Wiley
Journal of Food Quality
Volume 2025, Article ID 3191264, 9 pages
https://doi.org/10.1155/jfq/3191264

WILEY

Research Article

Comparative Study of Pectin and Kappa-Carrageenan-Based Films Loaded With Boswellic Acid and Acetyl-11-Keto-Beta-Boswellic Acid for Active Packaging

Yasir Abbas Shah,[1] Saurabh Bhatia,[1,2] Ahmed Al-Harrasi,[1] Mohammad Tarahi,[3] Talha Shireen Khan,[1] Tanveer Alam,[4] Esra Koca,[5] and Levent Yurdaer Aydemir[5]

[1]*Natural and Medical Sciences Research Center, University of Nizwa, P.O. Box 33, Birkat Al Mauz, Nizwa 616, Oman*
[2]*School of Health Science, University of Petroleum and Energy Studies, Dehradun 248007, India*
[3]*Department of Food Science and Technology, School of Agriculture, Shiraz University, Shiraz, Iran*
[4]*Sabanci University Nanotechnology Research and Application Center, Sabanci University, Orta Mahalle, Universite Caddesi, No. 27, Tuzla 34956, Istanbul, Türkiye*
[5]*Department of Food Engineering, Faculty of Engineering, Adana Alparslan Turkes Science and Technology University, Adana 01250, Türkiye*

Correspondence should be addressed to Saurabh Bhatia; sbsaurabhbhatia@gmail.com

Received 8 September 2024; Accepted 8 January 2025

Academic Editor: Cristina Anamaria Semeniuc

Copyright © 2025 Yasir Abbas Shah et al. Journal of Food Quality published by John Wiley & Sons Ltd. This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

Active packaging is crucial for extending shelf life, maintaining food quality, and reducing reliance on synthetic preservatives by preventing spoilage, oxidation, and microbial contamination. In this study, boswellic acid (BA) and acetyl-11-keto-beta-boswellic acid (AKBA) were isolated from the oleo-gum resin of *Boswellia sacra* and incorporated into the pectin and kappa-carrageenan-based films for active packaging. The fabricated films were analyzed to examine the effect of these active compounds on the mechanical, optical, barrier, chemical, structural, hydrophobic, and antioxidant properties. The incorporation of AKBA and BA into the films resulted in a decrease in transparency and tensile strength; however, the elongation at break did not show any significant variations. The film samples exhibited improved antioxidant properties as confirmed by DPPH and ABTS free radical scavenging activities. The ΔE value represented the overall color difference in AKBA- and BA-loaded films as compared with the blank film sample. The FTIR spectrum of AKBA- and BA-loaded films exhibited shifts and intensity changes, suggesting the intermolecular interaction within the film matrix. Moreover, the water contact angle measurements revealed that the films containing AKBA showed a noticeable increase in contact angles, reflecting an enhancement in hydrophobicity. The current study offers a valuable approach for developing active packaging films with potential applications in food packaging.

Keywords: active packaging; biopolymeric films; food packaging; organic acids

1. Introduction

Food packaging reduces the spoilage of food products and extends their shelf-life by acting as a barrier against external spoilage factors, such as microorganisms, moisture, oxygen, and UV light. The food packaging market has attracted more attention due to the increase in consumer demand for ready-to-eat and packaged food products because of urbanization and lifestyle changes. However, conventional food packaging materials (e.g., plastic, glass, and metal) have significant environmental impacts due to their non or less biodegradable nature and are difficult to recycle due to their complex composition. To deal with these harmful effects, it is necessary to reduce the consumption of conventional food

packaging materials, especially plastics, which increases the need to replace them with environmentally friendly and sustainable packaging materials [1–3].

Due to the film forming, thickening, gelling, stabilizing, and emulsifying properties of pectin and carrageenan, these natural polymers have been used in different sectors of the food, pharmaceutical, and cosmetic industries for years. Pectin is a complex polysaccharide, mainly consisted of homogalacturonan (HG), in addition to rhamnogalacturonan I and II (RG-I and -II), xylogalacturonan (XG), and apiogalacturonan (AG) [4, 5]. The degree of methyl-esterification (DM) is also another important structural factor of pectin, which determines their gelling and functional properties [6]. Carrageenan is a natural, biodegradable, biocompatible, water-soluble linear polysaccharide which is extracted from the cell wall of red seaweed. It is composed of alternating D-galactose and 3, 6-anhydrogalactose units linked by α-1,3- and β-1,4-glycosidic bonds [7]. Carrageenan is divided into three main categories: kappa (κ), iota (ι), and lambda (λ), which contain about 25%–30%, 28%–30%, and 32%–39% of sulfate ester groups, respectively. These structural differences significantly affect their functional attributes in food products, as kappa- and iota-carrageenan are capable of forming thermo reversible gels, while lambda-carrageenan can act as a thickener, not a gelling agent [8, 9]. It was also reported that the thin films produced with κ-carrageenan have better moisture barrier and mechanical properties compared to the ι-carrageenan-containing film samples [10].

Despite a wide range of techno-functional advantages, pectin and κ-carrageenan have several drawbacks, which restrict their further applications in the food packaging industry. For instance, pectin-based edible films showed poor physical properties with brittle structure and low tensile strength [11]. Almost the same results were reported by Martins et al. [12] for the biopolymeric films made from pure κ-carrageenan. The combination of pectin and κ-carrageenan has attracted much attention from different industries, as an effective way to develop food or pharmaceutical products with the desired physicochemical properties, such as dairy desserts [13], low-fat beef frankfurters [9], rice extrudates [14], hydrogels [5], and nanoparticles [15]. However, there is still a lack of in-depth study on how these biopolymers interact with each other to form composite films with favorable mechanical and barrier properties.

Boswellic acids (BAs) are natural mixture of pentacyclic triterpene acids, obtained from the oleo-gum resin of various Boswellia species, such as *Boswellia carteri*, *Boswellia sacra*, and *Boswellia serrata*. The six common types of BAs are α and β-boswellic acids (α and β-BAs), acetylated α and β-boswellic acids (α and β-ABAs), 11-keto-β-boswellic acid (KBA), and acetyl-11-keto-β-boswellic acid (AKBA). It has been proven that these organic acids have several bioactivities such as anti-inflammatory, antiseptic, anxiolytic, and anticarcinogenic effects [16, 17]. BAs can also be used for food preservation purposes due to their antibacterial, antifungal, and antioxidant properties [18]. In addition, Reshmy et al. [19] reported that BAs can easily interact with biopolymer molecules, such as cellulose, through H-bonding due to the presence of -OH and -COOH functions in their structure, which ultimately affects the structural, physical, and mechanical properties of the resulting films. Therefore, it is necessary to evaluate the effects of BAs on both biological and techno-functional properties of biopolymeric films from different sources to obtain an active film with the desired features.

Therefore, the aim of the present study was to develop a novel composite film based on pectin and κ-carrageenan incorporated with active ingredients including BA and AKBA as a favorable alternative to traditional packaging. In this respect, different aspects of composite film, e.g., the barrier, optical, mechanical, structural, and antioxidant properties were evaluated.

2. Materials and Methods

2.1. Materials. Pectin (pure, M.W 25,000–50,000) and carrageenan (Kappa, Refined ex.*Eucheuma Cottonii*) were procured from Sisco Research Laboratories Pvt. Ltd. (SRL), India. Glycerol (anhydrous, purity 99%) was purchased from BDH Laboratory Supplies, UK. The other chemicals used in this study were obtained from Sigma-Aldrich (St. Louis, MO, USA).

2.2. Extraction of BA and AKBA. The oleo-gum resin of *Boswellia sacra* was collected from various sites within the Dhofar governorate in Oman. It was then verified by the herbarium at the Natural and Medicinal Sciences Center, University of Nizwa, Sultanate of Oman.

To begin the extraction process for BA, 1.0 kg of resin was placed in a 5.0 L round-bottom flask (RBF) and combined with 3.0 L of pure toluene. This mixture was extracted at room temperature for 3 h with continuous stirring. After the extraction, the mixture was filtered, yielding 2.4 L of filtrate. The filtrate was then distilled under reduced pressure at 50°C until the volume was reduced to 1.0 L. This concentrated toluene solution was subsequently extracted with 0.5N NaOH solution. The resulting NaOH layer was acidified with 2N HCl until the pH reached 2–3 and then re-extracted with toluene. The toluene was evaporated under vacuum at 50°C, resulting in a viscous oily substance weighing 270 g, containing 50% BA. This BA (50%) was dissolved in dilute NaOH and filtered to remove any undissolved materials. The mixture was then cooled to 0°C–5°C and acidified with dilute HCl to a pH of 2.0, with HCl added dropwise and continuous stirring. Precipitation was observed, allowed to settle, and the precipitates were filtered. Finally, the precipitate was dried under vacuum to obtain 210 g of off-white solids, containing 70% BA.

For the extraction of AKBA, a 400 g sample of air-dried and ground *Boswellia sacra* resin was extracted using 100% methanol (MeOH) over a period of 2 days, following the procedure described by Khan et al. [20]. This extraction process yielded 285 mg of AKBA, identified as a UV-active compound with a retention time of 42 min, at a flow rate of 4 mL per minute [20].

2.3. Preparation of Active Films. To prepare the active films using the casting method, the following steps were followed based on the compositional ratios detailed in Table 1. The film-forming solutions consisted of pectin, κ-carrageenan, glycerol, and either AKBA or BA in specified amounts. Initially, 1.5% (*w*/*v*) pectin and 1% (*w*/*v*) κ-carrageenan were dissolved in distilled water separately, ensuring thorough mixing until a homogenous solution was achieved. Concurrently, both biopolymeric solutions were mixed and 1% (*v*/*v*) glycerol was added as a plasticizer to the solution and stirred consistently to ensure uniform distribution. For the preparation of blank films (PC), no active compounds were added. For PCA1 and PCA2 films, 30 and 50 mg of AKBA were added respectively. Similarly, for PCT1 and PCT2 films, 30 and 50 mg of BA were added, respectively. These components were mixed thoroughly to ensure even distribution of the active compounds within the film-forming solution. Once fully blended, the resulting mixtures were then casted into plastic petri plates and spread evenly to form a thin film. The petri plates containing film forming solutions were left to dry at room temperature until they formed a solid, uniform layer. Upon drying, the films were carefully peeled off from the plate surface and stored under controlled conditions until further analysis.

2.4. Thickness and Mechanical Parameters. The thickness of the film samples was determined using a digital micrometer (Yu-Su 150, Yu-Su Tools), as described by Bhatia et al. [21]. Five random measurements were measured to obtain the average thickness of each film in millimeters. The mechanical properties of the films were obtained according to the ASTM D882 standard method [22]. Films were cut into 60 mm by 7 mm dimensions and subjected between the grips of the texture analyzer (TA. XT plus, Stable Micro Systems, Godalming, England). The tensile strength (TS) and elongation at break (EAB) parameters were calculated using Exponent Connect software platform.

2.5. Water Permeability and Moisture Content. The water vapor permeability (WVP) of the films was evaluated by conditioning the samples in a desiccator for 10 days, where the humidity was maintained at 50%. The humidity levels were controlled by adding silica gel (0% humidity) and water (100% humidity). The WVP (g mm m^{-2} h^{-1} kPa^{-1}) was calculated using the following equation (1):

$$\mathrm{WVP} = \frac{(\Delta m)}{(\Delta t \times \Delta p \times A)} \times d. \quad (1)$$

In equation (1), "$\Delta m/\Delta t$" represents the weight of the moisture gain per unit of time (g/d), "Δp" symbolizes the water vapor pressure among the two sides of the film (kPa), "A" signifies the film surface (m^2), and "d" represents the thickness of the film in millimeters.

The gravimetric technique was used to evaluate the MC of the film samples. The samples were cut into 3 cm by 4 cm dimensions and weighed (W_1). After that, the films were dried at 105°C until a constant weight reached (W_2). The MC of edible films was calculated using the following equation (2):

$$\mathrm{MC} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100. \quad (2)$$

2.6. Opacity and Color Properties. The transparency of the films was assessed as described by Zhao, Wang, and Liu [23] using an ONDA-Vis spectrophotometer (Padova, Italy) at a wavelength of 550 nm. In addition, the color properties of the samples were evaluated using a spectrophotometer CR-5 (Konica Minolta, Tokyo, Japan). The overall color difference (ΔE) of films was calculated by the following equation (3):

$$\Delta E = \left(\Delta L^2 + \Delta a^2 + \Delta b^2\right)^{1/2}. \quad (3)$$

In equation (3), "ΔL," "Δa," and "Δb" represent the differences between the color parameters of the films (i.e., lightness (L^*), redness (a^*), and yellowness (b^*)) and the white standards ($L^* = 95.9$, $a^* = -0.09$, and $b^* = 0.1$).

2.7. X-Ray Diffraction (XRD) Analysis. The XRD analysis was carried out by a Bruker D8 Discover instrument at the voltage of 40 kV. The films were scanned with the scan speed of 0.500 s/point in the 2θ range of 5°–55°.

2.8. Fourier Transform Infrared (FT-IR) Spectroscopy Analysis. The FT-IR spectra of the fabricated films was obtained in the range of 4000–400 cm^{-1} and a resolution of 4 cm^{-1} using an InfraRed spectrometer (Bruker Tensor 37, Ettlingen, Germany).

2.9. Antioxidant Activity. The antioxidant activity of the films was evaluated using ABTS cation and DPPH free radical scavenging activities. The ABTS free radical binding capacity of film samples was measured according to a method described by Re et al. [24] with some modifications by measuring the change in absorbance at 734 nm for 6 min after 30 s vortexing with 50 mg of film and 1.9 mL of 7 mmol/L ABTS radical solution produced with potassium persulfate solution (2.45 mM).

DPPH free radical scavenging activity was determined according to method described by Brand-Williams, Cuvelier, and Berset [25]. Hundred mg of film samples were mixed with 1.95 mL of DPPH solution and the absorbance was adjusted (0.7 ± 0.2). The mixture was vortexed for 30 s and incubated in the dark at ambient temperature for 30 min. The absorbance at 517 nm was measured with a spectrophotometer (ONDA Vis spectrophotometer). The results were expressed as percentage inhibition of DPPH radical and ABTS cationic radical with the average of three replicates.

2.10. Water Contact Angle Measurements. The wettability properties of the films were analyzed employing OCA11 Contact Angle Analyzer (Data Physics Instruments GmbH, Germany). Using a microliter pipette, 1 μL drop of water was placed on a film sample of dimensions 2 × 2 cm and the contact angle was determined using dpiMAX software. The water droplet was imaged using an OCA11 camera (1044 × 1080 pixels).

Table 1: The compositional ratios of the components used for the film forming solutions.

Codes	Pectin (*w*/*v*) (%)	κ-Carrageenin (*w*/*v*) (%)	Glycerol (*v*/*v*) (%)	AKBA or BA (*w*/*v*)
PC	1.5	1	1	Blank
PCA1	1.5	1	1	AKBA 30 mg
PCA2	1.5	1	1	AKBA 50 mg
PCT1	1.5	1	1	BA 30 mg
PCT2	1.5	1	1	BA 50 mg

Note: PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.
Abbreviations: AKBA, acetyl-11-keto-beta-boswellic acid; BA, boswellic acid.

2.11. Statistical Analysis. In the current study, the average ± standard deviation (SD) of the triplicates was reported. Statistical comparisons among means were carried out via ANOVA and Duncan's tests using the SPSS (Version 17.0, IBM Company, Chicago, IL, USA).

3. Results and Discussion

3.1. Thickness and Mechanical Parameters of the Films. Table 2 shows that there are apparent differences in the properties of the films mainly the thickness, mechanical strength, and elongation at break. Film thickness increased with the incorporation of AKBA and BA. The blank film, PC, had the minimum thickness of 0.052 mm; however, active compound-loaded films showed increases in thickness. Such gain in thickness indicates that AKBA and BA are contributing to the structural build-up of the films matrix so that they become thicker and denser. It has been reported that the optimal thickness for polymeric films should be ≤ 0.25 mm [26]. However, the film samples that were prepared for this research study are within the thickness limits prescribed for polymeric films.

With the addition of AKBA and BA, the tensile strength of the films reduced. The blank film (PC) had the highest tensile strength of 22.07 MPa. On the other hand, films that contained AKBA and BA had much lower tensile strength values. The decrease in tensile strength shows that the incorporation of these active compounds weakens the active film structure, and thus they were not able to withstand external forces. It has been concluded that, for packaging films, a tensile strength of at least 3.5 MPa is required [27]. With the addition of AKBA and BA, the elongation at break was also decreased. The blank film (PC) was more flexible with 33.42% EAB. This compromised EAB means that the films became were more prone to cracking upon bending due to the addition of AKBA and BA. Earlier research has documented the influences of different organic acids on the mechanical properties of the films and their incorporation as well. The introduction of these organic acids in several studies resulted in decreased tensile strength but on the other hand led to improved flexibility of the films [28–30].

3.2. Water Permeability, Moisture Content, and Antioxidant Properties of the Films. WVP is a crucial parameter in evaluating the barrier properties of films, particularly in food packaging applications. In this study, the WVP values ranged from 0.436 g^* mm/(m2$^*h^*$ kPa) for the blank film (PC) to 0.867 g^*mm/(m2$^*h^*$ kPa) for the film containing 50 mg of BA (PCT2) as presented in Table 3. The blank film had the lowest WVP, indicating it is the most effective barrier to moisture. The addition of AKBA (PCA1 and PCA2) and BA (PCT1 and PCT2) progressively increased the WVP, suggesting that these compounds disrupt the matrix structure of the film, making it more permeable to water vapor. The increasing WVP with higher concentrations of AKBA and BA could be attributed to the increased free volume within the film matrix, which facilitates moisture transmission. The literature reports that organic acids, when incorporated into films, function as plasticizers, creating free space within the film matrix, and thereby facilitating the passage of moisture [28]. Similar results were obtained in our previous study in which the addition of AKBA increased the WVP values of the pectin and sodium alginate composite films [31].

Table 2: Thickness and mechanical parameters of the films.

Film samples	Thickness (mm)	Tensile strength (MPa)	Elongation at break (%)
PC	0.052 ± 0.008^a	22.07 ± 1.19^a	33.42 ± 2.51^a
PCA1	0.084 ± 0.009^b	7.75 ± 0.42^b	17.82 ± 1.20^b
PCA2	0.102 ± 0.008^c	5.04 ± 0.43^d	18.51 ± 1.01^b
PCT1	0.103 ± 0.010^c	5.68 ± 0.62^d	18.46 ± 1.15^b
PCT2	0.098 ± 0.010^c	6.88 ± 0.26^c	17.81 ± 0.98^b

Note: PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.
*Mean values, along with their standard deviations, are expressed as the mean ± standard deviation. The alphabets signify statistical significance difference ($p < 0.05$).

Moisture content is an indicator of the amount of water the film can retain. In this study, the blank film (PC) and the film with 30 mg of AKBA (PCA1) had almost similar MC values, suggesting that the introduction of a small amount of AKBA does not significantly affect the film's ability to retain moisture (Table 3). However, as the concentration of AKBA increased to 50 mg (PCA2), the MC drops significantly. Results of the current study align with the literature, in which a study reported that incorporation of the AKBA reduced the moisture content of the biopolymeric films [31]. Conversely, films containing BA (PCT1 and PCT2) showed higher MC, with PCT1 having the highest value. These results are consistent with studies showing that the inclusion of certain bioactive compounds can increase or decrease the moisture content depending on their interaction with the film's matrix.

Table 3: Water permeability, moisture content, and antioxidant properties of the films.

Film samples	WVP ((g*mm)/(m2*h*kPa))	Moisture content	DPPH radical (inhibition %)	ABTS cation radical (inhibition %)
PC	0.436 ± 0.025^{a}	41.86 ± 0.13^{b}	12.85 ± 0.84^{a}	26.70 ± 0.50^{a}
PCA1	0.696 ± 0.023^{b}	42.69 ± 0.97^{b}	14.04 ± 0.68^{ab}	35.06 ± 0.57^{b}
PCA2	0.836 ± 0.004^{c}	31.49 ± 0.29^{a}	14.52 ± 0.38^{b}	38.00 ± 0.75^{c}
PCT1	0.862 ± 0.014^{d}	49.48 ± 0.38^{c}	27.83 ± 0.76^{c}	38.71 ± 0.43^{c}
PCT2	0.867 ± 0.010^{d}	47.39 ± 1.36^{c}	35.12 ± 0.66^{d}	40.89 ± 0.92^{d}

Note: PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.

*Mean values, along with their standard deviations, are expressed as the mean ± standard deviation. The alphabets signify statistical significance difference ($p < 0.05$).

The antioxidant properties of the films were evaluated using DPPH and ABTS inhibition assays, which measure the ability of the films to scavenge free radicals. The results obtained for the antioxidant properties of the films are presented in Table 3. The blank film (PC) showed the lowest antioxidant activity, as expected, since it contains no active ingredients. When 30 mg of AKBA was added (PCA1), there was a slight increase in antioxidant activity, with further improvements observed at the higher AKBA concentration (PCA2). This suggests a dose-dependent effect of AKBA on the antioxidant capacity of the film. However, the most significant enhancement in antioxidant activity was observed in the films containing BA (PCT1 and PCT2), particularly at the 50 mg concentration (PCT2). BA is known for its strong antioxidant properties, which are reflected in the high DPPH and ABTS inhibition percentages [32, 33]. The results confirm that BA, especially at higher concentrations, can significantly enhance the antioxidant properties of the films, making them more effective in applications requiring antioxidant protection.

3.3. Opacity and Color Attributes of the Films. Opacity refers to the degree to which a film obstructs light from passing through it, and it plays a crucial role in packaging applications where protection from light is necessary. In this study, the blank film (PC) showed the lowest opacity at 1.50, indicating that the film is more transparent (Table 4). When 30 mg of AKBA was added (PCA1), opacity increased significantly to 13.56 and further rises to 17.11 with 50 mg of AKBA (PCA2). This observation suggests that AKBA increases the film's density or particle dispersion, reducing its transparency. Films with BA (PCT1 and PCT2) also showed increased opacity compared with the blank but remain lower than the AKBA films, with values of 6.47 and 8.61, respectively. The "L^*" value indicates the lightness of the films. The blank film (PC) showed the highest "L^*" value of 96.39. Adding 30 mg of AKBA (PCA1) reduces lightness to 91.17, and this further dropped significantly to 83.35 with 50 mg of AKBA (PCA2). Films containing BA (PCT1 and PCT2) maintain higher lightness values compared with AKBA films, with "L^*" values of 95.27 and 92.72, respectively, suggesting that BA has a lesser impact on the lightness of the films compared with AKBA.

The "a^*" value represents the red/green scale, where positive values indicate red and negative values indicate green color. The blank film (PC) had a near-zero "a^*" value (0.02), indicating a neutral color. The introduction of AKBA increases the "a^*" value significantly to 0.71 in PCA1 and 0.84 in PCA2, suggesting that AKBA imparts reddishness to the film. Films with BA also showed an increase in "a^*" values with PCT1 at 0.23 and PCT2 at 0.35. This indicates that both AKBA and BA shift the color toward red, but AKBA had a stronger effect. The "b^*" value measures the shift along the blue-yellow spectrum and the blank film (PC) had a low "b^*" value of 0.65. The addition of AKBA increased this value to 2.31 in PCA1 and further to 2.97 in PCA2, indicating that AKBA contributes to a yellowish hue in the films. BA also increased the "b^*" values, with PCT1 at 2.22 and PCT2 at 3.97, showing that both compounds contribute to a yellow shift, with BA having a stronger effect at higher concentrations.

The ΔE^* value represents the overall color difference compared to the blank film (PC). The blank film had the lowest ΔE^* at 0.74. As AKBA concentration increases, ΔE^* rises significantly to 4.81 in PCA1 and 12.47 in PCA2, indicating a substantial overall color change. Films with BA showed moderate ΔE^* values of 2.32 in PCT1 and 4.85 in PCT2, reflecting noticeable but less pronounced color changes compared with AKBA. This suggests that AKBA induces more significant color alterations in the films than BA, likely due to stronger pigmentation or interaction with the film matrix. Previous studies have also concluded similar findings in which the incorporation of different ingredients caused an increase in the color variation of the resultant films [31, 34, 35].

3.4. XRD Analysis. The XRD pattern for the control sample (PC) shows a broad peak centered around 20° 2θ (Figure 1). This broad peak indicates that the control film lacks any significant crystalline structure. Both PCA1 and PCA2 show more defined peaks compared to the control, indicating the presence of crystalline phases. The sharper peaks suggest the incorporation of AKBA has led to some degree of crystallinity in the films. The % crystallinity values for the PCA1 and PCA2 samples were 26.5% and 23.5%, respectively. The patterns for PCT1 and PCT2 also showed defined peaks, similar to PCA1 and PCA2, indicating the presence of crystalline phases. The PCT1 and PCT2 exhibited crystallinity values of 16.2% and 13.2%, respectively. These findings suggest that AKBA and BA contributes to the crystallinity of the film. The control sample (PC) was predominantly

Table 4: Opacity and color attributes of films.

Samples	Opacity	L^*	a^*	b^*	ΔE^*
PC	1.50 ± 0.06^{a}	96.39 ± 0.02^{e}	0.02 ± 0.01^{a}	0.65 ± 0.03^{a}	0.74 ± 0.01^{a}
PCA1	13.56 ± 0.31^{d}	91.17 ± 0.83^{b}	0.71 ± 0.02^{d}	2.31 ± 0.19^{b}	4.81 ± 0.16^{c}
PCA2	17.11 ± 0.37^{e}	83.35 ± 0.78^{a}	0.84 ± 0.01^{e}	2.97 ± 0.07^{c}	12.47 ± 0.07^{d}
PCT1	6.47 ± 0.24^{b}	95.27 ± 0.19^{d}	0.23 ± 0.01^{b}	2.22 ± 0.01^{b}	2.32 ± 0.20^{b}
PCT2	8.61 ± 0.12^{c}	92.72 ± 0.47^{c}	0.35 ± 0.0^{c}	3.97 ± 0.01^{d}	4.85 ± 0.04^{c}

Note: PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.

*The letters (a, b, c, d, and e) assigned to distinct values within a given column denote statistically significant differences ($p < 0.05$). "L" represents lightness, "a^*" indicates the green to red color range, "b^*" reflects the blue to yellow color range, and "ΔE^*" quantifies the overall change in color.

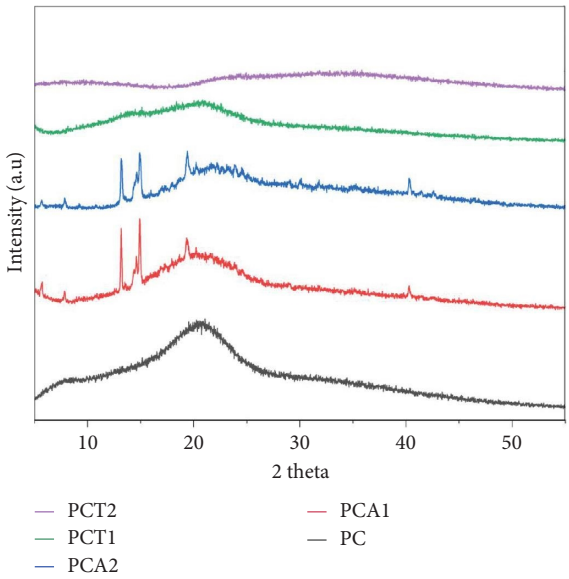


Figure 1: XRD analysis of the films. PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.

amorphous, while the addition of AKBA and BA introduces crystalline phases into the films, as indicated by the sharp peaks in the XRD patterns of PCA1, PCA2, PCT1, and PCT2. This suggests that both AKBA and BA promote the formation of crystalline structures within the films, which could influence the physical properties of the materials, such as their mechanical strength and stability. A previous study on organic acid-modified starch–CMC materials also reported the similar findings in which the hydrolysis of citric and acetic acids breaks chemical bonds, forming short-chain fragments that recrystallize into a quasicrystalline structure, increasing crystallinity [36].

3.5. FT-IR Spectroscopy. The FT-IR spectra demonstrated the transmittance profiles of the different film samples. The spectra exhibit distinct absorption bands corresponding to various functional groups present in the films (Figure 2). The PC spectrum, serving as the control, exhibits characteristic absorption bands, including those around 1034 cm^{-1}, which could be attributed to the C-O stretching vibrations, commonly seen in polysaccharide compounds like pectin. The spectra for PCA1 and PCA2 films display similar absorption bands to the blank film but with noticeable shifts and intensity changes in specific regions. For instance, a prominent peak around 1230 cm^{-1} indicating the interaction of AKBA with the polymer matrix. The spectra of PCT1 and PCT2 also demonstrate characteristic bands of both the polymer matrix and the active component (BA). Notable peaks include 1034 cm^{-1} (C-O stretching), similar to the blank, but with added intensity near 1230 cm^{-1} and 1640 cm^{-1}, suggesting enhanced interactions due to the presence of BA. A significant broad peak around 3310 cm^{-1} in PCT1 and PCT2 corresponds to the O-H stretching vibrations was observed, indicating the presence of hydroxyl groups, possibly from glycerol component. Similar results were observed in a previous study where the organic acid incorporated films showed shifting of bands associated with hydroxyl groups [36].

3.6. Water Contact Angle Measurements. The water contact angle measurements provided insights into the hydrophobic properties of the films and are crucial for understanding the surface characteristics of these materials (Figure 3). The control film (PC), which does not contain any active compounds, exhibited contact angles of 63.17° and 63.18°. These values serve as the baseline for comparison with other films. The films containing AKBA showed a noticeable increase in contact angles, reflecting an enhancement in hydrophobicity. Specifically, the PCA1 film, which contains 30 mg of AKBA, exhibited contact angles of 64.74° and 62.53°. The PCA2 film, with a higher concentration of 50 mg AKBA, demonstrated even greater contact angles of 72.83° and 71.88°. This trend suggests that the incorporation of AKBA, especially at higher concentrations, increases the hydrophobic nature of the film's surface. In contrast, the films containing BA showed varying results. The PCT1 film, which contains 30 mg of BA, had contact angles of 61.13° and 62.68°, slightly lower than the control film, indicating a lower hydrophobicity. On the other hand, the PCT2 film, with 50 mg of BA, displayed contact angles of 61.91° and 61.68°, which are also slightly lower than the control. The observed differences in contact angles suggest that the presence and concentration of AKBA significantly increase the hydrophobicity of the films, as reflected by the higher contact angles in PCA1 and PCA2. In contrast, BA at both tested

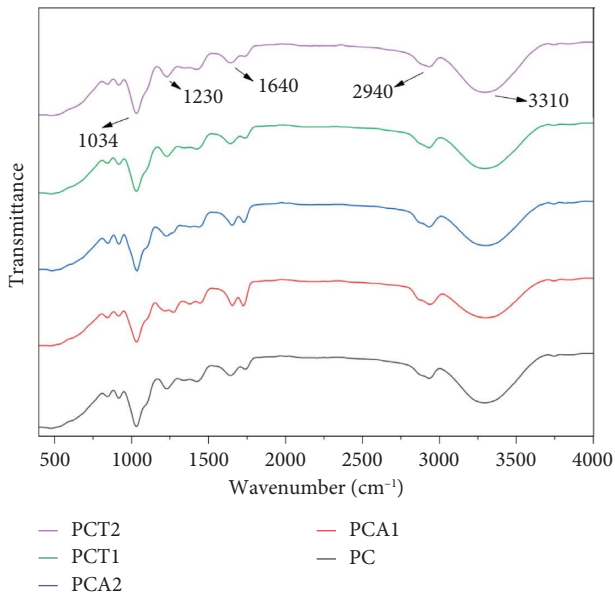


Figure 2: FTIR analysis of the films. PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.



Figure 3: Water contact angle measurements the films. PC is the blank film without active compounds. PCA1 and PCA2 contain 30 and 50 mg of AKBA, respectively. PCT1 and PCT2 contain 30 and 50 mg of BA, respectively.

concentrations does not significantly enhance hydrophobicity, as indicated by the lower or comparable contact angles to the control. Previous studies reported similar findings in which the addition of functional ingredients improves the hydrophobicity of the resultant films [37, 38].

4. Conclusion

The present study successfully demonstrated the incorporation of BA and AKBA into pectin and kappa-carrageenan-based films, aimed at enhancing their potential for

active packaging applications. The antioxidant properties of the films were significantly improved, making these films particularly suitable for packaging applications where oxidative stability is important. The increase in water contact angles, particularly in AKBA-loaded films, indicates enhanced hydrophobicity, which is beneficial for reducing moisture transmission in packaging applications. In conclusion, the integration of BA and AKBA into pectin and kappa-carrageenan films presents a viable strategy for developing active packaging materials with improved antioxidant and hydrophobic properties. In addition, future studies should address the release profile of BA and AKBA in food packaging and the risk of heavy metals or toxic metabolite leaching. This is crucial to ensure the safe use of BA and AKBA, given its complex composition and the evolving understanding of plant extract safety in food packaging applications.

Data Availability Statement

The data used to support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

Author Contributions

Yasir Abbas Shah and Saurabh Bhatia: conceptualization; Esra Koca and Levent Yurdaer Aydemir: formal analysis; Yasir Abbas Shah, Talha Shireen Khan, Tanveer Alam, Esra Koca, and Levent Yurdaer Aydemir: methodology; Yasir Abbas Shah and Esra Koca: software; Saurabh Bhatia and Ahmed Al-Harrasi: supervision; Saurabh Bhatia, Mohammad Tarahi, and Yasir Abbas Shah: wrote the original draft; Saurabh Bhatia, Yasir Abbas Shah, and Mohammad Tarahi: reviewing and editing.

Funding

There was no funding received for this work.

Acknowledgments

The authors are thankful to the Natural and Medical Sciences Research Center, University of Nizwa, Oman, for providing research facilities to conduct the current study.

References

[1] W. Y. Tong, A. R. Ahmad Rafiee, C. R. Leong, et al., "Development of Sodium Alginate-Pectin Biodegradable Active Food Packaging Film Containing Cinnamic Acid," *Chemosphere* 336 (2023): 139212, https://doi.org/10.1016/j.chemosphere.2023.139212.

[2] W. Zhang, S. Hedayati, M. Tarahi, et al., "Advances in Transglutaminase Cross-Linked Protein-Based Food Packaging Films; A Review," *International Journal of Biological Macromolecules* 253 (2023): 127399, https://doi.org/10.1016/j.ijbiomac.2023.127399.

[3] W. Zhang, M. Hadidi, A. C. Karaca, et al., "Chitosan-Grafted Phenolic Acids as an Efficient Biopolymer for Food Packaging Films/Coatings," *Carbohydrate Polymers* 314 (2023): 120901, https://doi.org/10.1016/j.carbpol.2023.120901.

[4] E. A. Günter, V. V. Martynov, B. A. Ananchenko, E. A. Martinson, and S. G. Litvinets, "The Gel Strength and Swelling in the Gastrointestinal Environment of Pectin/κ-Carrageenan Gel Particles Based on Pectins With Different Degrees of Methylesterification," *Materials Today Communications* 33 (2022): 104986, https://doi.org/10.1016/j.mtcomm.2022.104986.

[5] S. Popov, N. Paderin, D. Khramova, E. Kvashninova, A. Melekhin, and F. Vityazev, "Characterization and Biocompatibility Properties in Vitro of Gel Beads Based on the Pectin and κ-Carrageenan," *Marine Drugs* 20, no. 2 (2022): 94, https://doi.org/10.3390/md20020094.

[6] A. M. Roque, D. Montinola, L. Geonzon, et al., "Rheological Elucidation of the Viscoelastic Properties and Network Interaction of Mixed High-Methoxyl Pectin and Kappa-Carrageenan Gels," *Food Hydrocolloids* 129 (2022): 107647, https://doi.org/10.1016/j.foodhyd.2022.107647.

[7] E. A. Günter, V. V. Martynov, V. S. Belozerov, E. A. Martinson, and S. G. Litvinets, "Characterization and Swelling Properties of Composite Gel Microparticles Based on the Pectin and κ-Carrageenan," *International Journal of Biological Macromolecules* 164 (2020): 2232–2239, https://doi.org/10.1016/j.ijbiomac.2020.08.024.

[8] B. B. Sedayu, M. J. Cran, and S. W. Bigger, "A Review of Property Enhancement Techniques for Carrageenan-Based Films and Coatings," *Carbohydrate Polymers* 216 (2019): 287–302, https://doi.org/10.1016/j.carbpol.2019.04.021.

[9] K. Candogan and N. Kolsarici, "The Effects of Carrageenan and Pectin on Some Quality Characteristics of Low-Fat Beef Frankfurters," *Meat Science* 64, no. 2 (2003): 199–206, https://doi.org/10.1016/s0309-1740(02)00181-x.

[10] G. A. Paula, N. M. Benevides, A. P. Cunha, et al., "Development and Characterization of Edible Films From Mixtures of κ-Carrageenan, ι-Carrageenan, and Alginate," *Food Hydrocolloids* 47 (2015): 140–145, https://doi.org/10.1016/j.foodhyd.2015.01.004.

[11] J. Huang, Z. Hu, L. Hu, G. Li, Q. Yao, and Y. Hu, "Pectin-Based Active Packaging: A Critical Review on Preparation, Physical Properties and Novel Application in Food Preservation," *Trends in Food Science & Technology* 118 (2021): 167–178, https://doi.org/10.1016/j.tifs.2021.09.026.

[12] J. T. Martins, M. A. Cerqueira, A. I. Bourbon, A. C. Pinheiro, B. W. Souza, and A. A. Vicente, "Synergistic Effects Between κ-Carrageenan and Locust Bean Gum on Physicochemical Properties of Edible Films Made Thereof," *Food Hydrocolloids* 29, no. 2 (2012): 280–289, https://doi.org/10.1016/j.foodhyd.2012.03.004.

[13] D. Arltoft, F. Madsen, and R. Ipsen, "Relating the Microstructure of Pectin and Carrageenan in Dairy Desserts to Rheological and Sensory Characteristics," *Food Hydrocolloids* 22, no. 4 (2008): 660–673, https://doi.org/10.1016/j.foodhyd.2007.01.025.

[14] H. He, H. Bian, F. Xie, and L. Chen, "Different Effects of Pectin and κ-Carrageenan on the Multiscale Structures and In Vitro Digestibility of Extruded Rice Starch," *Food Hydrocolloids* 111 (2021): 106216, https://doi.org/10.1016/j.foodhyd.2020.106216.

[15] O. Jones, E. A. Decker, and D. J. McClements, "Thermal Analysis of β-Lactoglobulin Complexes With Pectins or

Carrageenan for Production of Stable Biopolymer Particles," *Food Hydrocolloids* 24, no. 2-3 (2010): 239–248, https://doi.org/10.1016/j.foodhyd.2009.10.001.

[16] N. K. Roy, D. Parama, K. Banik, et al., "An Update on Pharmacological Potential of Boswellic Acids Against Chronic Diseases," *International Journal of Molecular Sciences* 20, no. 17 (2019): 4101, https://doi.org/10.3390/ijms20174101.

[17] U. Varia, D. Joshi, M. Jadeja, H. Katariya, K. Detholia, and V. Soni, "Development and Evaluation of Ultradeformable Vesicles Loaded Transdermal Film of Boswellic Acid," *Future Journal of Pharmaceutical Sciences* 8 (2022): 39–16, https://doi.org/10.1186/s43094-022-00428-2.

[18] P. Jaroš, E. Timkina, J. Michailidu, et al., "Boswellic Acids as Effective Antibacterial Antibiofilm Agents," *Molecules* 27, no. 12 (2022): 3795, https://doi.org/10.3390/molecules27123795.

[19] R. Reshmy, E. Philip, P. Vaisakh, et al., "Development of an Eco-Friendly Biodegradable Plastic From Jack Fruit Peel Cellulose With Different Plasticizers and Boswellia Serrata as Filler," *Science of the Total Environment* 767 (2021): 144285, https://doi.org/10.1016/j.scitotenv.2020.144285.

[20] A. Khan, A. Al-Harrasi, N. U. Rehman, et al., "Loading AKBA on Surface of Silver Nanoparticles to Improve Their Sedative-Hypnotic and Anti-Inflammatory Efficacies," *Nanomedicine* 14, no. 21 (2019): 2783–2798, https://doi.org/10.2217/nnm-2019-0211.

[21] S. Bhatia, Y. A. Shah, A. Al-Harrasi, et al., "A Novel Film Based on a Cellulose/Sodium Alginate/Gelatin Composite Activated With an Ethanolic Fraction of Boswellia Sacra Oleo Gum Resin," *Food Science and Nutrition* 12, no. 2 (2023): 1056–1066, https://doi.org/10.1002/fsn3.3819.

[22] Astm, "Standard Test Methods for Tensile Properties of Thin Plastic Sheeting, Method D882-10," https://doi.org/10.1520/D0882-10.

[23] J. Zhao, Y. Wang, and C. Liu, "Film Transparency and Opacity Measurements," *Food Analytical Methods* 15, no. 10 (2022): 2840–2846, https://doi.org/10.1007/s12161-022-02343-x.

[24] R. Re, N. Pellegrini, A. Proteggente, A. Pannala, M. Yang, and C. Rice-Evans, "Antioxidant Activity Applying an Improved ABTS Radical Cation Decolorization Assay," *Free Radical Biology and Medicine* 26, no. 9-10 (1999): 1231–1237, https://doi.org/10.1016/s0891-5849(98)00315-3.

[25] W. Brand-Williams, M.-E. Cuvelier, and C. Berset, "Use of a Free Radical Method to Evaluate Antioxidant Activity," *Lebensmittel-Wissenschaft & Technologie Food Science and Technology* 28, no. 1 (1995): 25–30, https://doi.org/10.1016/s0023-6438(95)80008-5.

[26] O. Skurtys, C. Acevedo, F. Pedreschi, J. Enrions, F. Osorio, and J. Aquilera, "Food Hydrocolloid Edible Films and Coating," (2011), https://ialimentoslem1.wordpress.com/wp-content/uploads/2013/12/food-hydrocolloid-edible-films-and-coatings.pdf.

[27] Y. J. Kim, H. M. Lee, and O. O. Park, "Processabilities and Mechanical Properties of Surlyn-Treated Starch/LDPE Blends," *Polymer Engineering & Science* 35, no. 20 (1995): 1652–1657, https://doi.org/10.1002/pen.760352012.

[28] S.-D. Yoon, "Cross-Linked Potato Starch-Based Blend Films Using Ascorbic Acid as a Plasticizer," *Journal of Agricultural and Food Chemistry* 62, no. 8 (2014): 1755–1764, https://doi.org/10.1021/jf4024855.

[29] N. Cao, Y. Fu, and J. He, "Mechanical Properties of Gelatin Films Cross-Linked, Respectively, by Ferulic Acid and Tannin Acid," *Food Hydrocolloids* 21, no. 4 (2007): 575–584, https://doi.org/10.1016/j.foodhyd.2006.07.001.

[30] S. D. Yoon, S. H. Chough, and H. R. Park, "Effects of Additives With Different Functional Groups on the Physical Properties of Starch/PVA Blend Film," *Journal of Applied Polymer Science* 100, no. 5 (2006): 3733–3740, https://doi.org/10.1002/app.23303.

[31] S. Bhatia, Y. A. Shah, A. Al-Harrasi, et al., "Pectin/Sodium Alginate Films Tailored With Acetyl-11-Keto-Beta-Boswellic Acid for Active Packaging," *International Journal of Biological Macromolecules* 261 (2024): 129698, https://doi.org/10.1016/j.ijbiomac.2024.129698.

[32] H. Hosseinzadeh, S. Ebrahimpour, M. Fazeli, S. Mehri, and M. Taherianfard, "Boswellic Acid Improves Cognitive Function in a Rat Model Through Its Antioxidant Activity:-Neuroprotective Effect of Boswellic Acid," *Journal of Pharmacopuncture* 20, no. 1 (2017): 10–17, https://doi.org/10.3831/kpi.2017.20.001.

[33] M. Gupta, S. Singh, A. kurmi, et al., "Correlation of Boswellic Acids With Antiproliferative, Antioxidant and Antimicrobial Activities of Topographically Collected Boswellia Serrata Oleo-Gum-Resin," *Phytomedicine* 2, no. 3 (2022): 100289, https://doi.org/10.1016/j.phyplu.2022.100289.

[34] M. d. Rocha, M. R. Loiko, E. C. Tondo, and C. Prentice, "Physical, Mechanical and Antimicrobial Properties of Argentine Anchovy (Engraulis Anchoita) Protein Films Incorporated With Organic Acids," *Food Hydrocolloids* 37 (2014): 213–220, https://doi.org/10.1016/j.foodhyd.2013.10.017.

[35] S. Bhatia, A. Al-Harrasi, Y. A. Shah, et al., "Physicochemical Characterization and Antioxidant Properties of Chitosan and Sodium Alginate Based Films Incorporated With Ficus Extract," *Polymers* 15, no. 5 (2023): 1215, https://doi.org/10.3390/polym15051215.

[36] P. Kaur, T. Alam, H. Singh, J. Jain, G. Singh, and A. Broadway, "Organic Acids Modified Starch–CMC Based Biodegradable Film: Antibacterial Activity, Morphological, Structural, Thermal, and Crystalline Properties," *Journal of Pure and Applied Microbiology* 17, no. 1 (2023): 241–257, https://doi.org/10.22207/jpam.17.1.14.

[37] Q. Chen, Y. Shi, G. Chen, and M. Cai, "Enhanced Mechanical and Hydrophobic Properties of Composite Cassava Starch Films With Stearic Acid Modified MCC (Microcrystalline Cellulose)/NCC (Nanocellulose) as Strength Agent," *International Journal of Biological Macromolecules* 142 (2020): 846–854, https://doi.org/10.1016/j.ijbiomac.2019.10.024.

[38] R. Corcoran and E. Vernersson, "Creating a Hydrophobic Barrier Film by Modifying Carrageenan With a Hydrophobic Compound" (2023).