3% wt. glycerol, commercial pectin, and experimental pectins produced unsatisfactory malleability and flexibility. Tween 20, a safe, non-toxic surfactant, was evaluated at two concentrations (3% wt. and 10% wt.) while maintaining a constant oil concentration (SCGE) of 5% wt. At 3% wt., the oil exuded, leaving an oily residue on the film surface. However, at 10% wt., the oil was effectively retained within the polymer matrix, yielding a satisfactory film. In addition, mechanical and solubility tests were conducted in films without additives such as the coffee grounds extract, bacterial cellulose, and Tween 20, used for oil emulsification and solubilization. The results showed that the pectin-based films degraded upon contact with water. The films without additives exhibited poor performance in mechanical tensile testing (Tensile strength: 1.6 ± 0.4 MPa, elongation at break: $6.5 \pm 0.4\%$) and high-water solubility ($91.7 \pm 2.8\%$).

Building on these findings, pectin-based films from coffee mucilage (M01) and coffee pulp (P01) were prepared with a base composition of 2 g of pectin per 100 mL of distilled water and using glycerol as a plasticizing agent and Tween 20 as an emulsifying agent (22 and 10%, respectively, based on pectin content). As observed in Table 1, all the formulations, except for M01 and P01, contained reinforcing agents, namely Spent Coffee Ground Extract (SCGE), obtained according to the methodology by Arango et al. [5] and bacterial cellulose obtained in a previous study by Rendon-Munoz [16], (5 and 35%, respectively, based on pectin content).

Following homogenization in an Ultra Turrax (IKA T25 DS1) for the cellulose formulation, the pectin/water and water/pectin/cellulose components were mixed for 45 min in the experimental process. Then, depending on formulation, glycerol/Tween 20 was added at 40 °C while being constantly stirred. Finally, SCGE was incorporated. After 20 min of stirring, the suspension was homogenized for 3 min and degassed at 40 °C for 15 min. The films (20 g of solution) were dried in a forced convection oven at 30 ± 1 °C for 24 h. The obtained films were preconditioned in a climate chamber at 50% RH and 25 °C for 48 h.

Table 1 Formulation of pectin-based films from coffee mucilage and coffee pulp

Code*	Bacterial cellulose (% wt.)	SCGE** (% wt.)
M01	0	0
M02	0	5
M03	35	0
M04	35	5
P01	0	0
P02	0	5
P03	35	0
P04	35	5

*M0x and P0x: pectin-based films from mucilage and pulp coffee, respectively

**SCGE: oil extract of spent coffee ground

Structural, Morphological, and Physicomechanical Characterization of the Films

For FTIR analysis, the Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) method was used. A Thermogravimetric Analysis (TGA) was performed on the extracted pectin and the prepared films using TA Instruments TGA Q5000 equipment with a heating ramp of 10 °C/min from 30 to 600 °C in a nitrogen atmosphere. Dataset was plotted as both, weight (%) and derivate of thermogravimetric (%/°C) curve as a function of temperature.

Pectin and the films were analysed by Field Emission Scanning Electron Microscopy (FE- SEM, JEOL 7100 F) to characterize the samples morphologically. For the pectin and the films, the acceleration voltages were set to 15 kV and 10 kV, respectively.

The thickness of the films was measured using a micrometer, and the obtained value was an average of eight points. The mechanical properties of the films were determined as per the ASTM D638 standard using a universal SHIMADZU machine (AGX-V). The tests were done using samples with a size of 10 mm x 50 mm. The crosshead speed was 1 mm/min, with an initial grip spacing of 50 mm. Finally, the Elongation at Break (EB) and Tensile Strength (TS) of the films were calculated using Eqs. (6) and (7), respectively.

$$EB(\%) = \frac{L - L_1}{L_1} \times 100 \tag{6}$$

$$TS\ (MPa) = \frac{\text{Ultimate Tensile Strength(N)}}{\text{Thickness(mm)} \times \text{width(mm)}} \tag{7}$$

Water Solubility and Degradation of the Films

Wu et al. [17] method was used to determine water solubility. The samples with a size of 20 mm x 20 mm were dried at 105 °C. After initial weight recording (w_o), they were suspended in 30 mL of distilled water under constant stirring at 50 rpm and 25 °C for 24 h. Subsequently, they were filtered, and the residual samples were dried at 105 °C for 24 h for final weight recording (w_1). Equation (8) was used to calculate the film's water solubility (w_S).

$$Ws(\%) = \frac{W_0 - W_1}{W_0} \times 100 \quad (8)$$

The degradation of the films in soil under uncontrolled conditions was assessed based on the methodology presented by Quader et al. [18], with some modifications. The samples with a size of 3 cm x 3 cm and an initial perimeter of 81 cm were buried in soil at a depth of 8 cm. Every 24 h, they were removed from the soil, carefully cleaned, dried in a forced convection oven at 40 °C for 20 min, and then placed in a desiccator for 20 min. Their initial and final weights were recorded in order to calculate the degradation rate using Eq. (9). Their perimeter was also recorded every 24 h. The samples with a size of 2 cm x 2 cm and an initial perimeter of 16 cm were exposed to an indoor environment under uncontrolled conditions. The weight loss and perimeter were monitored according to the exposure time (every 4 days).

$$\text{Degradation}(\%) = \frac{\text{Initial weight} - \text{final weight}}{\text{Initial weight}} \times 100 \quad (9)$$

Results and Discussions

Extraction and Characterization of Pectin from Coffee Mucilage and Pulp

The extraction of pectin from coffee wastes is conditioned by operating variables such as temperature and hydrolysis time. According to the results, the TM4 and TP4 treatments presented the best extraction parameters, with pectin yields of 10.98% and 6.14%, respectively (Table 2). Time and the time-temperature interaction were found to significantly influence the extraction of pectin from coffee mucilage ($P < 0.05$). Concerning the extraction of pectin from coffee pulp, temperature was found to have a significant effect ($P < 0.05$) and contributed positively to the temperature–time interaction, with time being the less relevant factor. This difference in terms of extraction parameters could be associated with the composition and structure of coffee mucilage and pulp. In fact, pectin is more readily available in coffee mucilage, while it is immersed in the lignocellulosic matrix in coffee pulp. Thus, in the latter, extraction parameters, such as temperature, need to be more severe. Yu et al. [19] reported a significant impact of temperature on pectin extraction, which, according to them, is linked to the disruption of plant cells and the rapid separation and dissolution of pectin. The best conditions for the extraction of pectin from coffee mucilage are 80 °C and 45 min, and those for the extraction of pectin from coffee pulp are 90 °C and shorter times (30 min). The pectin extraction yields obtained in this study are close to those obtained by Serrat-Díaz [20], who reported a 4.99% pectin yield from Robusta coffee pulp. Regarding other plant matrices, important pectin extraction yields have also been reported, including an 18.90% yield from cider marc [19] and a 6.05% yield from fig skin using a temperature of 90 °C and a hydrolysis time of 60 min [21]. Clearly, pectin extraction and yield depend on the composition and structural characteristics of the source.

Table 2 Yield of pectin extractions from Arabica coffee mucilage and pulp

Treatment*	T (°C)	Time (min)	Yield** (%)
TM1	70	30	8.65 ± 0.19
TM2	80	30	7.75 ± 1.28
TM3	70	45	8.58 ± 1.36
TM4	80	45	10.98 ± 0.28
TP1	90	30	5.64 ± 0.66
TP2	70	50	3.24 ± 0.43
TP3	70	30	3.82 ± 0.17
TP4	90	50	6.14 ± 0.18

*TMx and TPx: Treatments for pectin extraction from mucilage and pulp coffee, respectively

**Dry matter basis (d.m.b)

Table 3 shows the equivalent weight, free acidity, methoxyl content, and DE of the pectins extracted from coffee mucilage and pulp. After comparing these values with those of commercial pectin, the latter showed a greater equivalent weight, possibly due to the numerous hydrolytic reactions of the polygalacturonase during the extraction process [11]. The acidity of the pectin from coffee pulp was 1.54 meq/g, which is higher when compared to that of the other pectins. This may be attributed to the presence of galacturonic acid in the polymer structure. In this regard, the pectin from coffee mucilage presented an acidity of 0.89 meq/g, which is similar to that of the pectin extracted from the sugar mango peel (0.86 meq/g) [22].

Pectins can be classified as High Methoxyl (HM) or Low Methoxyl (LM) pectins depending on their methoxyl content and galacturonic acid content. HM pectins must have at least 6.70% of methoxyl content and more than 50% of

Table 3 Chemical characterization of the pectins under analysis

	Pectin from coffee mucilage	Pectin from coffee pulp	Commercial pectin
Equivalent weight (mg/meq)	1127.95 ± 3.24	647.57 ± 4.46	1825.29 ± 5.70
Free acidity (meq/g)	0.89 ± 0.00	1.54 ± 0.01	0.55 ± 0.00
Methoxyl content (%)	7.05 ± 0.27	4.85 ± 0.25	7.01 ± 0.22
Degree of esterification (%)	81.40 ± 2.08	72.74 ± 0.32	59.20 ± 1.18

esterified galacturonic acid content. Since the methoxyl content of the pectin from coffee mucilage was 7.05%, and its DE was 81.40%, it can be considered an HM pectin. In their study, Valdespino-León et al. [11] reported similar values: a methoxyl content of 6.21% and a DE of 84.90%. Regarding the pectin from coffee pulp, its DE (72.74%) was higher than that of the commercial pectin, and its methoxyl content was below the limit to be considered an HM pectin (4.85%). Similar results have been reported for commercial apple pectin, with a DE of 80.19% and a methoxyl content of 1.78% [23].

The galacturonic acid content in the pectin from coffee pulp was 2.78 g/L (Fig. 1A), which was higher than that in the pectin from coffee mucilage (2.09 g/L) (Fig. 1B). Due to the high prevalence of galacturonic acid units in the polymer structure, a predominant structure of homogalacturonan was observed [11]. This is the simplest region, composed of units of the acid linked by α 1–4 bonds. In addition, the galacturonic acid content coincides with the free acidity results. The pectin from the coffee pulp was found to be the most acidic, and the presence of sugars, such as glucose (4.43 g/L), xylose (7.96 g/L), arabinose (4.00 g/L), and acetic acid (0.02 g/L) in it was confirmed. The pectin from coffee mucilage, in contrast, had a lower concentration of such sugars (2.24 g/L of glucose, 4.29 g/L of xylose, and 1.02 g/L of arabinose) and the same concentration of acetic acid. The presence of sugars and acetic acid in pectins could be associated with side chain regions in the polymer structure composed of sugar and acid residues. These regions are the most complex and are known as Rhamnogalacturonan II [24]. According to Valdespino et al. [11], the presence of glucose may be related to possible cellulose and hemicellulose residues in the coffee mucilage.

The pectin from coffee mucilage and that from coffee pulp exhibit morphological differences. The pectin from coffee mucilage has a heterogeneous surface morphology with pores, as seen in the SEM images (Fig. 2), giving it a slight appearance of being "porous" (Fig. 2A). In the case of the pectin from coffee pulp, it has a slightly

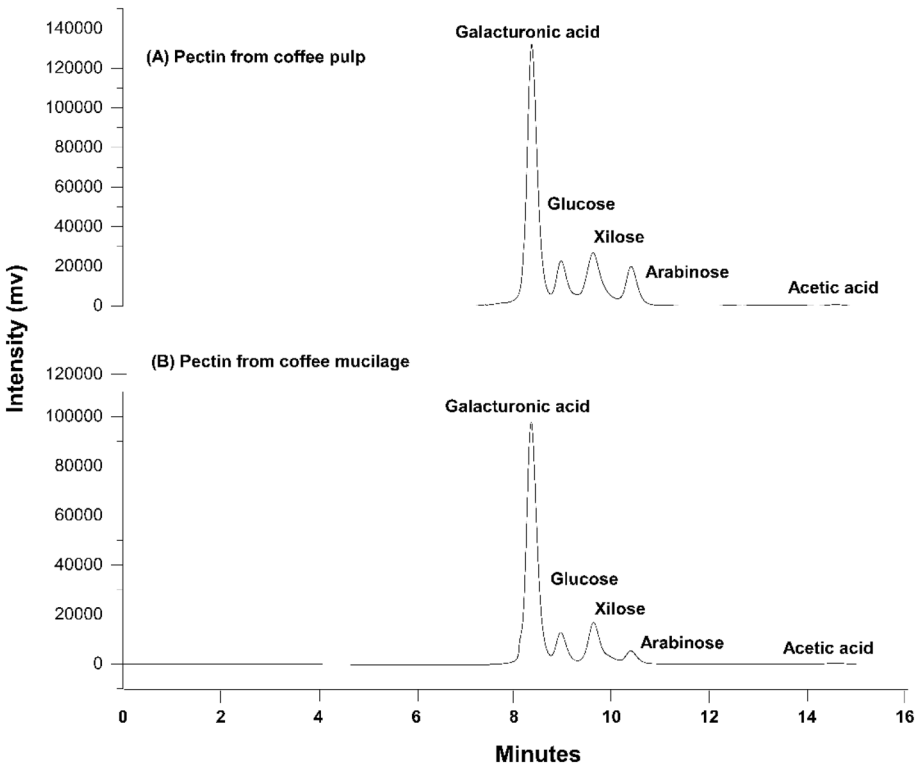

Fig. 1 HPLC chromatogram of hydrolyzed samples of pectin from coffee pulp (**A**) and pectin from coffee mucilage (**B**)

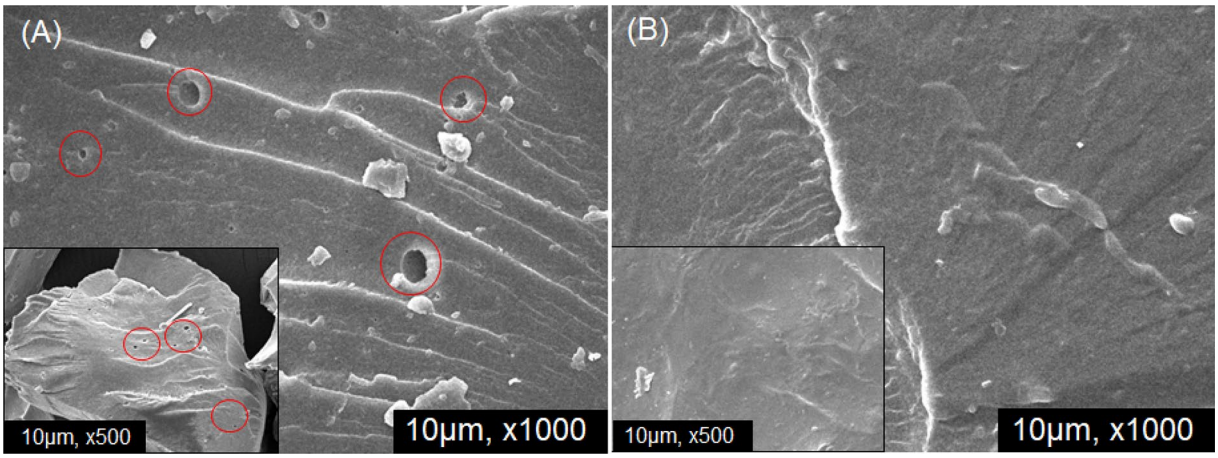

Fig. 2 Scanning Electron Microscopy (SEM) of pectin from **A** coffee mucilage and **B** coffee pulp

heterogeneous surface with no pores (Fig. 2B). These differences in morphology are associated with the extraction and drying conditions. For instance, as indicated by Liew et al. [25], abrupt temperature changes during extraction can have a direct impact on surface morphology. Likewise, Sengar et al. [26] reported significant changes in the morphology of pectin extracted from tomato residues when using different extraction techniques. Hence, the morphological differences between both pectins could be linked to extraction conditions such as temperature and extraction time, as well as to the chemical and structural composition of the raw material from which they were extracted.

The FTIR spectra (Fig. 3) showed a wide absorption band between 3,600 and 2,500/cm (3,305/cm), which corresponds to the stretching of the hydroxyl group [27] and is related to the vibrations of the inter- and intramolecular hydrogen bonds of the galacturonic acid (GalA) of the polymer [28]. The peaks at 2833 and 2937/cm in the adsorption band between 3000 and 2800/cm are attributed to the symmetrical and asymmetrical stretching of the C-H functional group [21]. The main functional group of pectins was present in the absorption band between 2000 and 1000/cm: the peaks at 1750, 1739, and 1629/cm correspond to the stretching vibration of the non-ionized C = O groups, and those at 1629, 1615, and 1531/cm correspond to the stretching vibration of the carboxylate ions (COO^-). The peaks of the esterified carboxyl groups (1750, 1739, and 1629/cm) were much more pronounced in the pectins from coffee pulp and coffee mucilage than in the commercial pectin, while those of the non-esterified carboxyl groups (1629, 1615, and 1531/cm) were more intense in the commercial pectin. Non-esterified carboxyl groups are considered key functional groups for pectin characterization because they provide information on chemical parameters such as the DE. The region between 1074 and 1043/cm revealed two peaks, which correspond to glycosidic bonds between the sugar units present in the polymer [29]. Such peaks were observed in the band between 1099 and 1911/cm, the band between 1011 and 915/cm, and the band between 1074 and 1011/cm in the pectin from coffee mucilage, the pectin from coffee pulp, and the commercial pectin, respectively.

The pectin from coffee mucilage and coffee pulp experienced a mass loss (10.37 and 10.83%, respectively) in the first event at a temperature below 100 °C, according to thermogravimetric characterization (Fig. 4). This behavior is associated with the release of water molecules. In their study, Balik et al. [30] reported water evaporation in pectin at temperatures between 75 and 110 °C due to its highly hydrophilic nature. In the second and third events, the pectins from coffee mucilage and coffee pulp exhibited mass losses of 8.90–35.57% (158.70–238.74 °C) and 20.34–20.25% (191.27–237.62 °C), respectively, which are attributed to the pyrolytic disintegration of pectin. Also, primary and secondary decarboxylation of the polymer was observed, as well as the possible breakage of bonds or functional groups along with the breakage of the chains [28]. Up to 300 °C, the pectin from coffee mucilage showed a reduction of 54.84% in its total weight. Between 300 and 600 °C, it experienced a mass loss of 19.02% of its initial weight, with a maximum loss at 238.74 °C. The pectin from coffee pulp, on the other hand, experienced a mass loss of 51.42% of its total weight up to 300 °C and a mass loss of 19.96% of its initial weight between 300 and 600 °C, with a maximum loss at 191.27 °C. When pectin is thermally degraded, a series of depolymerization reactions take occur, and at temperatures above 250 °C, lateral functional groups are released and the chains break, resulting in secondary degradation, according to Balik et al. [30]. Moreover, residues such as coal begin to gasify at temperatures between 600 and 800 °C.

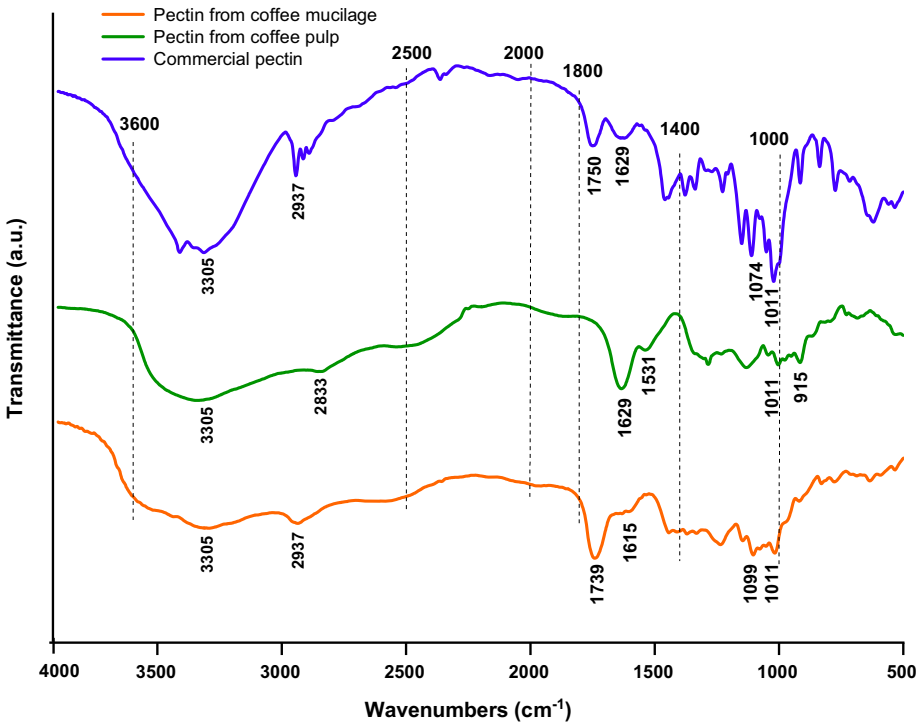

Fig. 3 FTIR spectra of the pectins under analysis

Pectin-Based Films: Structural and Physicomechanical Characterization

The pectin-based films from coffee mucilage and those based on pectin from coffee pulp were easily removed from the Petri dishes. Since the polymeric material was properly formed and the matrix showed good uniformity and flexibility, they did not become brittle. The two films were distinguished from one another by the characteristic color of pectins: they were translucent yellowish if based on pectin from coffee mucilage (Fig. 5A) and non-translucent brown if based on pectin from coffee pulp (Fig. 5B). Their final

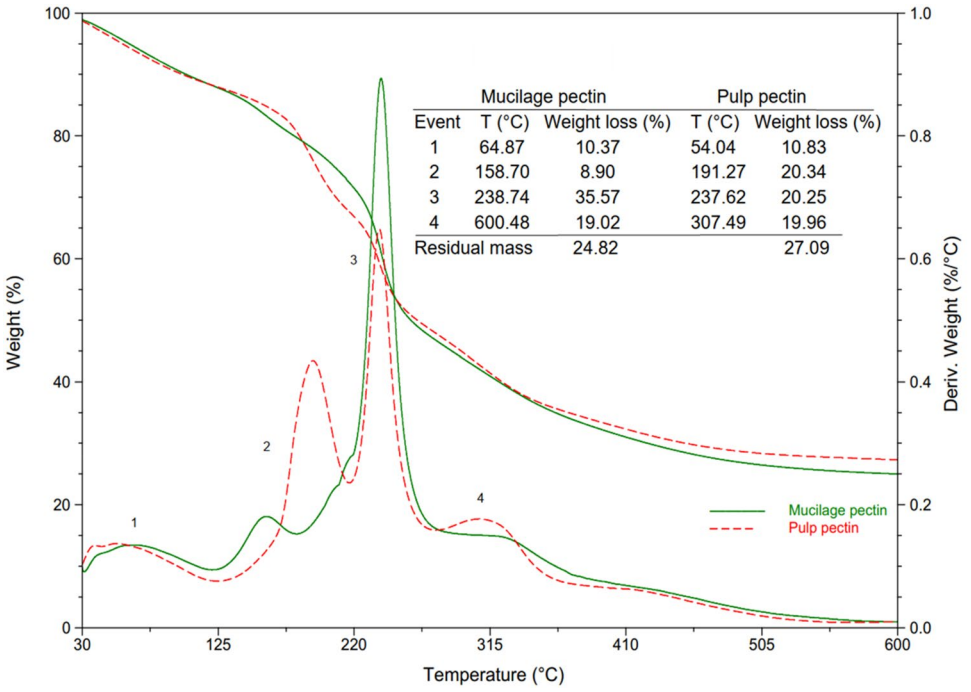

Fig. 4 Thermogravimetric Analysis (TGA) curves of the pectin from coffee mucilage and coffee pulp

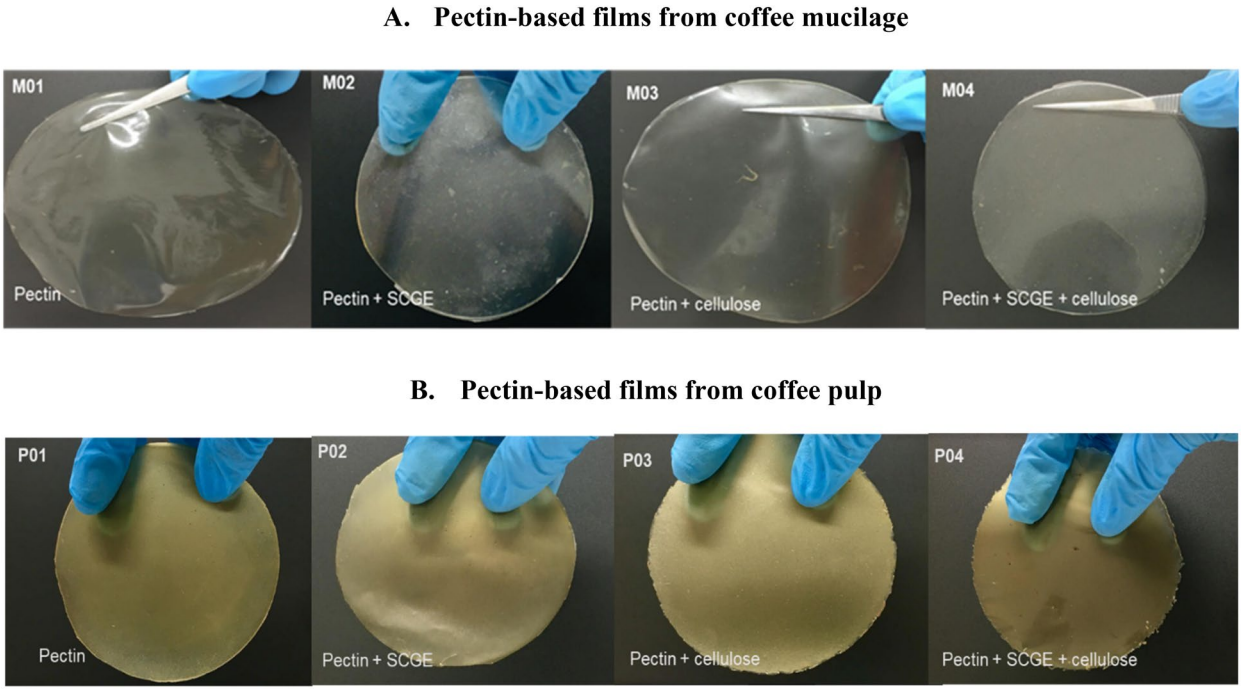

Fig. 5 Pectin-based films from coffee mucilage (**A**): pectin film (M01); pectin and SCGE film (M02); pectin and cellulose film (M03); pectin, SCGE, and cellulose film (M04). The pectin-based films from the coffee pulp (**B**) follow the same dynamics

appearance was not altered by the incorporation of reinforcing agents, namely lipophilic extract of coffee grounds (SCGE) and cellulose. Also, the addition of the lipophilic extract to the formulation left no oily feel on the surface of the films containing it, which is attributed to the use of Tween 20 as an emulsifying agent.

According to the FTIR spectra (Fig. 6), the films containing reinforcing agents (M04 and P04) showed a slight increase in the number of peaks in the region between 3800

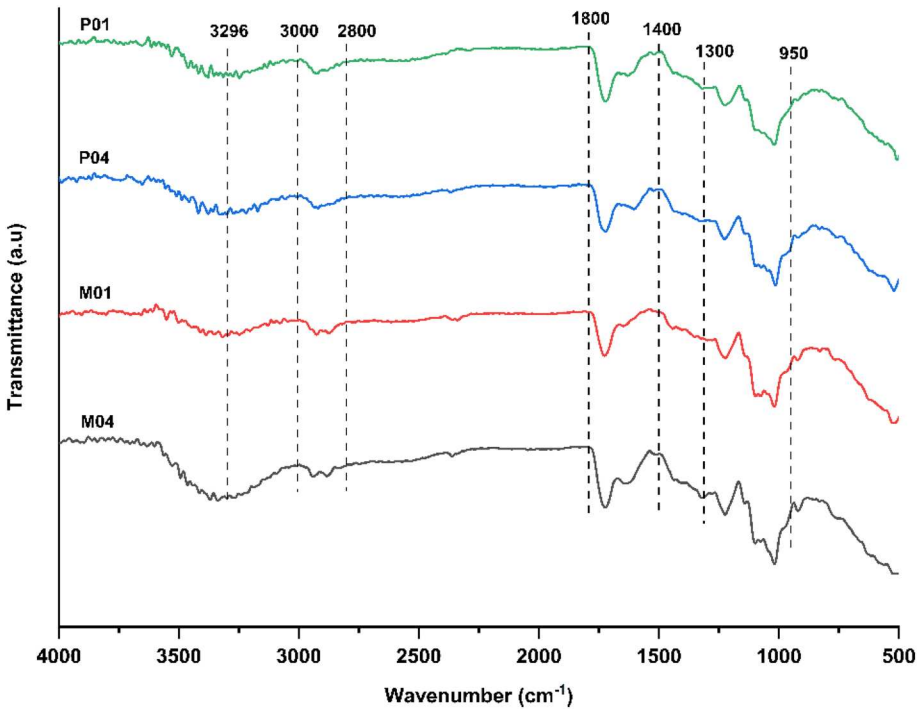

Fig. 6 FTIR spectra of the prepared films from mucilage pectin (M) and coffee pulp (P). Pectin, SCGE, and cellulose film (M04); pectin film (M01). Pectin-based films from the coffee pulp (P04) and (P01) follow the same dynamics

and 2300/cm (3296/cm), which was more evident in the M04 film. This is associated with the intermolecular effects of polyvalent −OH alcohols caused by glycerol, mainly the asymmetric stretching effects between hydroxyl groups and carbon. The band observed between 3000 and 2800/cm is attributed to the symmetric and asymmetric stretching of the C–H groups, with the M04 film showing better stretching, possibly due to the interaction of the methyl (CH_2) groups of the SCGE and cellulose. The FTIR spectra of the pectins from coffee mucilage and coffee pulp revealed a moderate intensity in the peaks observed between 1800 and 1400/cm, which correspond to the vibrations of the $-CH_2$ and −CH bonds mostly from cellulose and the vibrations of the C=O bonds in the carbonate ion (CO_3^{2-}) [31]. These intense peaks in the absorption bands between 950 and 1300/cm may be caused by the vibrations of the glycosidic bonds.

Regarding the TGA curves of the films containing pectin and reinforcing agents (Fig. 7), a first event was observed at a temperature below 100 °C, with temperature peaks at 52.69 and 69.48 °C in the M04 and P04 films, respectively. This first event is associated with the moisture loss caused by the evaporation of physically absorbed water molecules bonded by hydrogen [9], as well as to the possible volatility of the compounds present in the lipophilic extract [10]. The second event, with temperature peaks of 169.14 and 170.52 °C was attributed to the loss of moisture and other compounds such as glycerol. In the third event, weight losses of 33.55 and 31.99% were recorded for M04 and P04, respectively, and this is associated with the degradation of both short-chain and long-chain pectins [10]. The degradation of cellulose, along with the generation of C, CO, and CO_2, occurred in the fourth event, with weight losses of 12.05 and 13.01% in M04 and P04, respectively. In their studies, Ye et al. [9] and Kian et al. [32] reported thermal transitions of cellulose between 250 and 400 °C and between 200 and 380 °C, respectively. A fifth event was observed at 391.95 and 396.33 °C in M04 and P04, respectively, and it is related to a possible degradation of the branched chains of the polymer [10]. The residual weights of the M04 and P04 films were 20.59 and 23.33%, respectively.

The SEM images of the films containing both reinforcing agents (cellulose and SCGE), i.e., the M04 (Fig. 8A) and P04 (Fig. 8B) films, showed a rough and heterogeneous surface, which suggests the presence of pores in the films based on pectin from coffee mucilage (M04). This could be attributed to a low interaction between the components of the polymer matrix, which reveals a low cross-linking of the polymers, as well as to a possible crystallization of the oil, which would result in the presence of discontinuities in the polymeric matrix. When pectin from coffee pulp was used (P04), the cross-sectional micrographs showed a more compact film, which may be associated with a good compatibility of the polymers and therefore a better interaction in the hydrogen bonds, which allows a good cross-linking of polymers.

Table 4 presents the mechanical properties, Tensile Strength (TS), and Elongation at Break (EB) of all the prepared films. As observed, the inclusion of reinforcing agents significantly improved the mechanical properties of the films when compared to the control formulation. For instance, the TS of M03 (containing cellulose) was 2.4 MPa, which was higher than that of M01 (1.6 MPa). In the case of the films based on pectin from coffee pulp, the TS of P04 was 3.4 MPa, which was higher than that of the control

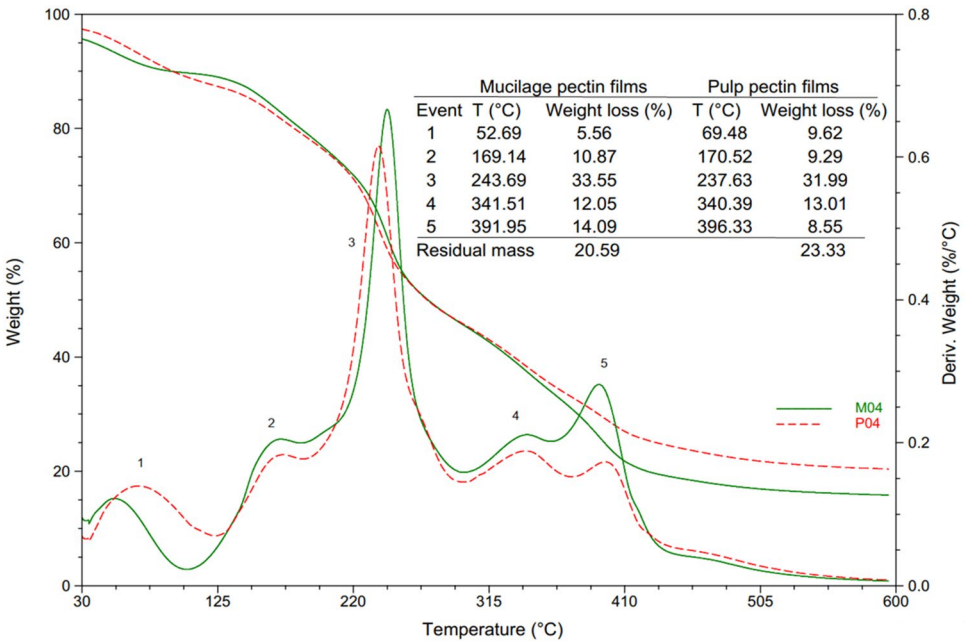

Fig. 7 Thermogravimetric Analysis (TGA) curves of the pectin-based films from coffee mucilage and coffee pulp

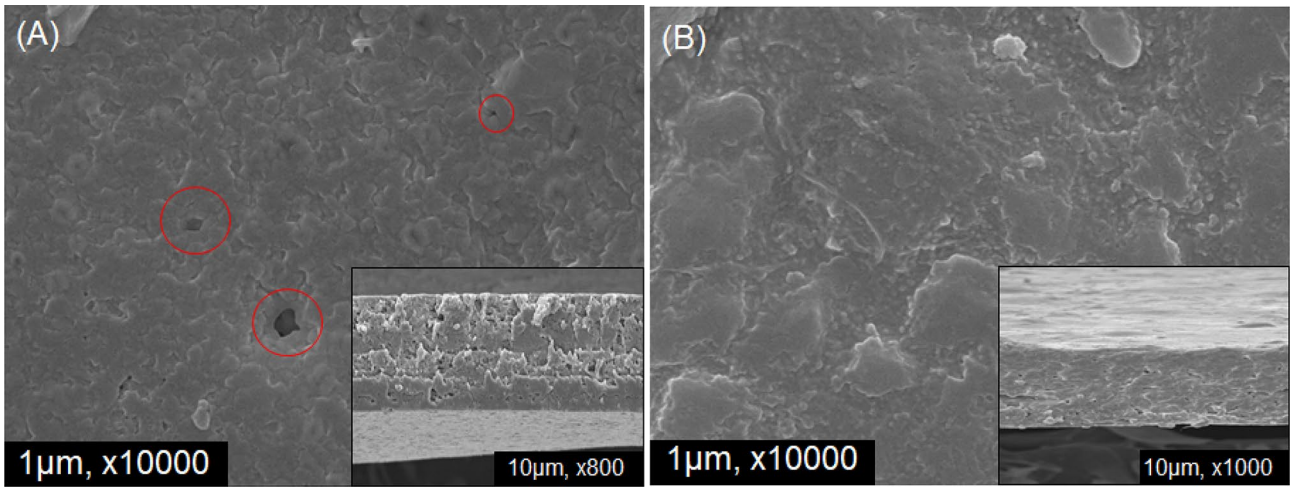

Fig. 8 Surface and cross-sectional micrographs of the pectin-based films from **A** coffee mucilage (M04) and **B** coffee pulp (P04)

Table 4 Mechanical properties and water solubility of the pectin-based films

Treatment	TS (MPa)	EB (%)	Thickness (mm)	WS (%)
M01	1.6 ± 0.4	6.5 ± 0.4	0.07 ± 0.01	91.68 ± 2.83
M02	1.6 ± 0.3	5.6 ± 1.3	0.08 ± 0.01	79.07 ± 7.89
M03	2.4 ± 0.2	6.8 ± 0.4	0.07 ± 0.01	63.52 ± 4.25
M04	2.2 ± 0.4	7.8 ± 2.5	0.09 ± 0.01	58.52 ± 1.95
P01	2.3 ± 0.6	13.0 ± 7.9	0.10 ± 0.03	74.15 ± 5.32
P02	3.0 ± 0.4	23.2 ± 9.6	0.08 ± 0.00	55.42 ± 5.40
P03	2.5 ± 0.9	24.2 ± 6.4	0.08 ± 0.00	39.52 ± 7.80
P04	3.4 ± 0.8	24.5 ± 3.9	0.07 ± 0.00	40.46 ± 5.26

TS (MPa) tensile strength, *EB* (%) elongation at break, *WS* (%) water solubility

formulation (2.3 MPa). This increase in tensile strength can be linked to the formation of a rigid and constant network of cellulose fibers connected by hydrogen bonds [33]. Also, the interaction between the pectin and cellulose molecules led to a better response in terms of resistance. Moreover, the incorporation of cellulose and the lipophilic extract of coffee grounds increased the elongation at break of the pectin-based films from coffee mucilage and those based on pectin from coffee pulp by up to 7.8 and 24.5%, respectively. Such incorporation provided the matrix with greater structural integrity, which is likely induced by a restriction of polymer chain movement caused by the strong hydrogen bonds between the fibers.

Pectin-Based Films: Water Solubility and Degradation

The incorporation of reinforcing agents had a positive effect on the water solubility of the prepared films (Table 4). During the first 4 h, the pectin-based films from coffee mucilage, with no cellulose and lipophilic extract of coffee grounds, were more easily dissolved in water when compared to the rest of the films. This result may be associated with their porous morphology and the presence of pores in their structure (Fig. 8A), which facilitates water diffusion, as well as with the possible dissociation of the ionic and hydrogen bonds. Likewise, the incorporation of cellulose and lipophilic extract contributed to the stability of the films. According to the results, the water solubility of the pectin-based films from coffee pulp was 58.5 ± 1.9% and that of the pectin-based films from coffee pulp was 39.5 ± 7.8%, which indicates a slow dissolution rate. The high solubility of the films in water after 24 h suggests that they could be used as packaging materials or edible packaging for dehydrated or fast-consuming products.

On the other hand, films based on pectin exhibit low tensile strength and high solubility, and they require reinforcing agents to improve their mechanical and barrier properties. Reinforcements such as cellulose [9] or extracts from agricultural by-products like lemon peels have been previously studied by other authors [10]. Cellulose, a polysaccharide with high tensile strength, has demonstrated effectiveness in enhancing the mechanical properties of pectin-based films. For example, El Halal et al. [34] reported improved tensile strength with cellulose incorporation, though at the cost of reduced elongation. Finally, the addition of an emulsifier to pectin films is crucial to enhance the stability of the oil-water interface, to facilitate the incorporation of bioactive compounds and to enhance functional properties.

Figure 9 shows a photographic record of the degradation under the soil, where the pectin-based films from coffee pulp presented a slower degradation rate up to 16 days of study. Concerning degradation in soil under uncontrolled conditions (Fig. 10A), the M01 film (based on pectin from

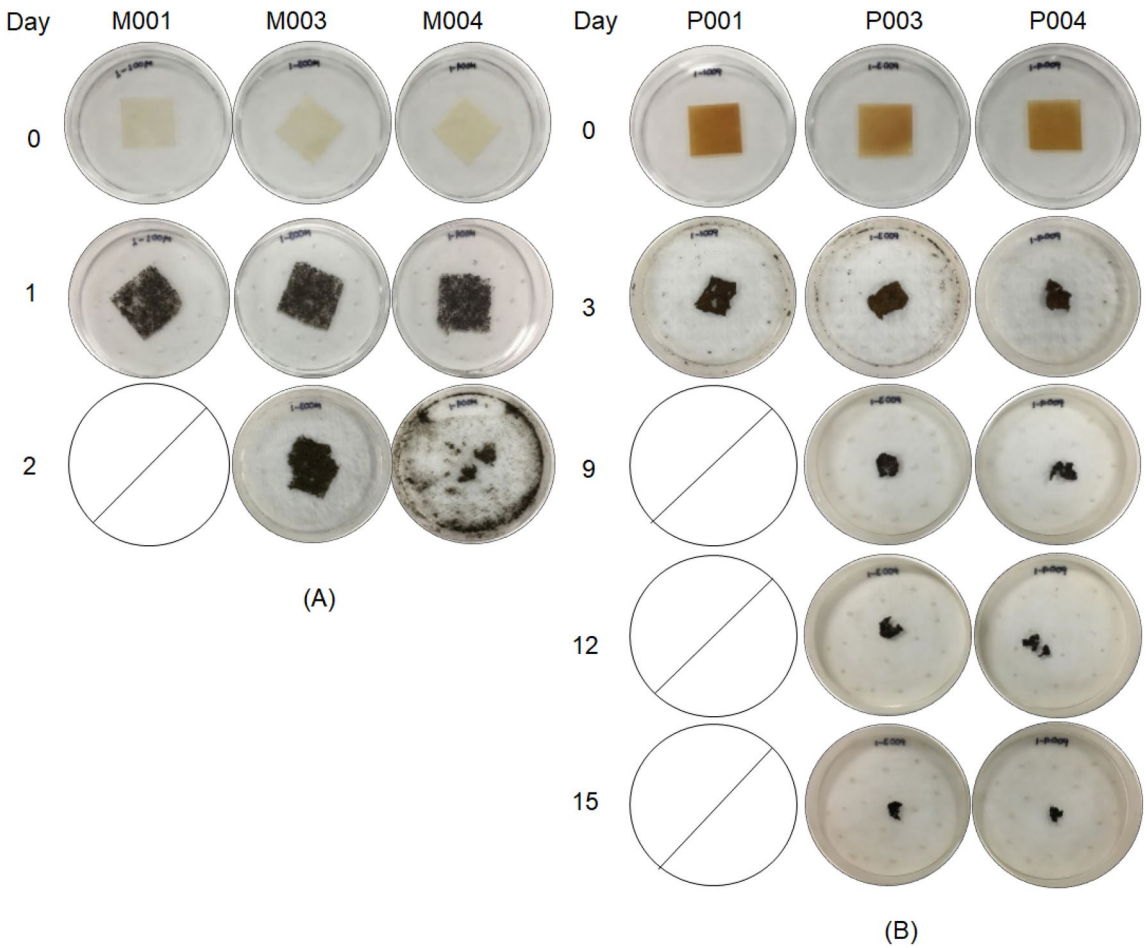

Fig. 9 Degradation test performed on the pectin-based films under soil: **A** from coffee mucilage and **B** from coffee pulp

coffee mucilage) degraded completely on day 2. The degradation rate of the M03 and M04 films, which contained reinforcing agents, was 55.3 and 88.9%, respectively. On day 4, the degradation rate of the control formulation was 93.3%, and it degraded completely on day 5. The P03 and P04 films, on the other hand, showed a degradation rate of 49.9 and 39.6%, respectively, and degraded completely on day 16. When compared to the films based on pectin from coffee mucilage, the films based on pectin from coffee pulp were more stable. This result is consistent with what has been reported in terms of water solubility. Cellulose and the lipophilic extract of coffee grounds contributed to better material stability, as they slowed down the polymer matrix disintegration caused by microbial activity. Moreover, the reinforced films exhibited a low perimeter reduction when compared to the control films (Fig. 10B). The reinforcing agents present in the polymer matrix acted as a barrier to degradation in the soil and thus slowed down disintegration.

The result of the films exposed to the environment exhibited the highest degradation of the pectin-based films from coffee mucilage. This is in line with the results of the degradation in soil test and confirms that the pectin-based films from coffee pulp are more stable. The exposure of the samples to air allowed us to observe the growth of microorganisms on their surface. On day 5, the presence of a white and cottony fungus was detected (Fig. 11A, B). According to the findings, films made from pectin, cellulose, and SCGE from coffee industry waste are a suitable new alternative with high potential in the food industry and low moisture content. The development and use of this bio-product help to mitigate the environmental problems brought on by agro-industrial waste and single-use plastics.

Conclusion

With a maximum pectin yield of 6.14 and 10.98%, respectively, coffee pulp and coffee mucilage are useful by-products from which pectin can be extracted using simple and environmentally friendly methods. Pectins from coffee mucilage and coffee pulp are presented as natural polymers with a high potential for the development of biodegradable films.

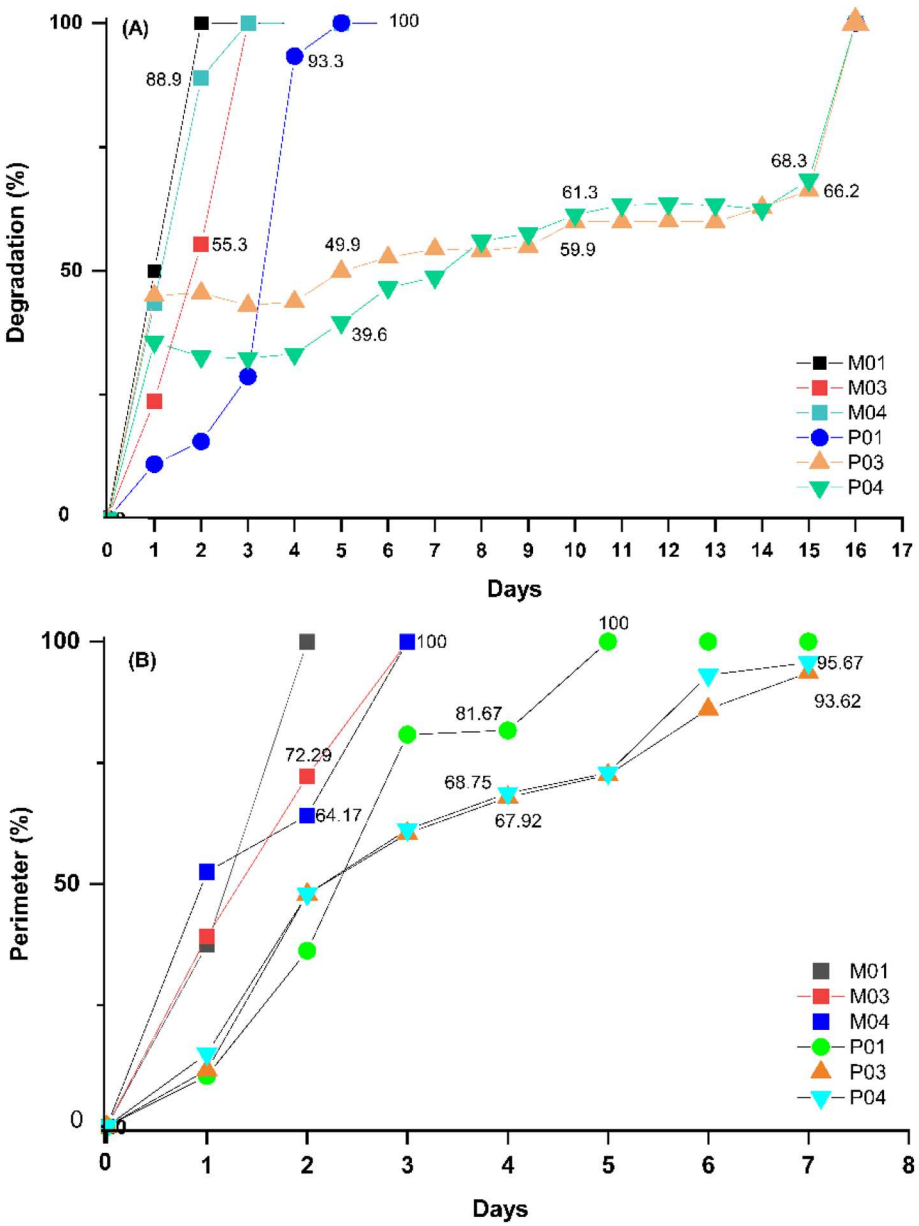

Fig. 10 Degradation (**A**)and perimeter reduction (**B**) in the pectin-based films from coffee mucilage and coffee pulp

However, due to the high solubility of pectins, reinforcing agents must be added to improve the physicomechanical and barrier properties of the films. With a tensile strength of 3.4 MPa, an elongation at break of 24.5%, and a water solubility of 40.46%, the reinforced pectin-based films from coffee pulp showed better mechanical and solubility properties when compared to the reinforced pectin-based films from coffee mucilage. This could be attributed to the morphological characteristics of the pectin and the presence of pores in the polymeric matrix of the pectin-based films from coffee mucilage.

The stability of the films in terms of biodegradation could be improved by adding reinforcing agents, resulting in exposure durations under soil of 3 and 16 days for the pectin-based films from coffee mucilage and those based on pectin from coffee pulp, respectively. Moreover, the addition of cellulose and lipophilic extract slowed down the degradation of the films when compared to the control formulations. This is due to the interaction of the functional groups of the compounds (e.g., the interaction between pectin and cellulose molecules), as well as to the antimicrobial activity of the lipophilic extract. The development of films based on pectin extracted from underexplored coffee industry by-products is a first step toward reducing environmental impact, as it creates a bio-product that can replace single-use plastics.

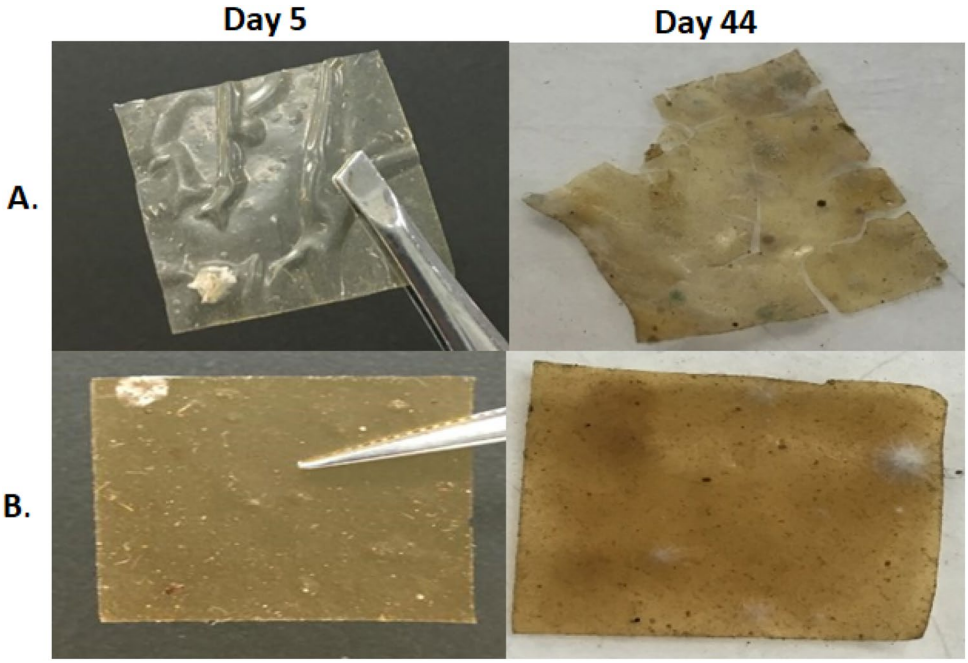

Fig. 11 Exposure of the pectin-based films **A** from coffee mucilage and **B** from coffee pulp on indoor environment under uncontrolled

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Data Availability Enquiries about data availability should be directed to the authors.

Declarations

Conflict of interest The manuscript was prepared and reviewed with the participation of the authors, who declared that there exists no conflict of interest that puts at risk the validity of the presented results.

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