

Synthesis and characterization of eco-friendly edible films based on pistachio shell

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Accepted: July 28, 2026

The transition toward biopolymer-based packaging is critical to mitigate the ecological crisis caused by petrochemical plastics. This study valorizes pistachio shells (*Pistacia vera* L.), a prominent agrowaste, as a lignocellulosic matrix to develop active edible films. Micronized shell powder was integrated into sodium alginate and carrageenan networks, plasticized with glycerol, and modified with peanut oil. Glycerol provided superior flexibility and structural integrity over sorbitol. Incorporating peanut oil yielded an exceptional moisture barrier, reducing the water vapor permeability (WVP) to 0.704–2.593 g/m²·h. Water solubility was highly controllable (48.67–86.90%), suiting diverse food moisture demands. *In vivo* 14-day trials on fresh produce confirmed that the carrageenan-based formulation (Car-S2.0-G10) substantially suppressed weight loss from ~70% to 40.51% in strawberries and from ~90% to 70.06% in apples compared to uncoated controls. These findings demonstrate a scalable, circular economy strategy for fruit preservation using agro-industrial residues.

Keywords: Edible film; pistachio shell; peanut oil; biodegradable packaging; nut waste

INTRODUCTION

Packaging is essential to extend food shelf life, maintain quality, and ensure transport safety. However, the non-biodegradable nature of petrochemical plastic packaging induces severe global ecological crises, driving intense interest in sustainable biopolymer-based edible films [1, 2]. These green technologies effectively valorize agricultural residues into high-performance coatings [3, 4]. Derived from natural polysaccharides, proteins, or lipids, edible films form physical barriers that regulate gas or moisture transfer while serving as structures for active additives [5-7]. Successful film fabrication requires precise control over core processing variables, including matrix optimization, formulation engineering, and barrier characterization [8-10]. Mapping molecular cross-linking networks in hydrocolloids is essential to design dynamic barrier configurations capable of replacing fossil-resource frameworks [11-14]. Agricultural residue recycling forms a core cornerstone of the circular economy. As a leading global producer of pistachios (*Pistacia vera* L.), Türkiye generates thousands of tons of hard shell waste annually during industrial processing [2], emphasizing their structural utility and widespread biomass availability [15]. These residues are highly rich in structural cellulose (30–50%), hydrophobic lignin (20–35%), and hemicellulose (15–25%) [2]. Due to this rigid lignocellulosic architecture, pistachio shell pieces can be effectively upcycled as

natural fillers in biodegradable film design [16]. economic, and biocompostable reinforcement phase for polymer matrices [17].

The primary objective of this study was to develop eco-friendly edible films by utilizing micronized pistachio shell powder as a functional matrix reinforcement. Peanut oil was incorporated as a hydrophobic lipid phase to minimize water vapor permeability, while glycerol, kappa-carrageenan, and sodium alginate established structural flexibility and gel matrix stability. The films were systematically characterized *via* FT-IR, thickness, water solubility, moisture content, and WVP analyses, and validated through *in vivo* shelf-life trials on fresh strawberry and apple slices.

EXPERIMENTAL

Materials

Pistachio shells (domestic agrowaste) and peanut oil (Nkaresabun) served as raw materials. Kappa-carrageenan (Model: 316, Benosen), sodium alginate (Alfasol), sorbitol (Zag Kimya), and glycerol (Merck) formed the film matrix. Distilled water was used as the solvent. Fresh strawberry and apple slices were selected as target food models to evaluate shelf-life extension performance.

Preparation of pistachio shell powder

The endocarp of pistachio shells was mechanically ground using a ring mill and sieved through a 250 µm mesh to obtain a uniform particle size distribution for optimal dispersion.

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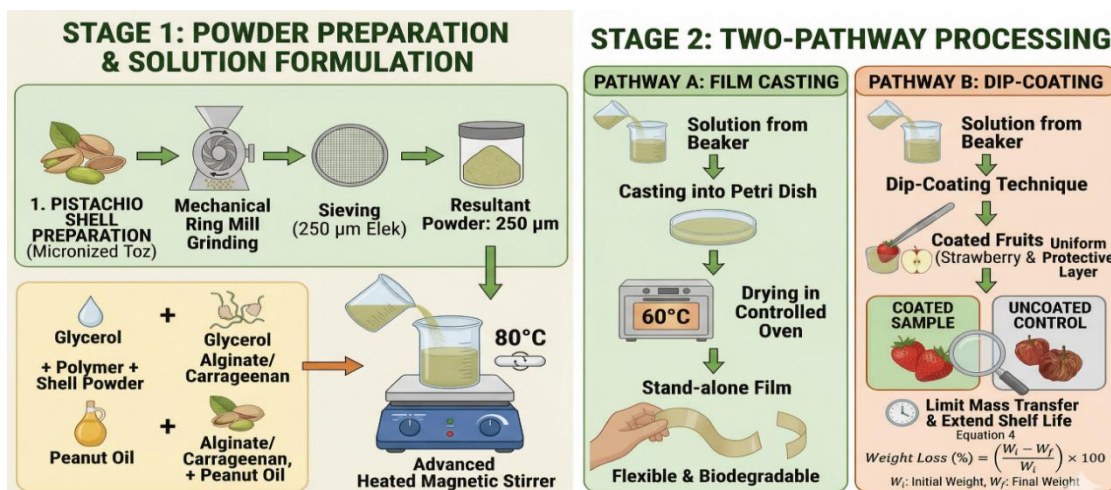


Figure 1. Schematic representation of the fabrication process of edible films

Table 1. Detailed formulations and component ratios of the edible films

Sample	Shell (g)	Polymer (g)	Glycerol (mL)	Additive (wt%)*	Plasticizer (wt%)*
Car-P2.5-G8	2.4	2.5	8	16.02%	67.29%
Car-P1.5-G8	2.4	1.5	8	17.17%	72.10%
Car-P1.0-G8	2.4	1.0	8	17.80%	74.78%
Car-S2.0-G10	2.0	1.0	10	12.82%	80.77%
Car-S1.5-G10	1.5	1.0	10	9.93%	83.44%
Alg-G6	2.4	2.5	6	19.26%	60.68%
Alg-G8	2.4	2.5	8	16.02%	67.29%

*Weight percentages (wt%) for additives (shell powder) and plasticizers (glycerol) are calculated relative to the total solid weight ($W_{total} = \text{Polymer} + \text{Shell} + \text{Glycerol}$). Glycerol density was taken as 1.26 g/mL.

The resulting micronized powder was stored in a desiccator at room temperature ($22 \pm 0.5^\circ\text{C}$) to prevent moisture absorption prior to formulation

Synthesis of edible films

The edible films were synthesized *via* the solvent casting method based on the formulations detailed in Table 1. Glycerol concentrations varied between 60.68% and 80.77% (w/w) relative to the total solid content. A multi-stage thermal dissolution protocol was executed at 80°C under continuous magnetic stirring. For the alginate series, glycerol, sodium alginate, and carrageenan were sequentially dissolved in 100 mL of distilled water for 30, 15, and 15 min, respectively (60 min total). For the carrageenan (Car) series, carrageenan was pre-dissolved at 80°C for 30 min before adding the other components. Subsequently, pistachio shell powder (1.5, 2.0, or 2.4 g; representing 9.93% to 21.90% of total solids) and peanut oil (2 mL) were incorporated into the mixture.

The final film emulsion was stirred at 80°C for an additional 15 min to achieve homogeneous lipid phase distribution. The mixtures were cast into

molds and dehydrated in a controlled oven at 60°C for 24 h until a stable weight was reached. The dried films were equilibrated in a desiccator ($22 \pm 0.5^\circ\text{C}$) before characterization. Fig. 1 depicts the detailed schematic workflow of the multi-stage fabrication and casting process of the pistachio shell-reinforced edible films. A systematic coding system was applied: Car and Alg denote carrageenan and sodium alginate matrices, respectively, while P, G, and S represent polymer weight (g), glycerol volume (mL), and pistachio shell powder load (g), respectively.

The macrostructural appearance and physical flexibility of the fabricated edible films are illustrated in Fig. 2.

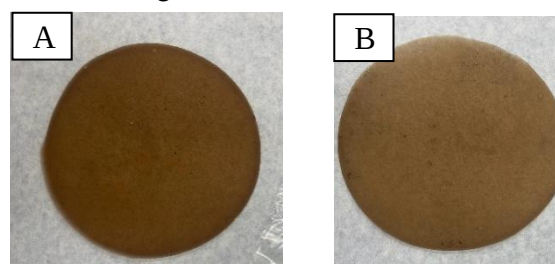


Figure 2. Macrostructural appearance of the produced A) Car-P2.5-G8 and B) Car-P1.0-G8

Characterization of edible films

- **Fourier transform infrared spectroscopy (FT-IR).** The chemical structures and functional group interactions of the synthesized edible films were analyzed using a Perkin Elmer Spectrum 100 FT-IR spectrometer. Spectral data were acquired via the attenuated total reflectance (ATR) technique by scanning over a wavenumber range of 4000 cm^{-1} to 650 cm^{-1} [18, 19].

- **Water vapor transmission rate (WVTR).** The moisture barrier capacity of the films was gravimetrically determined according to the modified ASTM E96 standard [20, 21]. Film samples ($2 \times 2 \text{ cm}^2$) were dried at 105 °C for 24 h to reach a constant mass, and then fixed airtight with parafilm to the mouths of test tubes containing 5 g of silica gel. The assemblies were placed in a constant humidity desiccator for 5 days. The weight change (ΔW) was recorded and the WVTR was calculated via Eqn. 1 [22]:

$$\text{WVTR} = \frac{\Delta W / \Delta t}{A} \quad (1)$$

where ΔW is the mass change (g), A is the exposed surface area (0.00015393 m^2), and Δt is the total experimental time (120 h).

- **Moisture content.** The moisture retention of the films was determined gravimetrically. Film specimens were dried at 105 °C until a constant weight was achieved. The moisture content percentage was calculated using Eqn. 2 [23, 24]:

$$\text{Moisture content (\%)} = \frac{M_1 - M_2}{M_1} \times 100 \quad (2)$$

where M_1 and M_2 represent the weights of the film before and after drying, respectively.

- **Water solubility.** To evaluate water resistance, dried film samples ($2 \times 2 \text{ cm}^2$, 105°C for 24 h) were immersed in 50 mL of distilled water and stirred at 150 rpm for 1 h using a magnetic stirrer. The insoluble fraction was separated via filter paper and dried at 105°C for 24 h to determine the final dry weight. The water solubility (WS) percentage was calculated using Eqn. 3 [24, 25]:

$$\text{WS (\%)} = \frac{W_0 - W_t}{W_0} \times 100 \quad (3)$$

where M_1 is the initial dry weight and M_2 is the remaining dry weight after dissolution.

- **Thickness.** The film thickness was measured at three random points using a digital micrometer (0.1 mm precision) to determine the average value [23, 26].

- **Fruit coating application.** The protective efficacy of the selected carrageenan and sodium alginate based formulations was evaluated on fresh strawberry and apple slices. The fruit samples were

treated using a standardized dip-coating technique to ensure a uniform surface layer. An uncoated control group was maintained under identical environmental conditions for validation. Over a 14-day storage period, the physical barrier performance against moisture transfer was quantitatively assessed by monitoring the weight loss percentage according to Eqn. 4 [27-29]:

$$\text{Weight loss (\%)} = \frac{W_i - W_f}{W_i} \times 100 \quad (4)$$

where W_i is the initial weight and W_f denotes the weight at day 14.

RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy (FT-IR)

The molecular architecture and functional group interactions of the synthesized edible films were monitored via FT-IR spectroscopy (Fig. 3). All membranes exhibited a prominent, broad absorption band at 3280–3307 cm^{-1} , corresponding to the O–H stretching vibrations of adsorbed water, glycerol, and the hydrophilic polymer backbones [18]. The successful integration of components was confirmed by the aliphatic C–H stretching peaks observed at 2925–2926 cm^{-1} , inherent to the $-\text{CH}_2-$ groups of carrageenan and alginate chains [19].

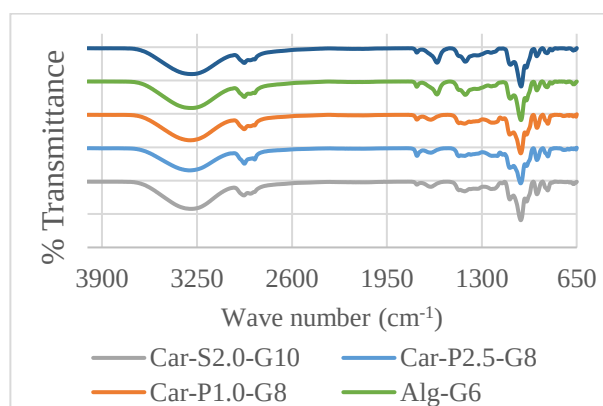


Figure 3. FTIR spectra of samples Car-S2.0-G10, Car-P2.5-G8, Car-P1.0-G8 and Alg-G6, Alg-G8

The presence of ester-based constituents was evidenced by the characteristic C=O stretching band between 1743–1746 cm^{-1} . Notably, the carrageenan-based formulations displayed a distinct peak at 1647–1648 cm^{-1} along with a symmetrical stretching band at 1412–1417 cm^{-1} , signifying the presence of carboxylate (COO^-) or sulfate (SO_3^-) groups. These shifts and peak combinations imply the formation of a robust intermolecular hydrogen-bonding network between the plasticizer and the biopolymer matrix, which is crucial for film flexibility. The fingerprint region further elucidated the polysaccharide skeleton and filler interactions. The absorption bands

in the 1103–1109 cm^{-1} and 1031–1034 cm^{-1} ranges are assigned to C–O stretching and glycosidic linkages (C–O–C), respectively, reflecting glycerol-matrix coordination [19]. Structural bending of the polymer chains was detected at 993–995 cm^{-1} and 922–924 cm^{-1} the latter specifically confirms the 3,6-anhydro-D-galactose units of carrageenan. Furthermore, the active SO_3^- stretching vibrations at 847–853 cm^{-1} validated the integrity of the carrageenan matrix. Finally, the spectral region between 670–730 cm^{-1} exhibited the aromatic C–H out-of-plane bending [30], confirming the successful incorporation of the lignocellulosic pistachio shell filler within the edible film framework. This structural consolidation is consistent with spectral parameters validated for extracted pistachio shell films [17]. Recent structural data by carbohydrate and hydrocolloid researchers [31–33] confirm that incorporating natural cross-linkers, chitosan networks, and polyphenolic agrowastes induce positive molecular assembly shifts without breaking the core polysaccharide skeleton.

Barrier properties and hydrolytic stability

The physical dimensions, moisture resistance, and hydrolytic stability of the synthesized edible films are consolidated in Table 2. The film thicknesses spanned a spectrum between 0.6 mm and 1.3 mm, where the sodium alginate series formed more compact layers (0.6–0.8 mm) than the carrageenan counterparts due to denser molecular packing during dehydration. This structural arrangement directly governed the barrier dynamics; the water vapor permeability (WVP) values ranged from 0.704 to 2.593 $\text{g/m}^2\cdot\text{h}$, demonstrating superior efficiency over conventional benchmarks.

Table 2. Comprehensive physical, barrier, and stability results of the edible films

Sample	Thickness (mm)	WVP ($\text{g/m}^2\cdot\text{h}$)	Moisture loss (%)	Water solubility (%)
Alg-G8	0.6	0.704	32.63	81.52
Alg-G6	0.8	1.998	30.77	86.90
Car-P1.0-G8	1.3	1.608	28.26	48.67
Car-P2.5-G8	0.9	1.424	24.02	59.94
Car-P1.5-G8	1.0	2.409	41.23	77.19
Car-S2.0-G10	1.0	0.861	35.62	84.19
Car-S1.5-G10	1.0	2.133	25.71	65.30

This high-efficiency performance is fundamentally driven by the inclusion of peanut oil

as a hydrophobic lipid phase [20], which establishes a tortuous path for water molecules within the polymer network [21, 34]. Similar structural designs utilizing active lipid barriers and agro-industrial particles have been shown to drastically restrict internal moisture transport channels [34–36].

The interplay between plasticization, matrix density, and filler load revealed clear correlations among WVP, moisture retention, and water solubility. In the alginate series, increasing glycerol from 6 mL to 8 mL minimized internal friction of the casting solution, yielding a thickness of 0.6 mm and optimizing interfacial adhesion with the lipid phase to achieve the minimum WVP of 0.704 $\text{g/m}^2\cdot\text{h}$. Strategic plasticizer adjustment expands the free volume between polymer chains [22, 23, 26], defining the physical assembly and water affinity of the sheets. Due to the hygroscopic nature of glycerol and the hydrophilicity of the sodium alginate backbone, both alginate formulations exhibited a high-water affinity, leading to elevated moisture content [23] and water solubility rates exceeding 81% [25]. This balance matches conventional biopolymer paradigms noting that natural hydrophilic hydrocolloids require precise formulation adjustments to withstand aqueous erosion [35–38]. Conversely, structural stability in carrageenan systems was governed by the density of the polysaccharide network and the lignocellulosic filler load rather than plasticizer dominance alone. Increasing carrageenan content reinforced the matrix; a comparative analysis between Car-P2.5-G8 and Car-P1.0-G8 showed an 11% reduction in water solubility due to a dense, dispersion-resistant gel network. The micronized pistachio shell powder functioned as a pivotal regulator of internal porosity and water resistance. Increasing the shell load by 0.5 g (comparing Car-S1.5-G10 and Car-S2.0-G10) tightened the biopolymer matrix, restricting free volume for vapor transport to lower the WVP to 0.861 $\text{g/m}^2\cdot\text{h}$. Concurrently, the hydrophobic lignin fraction of the higher filler load restricted moisture absorption—inducing a 10% shift in moisture content—and enhanced resistance against aqueous erosion to lower water solubility by 19%. A similar physical paradigm confirms that the strategic dispersion of crystalline shell residues within polymer networks modifies thermal stability, matrix spacing, thickness parameters, and subsequent moisture flux behavior depending on the absolute loading ratio [17, 39]. These findings show that balancing hydrophobicity, water resistance (48.67% to 86.90%), and barrier parameters can be precisely tailored by optimizing glycerol-lipid-filler ratios to satisfy active packaging specifications.

Practical application: shelf-life extension of perishable fruits

The practical efficacy of the synthesized edible films was demonstrated by monitoring the weight loss of highly perishable strawberry and apple slices over a 14-day storage period (Table 3). While uncoated control strawberries suffered a severe mass reduction of ~70%, the application of the Car-S2.0-G10 formulation successfully restricted this loss to 40.51%, significantly outperforming the Alg-G6 coating (68.03%). A similar trend was observed for apple slices; uncoated samples reached a near-total mass reduction of 90%, whereas the Car-S2.0-G10 coating provided superior moisture retention by limiting the loss to 70.06%, outperforming the 86.59% loss recorded for the Alg-G6 formulation. These suppression trends align with recent active coatings outlined for post-harvest preservation using agrowaste residues [1, 27, 31, 32], as well as sustainable green packaging paradigms [34, 40].

Table 3. Weight loss analysis of coated strawberry and apple samples over a 14-day storage period

Sample	Initial weight (g)	Weight at day 14 (g)	Weight loss (%)
<i>Strawberry samples</i>			
Car-S1.5-G10	11.11	6.33	43.02
Car-S2.0-G10	10.96	6.52	40.51
Alg-G5 (GA-2)	13.04	4.40	66.26
Alg-G6 (GA-3)	20.24	6.47	68.03
<i>Apple samples</i>			
Car-S1.5-G10	6.67	1.85	72.26
Car-S2.0-G10	8.75	2.62	70.06
Alg-G5 (GA-2)	10.11	1.76	82.59
Alg-G6 (GA-3)	15.21	2.04	86.59

The variation in these results is governed by the interplay between the polymer matrix, plasticizer concentration, and filler load. The carrageenan-based Car-S2.0-G10 formulation emerged as the most effective coating because the inherently denser structural network of carrageenan, when reinforced with a 2.0 g pistachio shell load, forms a robust physical barrier that regulates respiration rates and minimizes transpiration-induced mass transfer. In contrast, the higher weight loss in the sodium alginate-based series is attributed to high glycerol concentrations, which enhance the hygroscopic nature of the coating and facilitate rapid moisture exchange with the environment. Furthermore,

variations among the carrageenan recipes suggest that structural stabilization provided by the pistachio shell powder is a critical regulator of film porosity; formulations with optimized filler-to-plasticizer ratios showed a more consistent ability to seal the fruit surface against mass loss. As established by innovative packaging reviews [3, 4, 29], upcycling fibrous wastes creates complex tortuous paths that retard environmental gas diffusion, stabilizing the post-harvest cellular integrity of perishable horticultural matrices [28, 33]. Although these loss percentages exceed some literature benchmarks due to experimental variables—such as high initial fruit moisture, specific plasticizer types, and ambient humidity—the substantial relative improvement over the control groups confirms the commercial potential of these waste-derived films as sustainable active coatings.

CONCLUSION

In this study, novel bio-based edible films were successfully developed by reinforcing sodium alginate and carrageenan matrices with micronized pistachio shell powder, plasticized with glycerol, and hydrophobic-modified using peanut oil. FT-IR spectroscopic analysis verified the successful integration of the lignocellulosic filler and the formation of a robust intermolecular hydrogen-bonding network within the biopolymer matrices. The structural modification significantly enhanced the functional packaging parameters, yielding exceptional moisture barrier efficiencies with tailored WVP values between 0.704 and 2.593 g/m²·h, alongside highly tunable water solubility profiles (48.67–86.90%). *In vivo* post-harvest storage trials on fresh strawberry and apple slices over a 14-day period validated the practical efficacy of the developed coatings. The optimized carrageenan-based formulation (Car-S2.0-G10) emerged as the most efficient protective layer, substantially suppressing critical mass losses to 40.51% in strawberries and 70.06% in apples compared to ~70% and ~90% reductions in uncoated control groups, respectively. Consequently, this study demonstrates that upcycling abundant agro-industrial residues into high-performance edible coatings offers a scalable, circular-economy-driven strategy to extend the shelf life of perishable horticultural produce while mitigating environmental plastic waste.

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