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Research Article

Enhancing water barrier and gas permeability of composite nano-coated biofilms for postharvest mango preservation

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Abstract

This research developed a biofilm integrating poly lactic acid (PLA) with a composite hydrophobic nanoemulsion (C-HyDEN) to evaluate their effectiveness in water barrier properties, gas permeability, and impact on the firmness of postharvest mango. We analysed Chitosan-HyDEN and Konjac-HyDEN films for water uptake (WU), water vapor permeability (WVP), diffusion coefficient, and gas permeability. We compared the best performance of composite coating to a single hydrophobic nanoemulsion (HyDEN) film in terms of mango firmness. Chitosan-HyDEN film demonstrated a significantly lower WU (1.06%) than Konjac-HyDEN film (2.14%). The Chitosan-HyDEN film exhibited lower WVP than the Konjac-HyDEN film, indicating its superior nanofiltration capabilities. Chitosan-HyDEN film had a higher diffusion coefficient than Konjac-HyDEN film, underscoring its effectiveness in reducing fruit water loss during storage. Composite film also proved more effective in controlling gas permeability than single HyDEN film. Due to its outstanding water barrier properties, Chitosan-HyDEN film was selected for firmness testing on postharvest mangoes, where it achieved the highest firmness retention (806.08±3.59 N) compared to single HyDEN film (614.26±3.55 N) and the commercial fungicide Antracol (174.75±5.35 N). These results indicate that Chitosan-HyDEN significantly enhanced the film's properties and effectiveness, thereby improving fruit products' storage life, safety, and quality.

Keywords: composite hydrophobic nanocoating, chitosan, konjac, antracol

Introduction

Postharvest loss in mangoes is a major problem for the worldwide food sector, resulting in large financial losses [1]. One of the most difficult issues in preserving mango quality throughout storage is the insufficient barrier qualities of traditional packaging materials [2]. These materials frequently fail to adequately prevent moisture loss and oxygen uptake, which can hasten fruit decay. Unfortunately, many bio-based films have intrinsic limitations regarding water and gas barrier performance [3]. In addition, green solvents such as hydrophobic deep eutectic

solvents (HDES) act as plasticisers to improve film flexibility and processability [4]. HDES have a broad range of applications due to their hydrophobicity and strong solvation capabilities for organic and inorganic molecules [5]. Combinations of two substances at a temperature below melting point are appropriate for extracting non-polar compounds because they are immiscible in water [6]. Toxic chemical solvents or pricy hydrophobic ionic liquids can be substituted with eco-friendly HDES, which have a high extraction efficiency for non-polar analytes [7]. Researchers are interested in polylactic acid (PLA) because of their

modulus and strength [8]. PLA is a top biomaterial with a broad range of medical applications that can polymers conventional replace based petrochemicals in industrial applications [9]. Starch and sugar are examples of renewable materials used to make PLA [10]. PLA is an element of biomaterials known as aliphatic polyesters, poly-a-hydroxy acids poly-a-esters. Lactic Acid hydroxypropanoic acid) is the water-soluble monomer used in its production. It exists in two enantiomeric forms: L-(+)-LA and D-(-)-LA [11].

Chitosan is a biodegradable copolymer compromising the linear polysaccharide of D-glucosamine and N-acetyl glucosamine [12]. The second most prevalent type of carbohydrate in nature is Chitosan [13]. It has been demonstrated to be advantageous, safe, biodegradable and biocompatible [14]. Chitosan is produced by Chitin deacetylation which contains the structural component of crustacean exoskeleton, like crabs and shrimps [15]. Chitosan is a deacetylated byproduct of chitin β -(1-4)-2-acetamido-2-deoxy-D-glucan [16]. Chitosan and chitin are comparable in that they are the most stable alkali and anti-acid compounds and this portion is also insoluble in common solvents [17].

Meanwhile, Konjac glucomannan (KGM), a linear polysaccharide derived from the konjac plant, has garnered significant attention for its potential applications in the food industry due to its exceptional water-holding capacity and gel-forming properties [18]. It consists of linear chains joined by units of mannose and glucose, and mixing KGM with water creates a thick gel, which is well-known for its remarkable ability to retain water [19]. KGM is a flexible polysaccharide that has shown promise in film development because of its ability to retain water [20]. KGM is a versatile chemical with biocompatibility applications because it is safe for human consumption [21].

Bio-based films, while environmentally friendly, frequently experience a variety of problems that limit their application. Common disadvantages include excessive water absorption and solubility [22], sensitivity to humidity [23], and poor barrier characteristics [24]. The application of composite nanocoating to improve the water and gas barrier properties of a bio-based film based on Chitosan and Konjac has not gone into great depth on this strategy. The importance of this study goes beyond a novel packaging material. This work can help reduce postharvest losses and improve food security by presenting sustainable and effective strategy for preserving mango quality.

Materials and Methods Materials

Menthol (purity >99%, CAS No: 2216-51-5), thymol (purity >99%, CAS No: 89-83-8), and Chitosan (CAS No: 9012-76-4) were procured from Take It Global Sdn. Bhd., Malaysia. Konjac glucomannan (CAS No: 37220-17-0) was sourced from the Institut Teknologi Sepuluh November (ITS) in Surabaya, Indonesia. Tween 80 (CAS No: 9005-65-6) was obtained from Sigma Aldrich Sdn. Bhd., Malaysia. The fungicide (70% Bayer Antracol) was purchased from a local market. 35 'Harumanis' mangoes, with a maturity index of 2 and free from disease or injury, were acquired from YA Mangga Harumanis Perlis, Malaysia, in compliance with the Federal Agricultural Marketing Authority Malaysia (FAMA) standards. Before the experiments, the 'Harumanis' mangoes were thoroughly washed with tap water and distilled water to remove any dirt or contaminants.

Design of experiment

HDES was prepared by combining menthol and thym ol in a 1:1 ratio, following the method described by G idado et al. [25]. The solid mixture was placed in a un iversal bottle, then magnetically stirred and heated at 60 °C using a hot plate stirrer (C-MAG HS 7, IKA, G ermany) for 15 min until a homogenous solution was obtained. To prepare the composite HyDEN, HDES, Tween 80 (surfactant), and chitosan/konjac were mix ed in an aqueous solution and magnetically stirred un til fully combined. The mixture consisted of 10% wt. HDES, 10% wt. Tween 80, 75% wt. aqueous phase (10 mM sodium phosphate, pH 7.0), and 5% wt. chito san/konjac, which were blended to form an oil-in-wat er nanoemulsion. Each concentration was then sonica ted for 15 min to ensure uniformity.

Table 1 presents the physicochemical properties of composite hydrophobic nanocoating.

Water uptake (WU)

The water uptake of Chitosan-HyDEN and Konjac-HyDEN films was evaluated by preparing films with varying concentrations (1%, 2%, 5%, and 7%) and incorporating PLA. Pure PLA films were used as controls. The initial weight of each film (mi) was recorded before immersing them in distilled water at ambient temperature. After 24 h, the films were removed, and their weight (mt) was measured. This process was repeated over 14 days, with weights measured at 24 h intervals. The percentage of water uptake (WU) was calculated using the equation provided by Petchwattana et al. [26]:

Table 1. Physicochemical properties of composite hydrophobic coating

	Menthol	Thymol	Chitosan	Konjac	Tween 80	Sodium phosphate
Chemical formula	$C_{10}H_{20}O$	$C_{10}H_{14}O$	$[C_6H_{11}NO_4]_n$	$C_{24}H_{42}O_{21}$	$C_{64}H_{124}O_{26}$	Na ₃ PO ₇
Molar mass (g mol-1)	156.27	150.22	161.16	666.6	1310	163.94
Melting point (°C)	34–36	48-51	103	152-156	-25	1583
Density (g/cm ³)	0.89	0.965	1.0	1.471	1.07 at 25 °C	1.62 at 20 °C

$$WU(\%) = \frac{mt-mi}{mi} \times 100$$
 (Eq. 1)

Where, m_i and m_t depicted the initial weight and the weight of the sample at time interval.

Water vapour permeability (WVP)

The water vapour permeability of Chitosan-HyDEN and Konjac-HyDEN films was determined using the method described by Gidado et al. [27], following the standardised ASTM E96 desiccant method. PLA was incorporated into Chitosan-HyDEN and Konjac-HyDEN solutions at varying concentrations (1%, 2%, 5%, and 7%) to form films, with pure PLA films serving as controls. The films were cut to fit the diameter of test vials and placed on top of vials containing 10 g of silica gel as a desiccant, then sealed with parafilm. Initial weights were recorded, and the vials were placed in a desiccator. The weight of the test vials was observed at hourly intervals over time, and water vapour permeability (WVP) was calculated using the following equation:

$$WVP = \frac{(\Delta m \times T)}{(A \times t \times \Delta P)}$$
 (Eq. 2)

Where, Δm = increased weight of test cell (g), T = film thickness (mm), A = exposed area of film (m²), t = transmission time interval (hour), ΔP = differential vapour pressure of water through film (kPa).

Diffusion coefficient

According to Gidado et al. [27], the moisture diffusion through the coatings was determined using Fick's law of diffusion using the following equations:

MR =
$$\frac{Mt-Me}{Mo-Me} = \frac{8}{\pi^2} exp \frac{(-\pi^2 Dt)}{4L^2}$$
 (Eq. 3)

MR = moisture ratio, D = effective diffusion coefficient (m^2/s), t = time (s), and L = thickness (mm) of sample layer in drying chamber. Mt = moisture level at time interval (t), Me = initial moisture, Mo = moisture at equilibrium. The diffusion coefficient can be calculated from the slope of the line (\propto) drawn between ln (MR) and time as:

$$\alpha = \frac{(\pi^2 Dt)}{4I^2}$$
 (Eq. 4)

 M_t = water uptake at immersion time (t), M_{sat} = saturation point, the k and n were estimated from the intercept and slope of the log plot of M_t/M_{sat} against time [28].

Gas permeability test

The gas permeability test was conducted using a constant pressure method with permeation equipment, as described by Thakur et al. [29], with slight modifications. Films incorporated with HyDEN, Chitosan-HyDEN, and Konjac-HyDEN were placed in a 1 L hermetic glass jar equipped with a septum and a lid for sampling. After 1 h at 20 ± 2 °C, the jars were stored at ambient temperature, and gas sampling was performed using a needle probe. A 5 mL gas sample was then injected into a thermal conductivity gas chromatograph (Gow-Mac 580, Bridgewater, USA) fitted with a stainless steel Haysep N column (80-100 mesh, 60 cm x 1 mm ID, Altech, Australia). The gas permeability was calculated as mL CO₂ produced per kg/h.

Firmness

The optimal bio-film was selected to evaluate the firmness of packaged mango fruits, comparing the results with HyDEN, Antracol, and a control treatment. Following the method described by Gidado et al. [27], the texture of the mango fruits was assessed using a texture analyser (TA XT Plus, Stable Micro Systems, Godalming, Surrey, UK). Mangoes were treated with four different types of coatings at Day 0 and was stored for 21. The firmness of the mangoes was measured at 7 days intervals of storage. Three mango fruits from each treatment and the control group were randomly selected for analysis. A 2 mm (P/2) probe was employed at a continuous speed of 3 mm/s, with measurements taken from various parts of the fruit.

Statistical analysis

The data were shown as the mean \pm the standard deviation. Measurements were conducted in triplicate, and the statistical analysis was performed using Microsoft Excel 2019. The study used one-way ANOVA with a significance threshold of p < 0.05.

Results and Discussion Water uptake

Water uptake (WU) in bio-films is a critical factor in assessing their initial performance. Understanding the water uptake properties is essential for evaluating bio-films' effectiveness in controlling moisture movement in fruits. **Figure 1** illustrates the percentage of water uptake for the prepared films at varying concentrations 14 days.

As shown in Figure 1, the percentage of water uptake after 14 days varied depending on the concentration of the bio-film components. The control, PLA, exhibited a water uptake of -4.32%. In comparison, Chitosan-HyDEN films at concentrations of 2%, 5%, and 7% showed significantly higher water uptake rates of 28.34%, 11.36%. and 56.42%, respectively. Meanwhile, Konjac-HyDEN films demonstrated lower water uptake, with values of 2.14%, 7.29%, and 54.75% at the same concentrations. Notably, Chitosan-HyDEN films had a lower water uptake at 2% concentration (1.06%) than Konjac-HyDEN films (2.14%). The increased pigment volume concentration in coatings typically enhances water absorption, although the highest values are observed near the critical concentration. At lower concentrations, water absorption is a slower process influenced by both water diffusion and structural changes [30].

Water vapour permeability (WVP)

Water vapour permeability (WVP) is an important component to include in the study because it has a direct impact on its resistance to moisture absorption and condensation [31]. Figure 2 illustrates the water permeability of the prepared films over a 7 h period. The water permeability of PLA/Chitosan-HyDEN films at concentrations of 1%, 2%, 5%, and 7% was consistently lower than that of PLA/Konjac-HyDEN films at all concentrations. In contrast, PLA films exhibited significantly higher permeability than the other treatments. The increased permeability is

attributed to enhanced microporosity and inner-pore interconnectivity in nanofiltration membranes, which can lead to rejection inefficiencies and selection defects [32]. The effect of various compatibilizers on WVP in PLA/Chitosan blends was discovered, and increased compatibility could reduce WVP [33].

Diffusion coefficient

The diffusion coefficient analysis is an important part of understanding the material qualities since it gives significant information about the rate at which molecules travel through it [34]. Figure 3 shows the diffusion coefficient of the prepared films. PLA as control showed 0.8 m²/s while Chitosan-HyDEN films for 1%, 2%, 5% and 7% showed 1.8 m²/s, 2.9 m²/s, 3.5 m²/s and 4 m²/s respectively. As for Konjac-HyDEN films shown 1.4 m²/s, 2 m²/s, 2.85 m²/s and 3 m²/s for the coatings of 1%, 2%, 5% and 7% respectively. The higher the diffusion coefficient of different types of films, the higher the water vapour permeability. Molecular diffusion coefficients were estimated using neural networks and multiple linear regressions [35].

Gas permeability test

The gas permeability test directly influences its ability to tolerate the passage of gases, which is critical in various applications such as packaging and filtration [36]. **Table 2** demonstrates the gas permeability test against different types of films. HyDEN film showed 0.58 of O₂/CO₂ selectivity, while Chitosan-HyDEN and Konjac-HyDEN films showed 1.04 and 1.91 of O₂/CO₂ selectivity, respectively. Films incorporated with composite coating show much better selectivity compared to single coating because the porosity of the tissue dictates effective O₂ diffusivity in film, with changes in membrane microstructure across cell membrane influencing gas exchange capabilities.

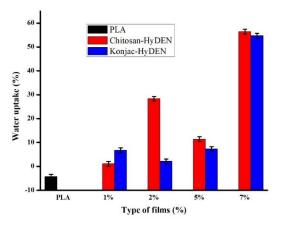


Figure 1. Water uptake of the prepared films (1%, 2%,5% and 7%)

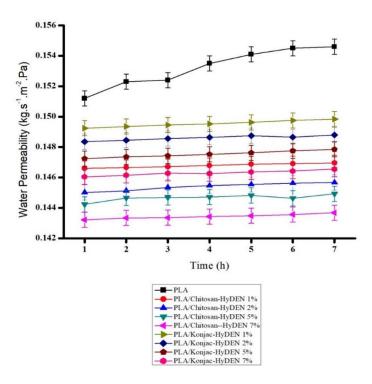


Figure 2. Water vapour permeability of the prepared films (1%, 2%, 5% and 7%)

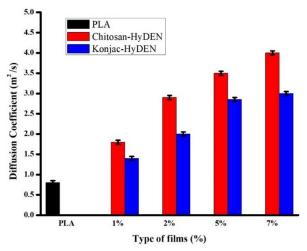


Figure 3. Diffusion coefficient of composite coatings (1%,2%,5% and 7%)

Firmness

Firmness analysis is an important part of evaluating material quality and texture since it has a direct impact on overall acceptability [37]. **Figure 4** illustrates the firmness of various treatments after the mango coating at Day 0. Firmness was evaluated over the storage period. Initially, the firmness of mangoes coated with Chitosan-HyDEN films, HyDEN films, Antracol, and control films were 13.51±1.51 N, 11.98±2.72 N, 10.85±3.14 N, and 10.58±4.18 N, respectively. By

day 21 of storage, the firmness had decreased to 806.08±3.59 N, 614.26±3.55 N, 174.75±5.35 N, and 2.69±0.93 N, respectively. These results indicate that Chitosan-HyDEN films maintained the highest firmness compared to the other treatments, particularly when compared to HyDEN films alone. The use of Chitosan and composite coatings in biobased films effectively preserved the firmness of mango fruits, aligning with the findings of Xing et al. [38].

Membrane Sample	Gas	Permeability,	O ₂ /CO ₂
		Barrer	Selectivity
HyDEN films	O_2	14.29	0.58
	CO_2	24.63	
Chitosan-HyDEN films	O_2	2515.83	1.04
-	CO_2	2423.83	
Konjac-HyDEN films	O_2	21191.25	1.91
	CO_2	11070.06	

Table 2. Gas permeability values of different types of films

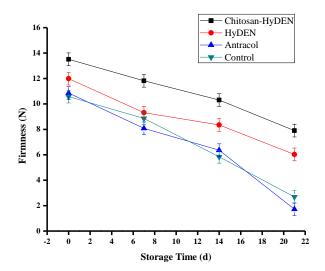


Figure 4. Firmness (N) of mango fruits treated with different types of films over a 21-day storage period

Conclusion

This study created a biofilm that combines poly lactic (PLA) and a composite hydrophobic nanoemulsion (C-HyDEN) to assess effectiveness regarding water barrier characteristics, gas permeability, and effects on postharvest mango firmness. We tested Chitosan-HyDEN and Konjac-HyDEN films for water uptake (WU), water vapor permeability (WVP), diffusion coefficient, and gas permeability. We compared the optimum performance of composite coating to a single hydrophobic nanoemulsion (HyDEN) film in terms of mango firmness. Chitosan-HyDEN film has a substantially lower WU than Konjac-HyDEN film. Chitosan-HyDEN film had lower WVP than Konjac-HyDEN film, showing stronger nanofiltration capabilities. Furthermore, Chitosan-HyDEN film showed a greater diffusion coefficient than Konjac-HyDEN film, demonstrating its efficacy in decreasing fruit water loss during storage. Composite films were also more effective at controlling gas permeability than single HyDEN films. Chitosan-HyDEN film was chosen for postharvest mango firmness testing due to its exceptional water barrier capabilities and achieved the highest firmness retention compared to single HyDEN

film and the commercial fungicide Antracol. These findings show that Chitosan-HyDEN greatly improved the film's characteristics and effectiveness, increasing fruit items' storage life, safety, and quality. Future research may investigate the synergistic effects of combining biofilm coatings with other preservation techniques, such as modified atmosphere packaging (MAP) or low temperature storage, to achieve optimal shelf-life extension in postharvest mangoes.

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