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Evaluation of smart packaging functions of black carrot extract with polysaccharide-based films

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This work aims to assess the manufacture of polysaccharide-based films derived from carrot waste incorporating pectin (P) and a developed alginate (A) matrix with black carrot extract (BC). P was extracted from carrot waste with a hot acid extraction methodology. The extraction yield was determined to be 17.71%. Biopolymer films designated A/P and A/P/BC were produced *via* the casting technique. In the films, carrot P and A solution were utilized at varying concentrations (0–1% by weight) to attain a total biopolymer content of 2% by weight. Furthermore, BC was integrated into the film at concentrations of 0.2%, 0.4%, and 0.6% (v/v). Analyses of opacity, swelling, moisture, dynamic mechanical strength, and scanning electron microscopy (SEM) were conducted to characterize the films. Furthermore, pH values of 4, 8, and 12 were chosen to evaluate the films' sensitivity to pH. The findings indicated that P concentration significantly influenced the opacity, swelling, and moisture content of the film samples, while concurrently reducing their mechanical strength. Additionally, as the amount of BC incorporated into the films grew, both opacity and mechanical strength enhanced, while moisture content diminished. The results revealed that carrot P and BC could be utilized in the production of smart packaging films. The production of these composite films represents an innovative strategy to address food waste that would otherwise be discarded, hence mitigating environmental effect. This concept has the ability to generate sustainable biobased packaging while simultaneously minimizing waste production.

Keywords: Smart packaging, polysaccharide-based, carrot pectin

INTRODUCTION

The European Union (EU) has introduced a new bioeconomic strategy designed to safeguard the environment and enhance the standard of living of its citizens. This approach will guarantee that all packaging in the EU market is recyclable and will decrease plastic consumption by 2030 [1]. Food packaging constitutes one of the greatest global sectors. The worldwide experiencing heightened interest in sustainable, functional packaging solutions that prolong food shelf life and reduce environmental impact, propelled by growing environmental consciousness. Researchers have examined migration, heat processing, dehydration, acidification, and the integration of antimicrobial/antioxidant compounds into plastic materials to inhibit food spoiling [2-4].

Recent advancements in science and technology have led in the creation of active and intelligent food packaging. Active packaging incorporates agents that engage with food to extend its shelf life, whereas smart packaging use colorimetric agents to assess and provide information regarding food quality without direct touch [4, 5]. pH-sensitive smart packaging often comprises a biodegradable natural or synthetic polymer matrix along with a pH-sensitive indicator [6]. Colorimetric compounds (anthocyanin, betacyanin, methyl red, alizarin) utilized as pH indicators provide customers with information regarding the state of the food [7].

Alginate is an economical, biocompatible, non-toxic, biodegradable polysaccharide having film-forming capabilities. Alginate's capacity to create compatible blends with various polymers and enhance the characteristics of composite materials positions it as a promising candidate for food packaging [5,8,9]. Pectin is a natural polymer located in the middle lamella of plant cell wall. Pectin has attracted interest from researchers as a food packaging material, extending beyond conventional food applications. Pectin's film-forming capability, excellent barrier qualities, non-toxic, compatibility with other polymers, carrier material for ph-responsive pigments and adaptability render it an advantageous choice for the creation of environmentally sustainable packaging [4]. It can be acquired from multiple sources. The majority of

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these sources consist of fruit (oranges, lemons, apple pomace) and vegetable (sugar beet, carrot, potatoes) biomass that is deemed waste after processing [10]. Pectin is extracted using dilute mineral acids such as hydrochloric acid and nitric acid in a conventional extraction method. Nonetheless, mineral acids include downsides including toxicity and damage to the environment. To deal with these issues, organic acids, specifically citric acid, may be favored for extraction [11, 12].

Carrot pulp is regarded as waste in the fruit juice industry; nevertheless, it can be utilized as animal feed or fertilizer. Pectin can be derived from biomass to provide green products with significant added value. In carrot pulp it is categorized as 'low methoxyl pectin' [13]. In the food industry, anthocyanins can be serve as a colorimetric indicator, providing a natural alternative to synthetic dyes for evaluating food quality especially in smart food packaging [14]. BC is abundant in bioactive components and contains a significant amount of anthocyanin. The anthocyanin included in black carrots demonstrates superior stability. Besides preserving its stability against light and heat, it can also preserve its characteristics at elevated pH levels [15, 16]. Tavasolli *et al.* [17] created pH-sensitive smart packaging films by incorporating anthocyanin derived from sumac into pectin-chitosan nanofibers by a solvent casting method.

This work developed films utilizing a polysaccharide-based matrix, utilizing the film-forming, non-toxic and biocompatible characteristics of alginate and pectin. Pectin was extracted from carrot pulp, and pH-sensitive films were created by including black carrot extract into the polysaccharide-based film matrix. The films were subjected to characterization tests, including mechanical strength, swelling, moisture, and opacity, to assess their suitability for smart food packaging. The extraction of pectin from carrot pulp, an agro-industrial byproduct, and the creation of pH-sensitive packaging from BC, alongside the exploration of its applicability, would substantially enrich the existing literature.

EXPERIMENTAL

Pectin extraction

Pectin was extracted from carrot pulp utilizing a typical thermal extraction method at 90°C for 80 minutes and a pH of 1.3 [13]. Subsequent to extraction, the mixture was subjected to filtration through a filter cloth and allowed to cool to ambient temperature. Ethanol was incorporated into the cooled solution at a 1:2 (v/v) ratio and allowed to precipitate in +4 °C temperature for 20 h. The precipitate was obtained by centrifugation at 1000 g for 15 min, washed with distilled water and subsequently dried in an air oven at 40 °C for 16 h to yield pectin. The pectin yield was determined by the mass difference between the dried pectin and the initial dry raw material.

BC extraction

A mixture of black carrot powder and citric acid solution was added to the flask at a ratio of 1 g to 10 mL and allowed to blend at ambient temperature. The solution underwent centrifugation at 3000 rpm for 5 min, after which the supernatant fractions were combined and adjusted with citric acid extraction solution to match the flask capacity [18].

Production of films

Film-forming solutions were prepared with a total polymer concentration of 2% (w/v) by adjusting the weight ratio of sodium alginate (A) to pectin (P) as detailed below: Formulations of 100:0, 90:10, 80:20, 70:30, 60:40, and 50:50 (A:P, w/w) were prepared, incorporating black carrot extract (BC), abundant in anthocyanins, at concentrations of 0.2%, 0.4%, and 0.6% (v/v) into the film formulation where A and P were utilized in equal ratios. The film solution was stirred at 50 °C for 1 h and cross-linked with 1% $CaCl_2$ [19]. The films were produced using the cast film method. Glycerol is preferred as a plasticiser to impart flexibility and films drying oven at 40 °C. In the last stage, physical cross-linking was executed using 2% $CaCl_2$ for an average of 30 min. The films produced were assigned as A, A9-P1, A8-P2, A7-P3, A6-P4, A5-P5, AP-BC2, AP-BC4, and AP-BC6, accordingly.

Film characterization

JEOLJSM-7100-F scanning electron microscope (SEM) was used used to study the A-P and A-P-BC films structure.

The film thickness was quantified using the Dasqua 4310 digital micrometer electronic gauge. The thickness of each film was measured at three locations: one at the center and two in the areas next to it. Tensile strength values of the films were determined by a Universal Testing Machine (Ankarin) with ASTM D882 standard.

The opacity, or light transmittance, of the films was evaluated using a UV-Vis spectrophotometer (UV1280, Shimadzu, Japan). The absorbance at 600 nm was determined using Equation 1.

$$Opacity = \frac{Abs_{600}}{x} \quad (1)$$

Abs_{600}: absorbance of the film at 600 nm, x: thickness of the film (mm).

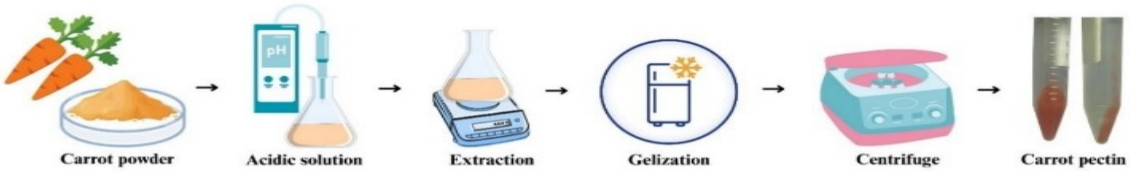

Figure 1. Schematic representation of the carrot pectin extraction

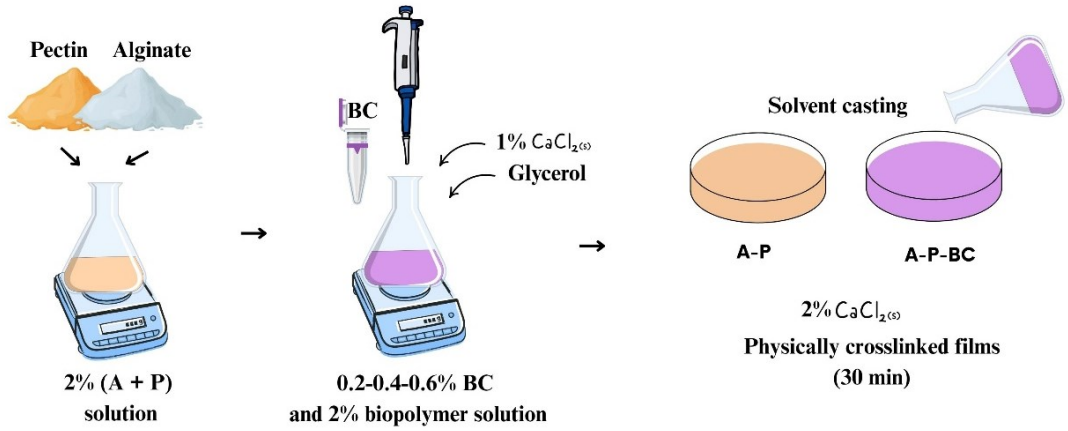

Figure 2. Schematic representation of the carrot P, A and BC composite films production

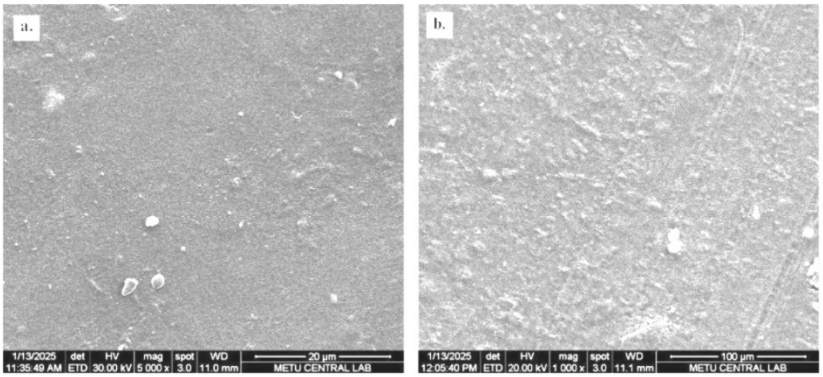

Figure 3. SEM images of (a) A5P5, (b) A-P-BC6 films

For the swelling ratio, samples of the films were weighed (Wd) and put in 30 mL of distilled water at room temperature for 24 h, after which they were taken out, wiped with filter paper and weighed again (Ws).

The swelling degree was determined using Equation 2.

$$Swelling\ ratio\ (\%) = \frac{W_s - W_d}{W_d} x100 \qquad (2)$$

The moisture content was determined by drying the films in an oven at 105 °C for 24 h until a constant weight was achieved. The moisture content was determined by dividing the difference between the starting weight (Wi) and the weight lost during drying (Ws) by the starting weight (Wi) (Equation 3).

$$Moisture\ content\ (\%) = \frac{W_i - W_s}{W_s} x100 \qquad (3)$$

For colorimetric measurement, AP-BC6 films were cut into 3 cm×3 cm squares, submerged in buffer solutions with pH values of 4, 8, and 12 at room temperature, and the films' sensitivity to varying pH conditions was evaluated [20]. The alteration in color of the films was assessed utilizing a colorimeter (Linshang, LS173, Shenzhen, China). The mean Hunter color indices (L*, a*, and b*) of the films were recorded three times. The overall color difference was determined based on the data at various pH levels utilizing Equation 4.

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \qquad (4)$$

MATERIALS

Alginate was from Sigma Aldrich; citric acid, ethanol were of analytical grade, purchased from Merck Chemicals, Türkiye. Fresh carrots (*Daucus carota L.*) and black carrot powder (*Daucus carota ssp. sativus var. atrorubens Alef.*) were purchased from a local market in Çanakkale. Black carrot

powder was utilized in its commercially available form.

RESULTS AND DISCUSSION

The elements utilized in film development have significance for surface morphology. The physical parameters of a film influence its mechanical and optical charaeristics [21]. A and P exhibit a uniform surface structure owing to their capacity to create smooth films. Conversely, tiny particle forms were seen at certain locations in the A5-P5 film (Figure 3a). A smooth surface is seen in the AP-BC6 film, likely resulting from the compatible interaction between the biopolymer matrix and BC (Figure 3b). This may result from the gelation produced during the incorporation of the cross-linking agent, which remained undissolved despite the inclusion of a plasticizer. Nonetheless, all fabricated biocomposite films often exhibit a smooth and uniform surface, notwithstanding the presence of minor particles in the SEM pictures (Figures 4b, 4c).

The swelling percentages of the A-P film series range from 28.571% to 120.588%. A swelling percentage of 120.588% was specifically noted in the A5-P5 control films. This condition may have resulted from the hydrophilic nature of the matrix structure and the ratios of biopolymers it comprises. The swelling percentage rose concomitantly with the increase of pectin concentration in the matrix (Figure 4). This rise results from pectin's hydrophilic nature, which facilitates its interaction with water via hydroxyl groups (-OH). Films with different ratios of BC extract added also had a constant A and P ratio. Consequently, the films disintegrated in an aqueous medium within 24 h. Consequently, the swelling percentage was incalculable. Moreover, the elevated anthocyanin concentration in BC has been shown to enhance the polarity of the films, resulting in their solubility in polar solvents like water [22].

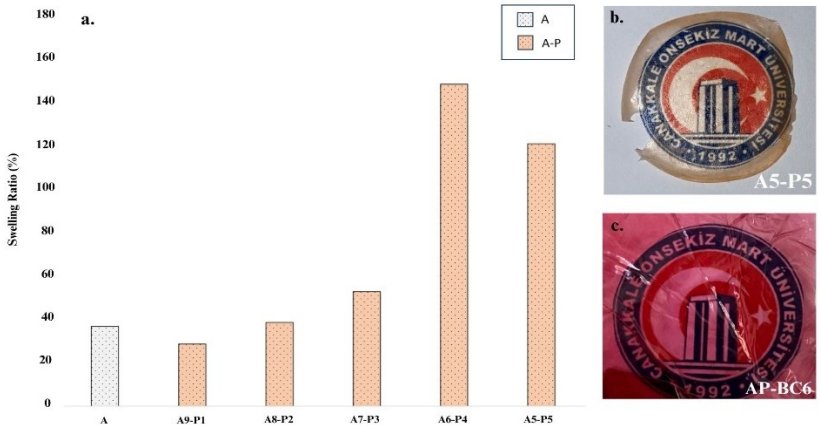

Figure 4. (a) Swelling ratio of A and A-P films, (b) A5-P5, (c) AP-BC6 biocomposite films

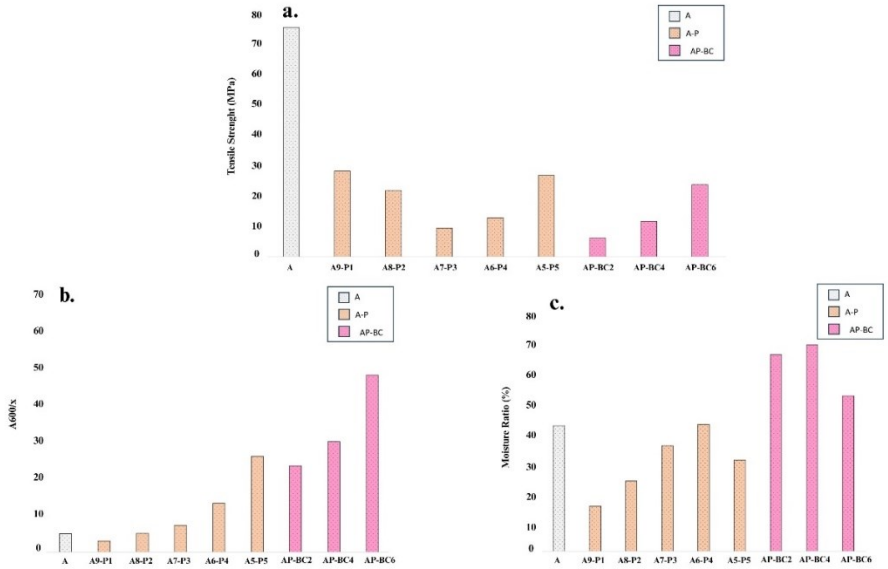

Figure 5. (a) Tensile strength, (b) Opacity, and (c) Moisture content of the biopolymer-based films.

The ability and performance of films are essential for maintaining the integrity of packaged food goods [23]. The A-P series films displayed thicknesses between 0.0287 mm and 0.053 mm, while the AP-BC series films revealed thicknesses from 0.010 mm to 0.014 mm (Figure 5a). The incorporation of BC influences film thickness, with an increase in BC correlating to greater thickness. The increase in thickness may result from the electrostatic repulsion provided by the anthocyanins in BC interacting with the biopolymers (A-P) and alterations in film density [24]. The tensile strength (TS) of pure A was 74.90 MPa, whereas the TS of A-P films varied between 9.477 and 28.019 MPa, and the TS of AP-BC films ranged from 6.167 to 23.571 MPa (Figure 2b). The incorporation of carrot pectin into the matrix resulted in a substantial reduction in mechanical strength. The maximum strength was recorded in films exhibiting an equal A-P ratio. A comparable scenario was noted in a study about renewable films composed of alginate and low-methoxyl pectin [25]. Nonetheless, as the incorporation of BC into the films increased, the mechanical strength correspondingly improved. The endurance of biocomposite films is influenced by polymer composition, intra- and intermolecular forces, and the structure of the film network.

A-P film solutions are typically apparent due to their uniform composition, with opacity increasing upon the incorporation of pectin into the films (Figure 5b). The opacity of the films markedly enhanced with higher BC concentration. The decrease in inter-chain spaces caused by double cross-linking of the films led to enhanced opacity [26]. Elevated opacity is believed to augment the films' capacity to shield light-sensitive meals from the detrimental impact of visible light [27].

The moisture percentages in A-P films were determined to be 16.071% and 45.098%, representing the lowest and highest values, respectively. The incorporation of carrot pectin into the film matrix resulted in a decrease of moisture content relative to the pure alginate film. Another study found that the moisture content of films containing alginate and pectin was 18.57% [26]. In the AP-BC film series, the moisture content varied from 55.294% to 73.333% (Figure 5c). Following the incorporation of BC into the alginate-carrot pectin films, the moisture content initially rose and subsequently declined. The hydrophilic -OH groups of polysaccharides will establish hydrogen bonds with the -OH groups in BC. This interaction may restrict the cross-linking of water molecules and polysaccharides, leading to variations in moisture content. A comparable outcome could occur contingent upon the sort of additives employed [20].

The increased content of anthocyanin components in black carrots correlates with pH-dependent variations. Table 1 presents the color spectra and color change data of the films. Certain literature research examined the pH sensitivity of the generated films without presenting a control film group [20, 28]. AP-BC6 films exhibit sensitivity to both acidic and basic buffers. At pH levels of 4, 8, and 12, the film's coloration transitioned from pale pink to brown. The color alteration transpired about 1 to 2 min. ΔE values beyond 5 signify discernible color alterations in the videos. As the pH value rises, the brightness (L*) of the films decreases, while the a* and b* values suggest a transition towards red-yellow shades, concluding in a brown tone. Films containing added BC are regarded as effective natural pH sensors.

Table 1. Colorimetric change of AP-BC6 films at pH levels 4, 8, and 12

pH	L*	a*	b*	ΔE	Color
4	75.47	18.15	9.05	32.64	
8	73.17	22.60	12.75	28.37	
12	57.24	12.00	8.11	34.55	

CONCLUSION

The development of pH-sensitive films by incorporating black carrot extract into alginate films, resulting in a suitable biocomposite with pectin derived from carrot waste, demonstrates potential for innovative smart packaging solutions. The findings indicated that the incorporation of BC into A-P films influenced their physicochemical characteristics. As the quantity of BC augmented, the film thickness, strength, and opacity escalated, whereas the moisture content diminished. The resulting films often exhibit a smooth surface and a slender structure. Films containing added BC are believed to function as natural pH indicators. The films demonstrate unpredictable actions when maintained at room temperature, rendering them more appropriate for assessing the quality of frozen food. Our research may yield novel concepts for the advancement of smart packaging, specifically with

the potential of biocomposite films derived from waste to tell consumers about freshness.

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