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Effect of Different Drying Temperatures on the Physicochemical Properties of Sago Starch-Bacterial Cellulose Film Incorporated with Gunuang Omeh Orange Essential Oil

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Abstract. *Extensive and irresponsible use of conventional plastic has brought serious problems to the planet due to its low biodegradability. In order to reduce the risks, packaging materials made from biodegradable materials are extremely needed. This study develops active packaging films using sago starch and bacterial cellulose incorporated with Gunuang Omeh orange peel essential oil. It also evaluated the effect of different drying temperatures on the physicochemical, mechanical, structural, and antimicrobial properties of the resulting films. The solvent casting method was used to prepare sample films with 3 drying temperatures (40, 45, and 50°C). The functional properties and antibacterial activity against E. coli and S. aureus of films with and without essential oil were characterized and analyzed. The results showed that drying temperature significantly influences the performance of the biofilms. Higher tensile strength (2.38 MPa) and lower moisture absorption were found at 45°C dried films. The presence of essential oil slightly increased water solubility and improved antibacterial activity, with inhibition zones ranging from 7.70–15.77 mm against E. coli and 4.83–5.75 mm against S. aureus. In conclusion, sago starch–bacterial cellulose films incorporated with Gunuang Omeh orange essential oil demonstrate a future potential as eco-friendly packaging materials, with drying temperature identified as a critical processing parameter for optimizing functional performance.*

Keywords: *sago starch; bacterial cellulose; orange essential oil; drying temperature; biocomposite.*

Type of the Paper: Regular Article.

1. Introduction

Petroleum-based plastics waste has become an urgent problem for Indonesia to overcome, especially single-use plastic waste from synthetic materials. This problem has attracted the attention of the Indonesian government to create special regulations for handling plastic waste [1]. One use of synthetic plastic is in food packaging. The widespread and dominant use of this type of polymer in the food packaging industry brings a huge risk to global environmental sustainability [2]. To overcome this problem, researchers have developed eco-friendly food packaging from natural polymers.

One of the most promising natural polymers that can be used for food packaging development is starch [3,4]. Among all starch sources, sago starch is one of the most promising in Indonesia because it is abundantly available in many regions of Indonesia. In 2023, West Sumatra had a sago plantation area of 1,437 hectares with a production of 666 tons [5]. However, pure starch films have weaknesses, namely brittleness and hydrophilicity [6]. To overcome their natural drawbacks, starch

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can be combined with reinforcement such as cellulose. Among all cellulose sources, bacterial cellulose shows the highest compatibility with the starch matrix due to its high purity [7]. According to previous studies, starch-bacterial cellulose film represented good functional properties, including mechanical properties and low water absorption [8]. Moreover, bacterial cellulose also showed good compatibility with various starches, such as wheat and maize starch [9]. In order to improve the moisture and water resistance of starch-based films, essential oils can be used to protect the food. Essential oils also provide antimicrobial properties to the packaging film [10].

Furthermore, West Sumatra has a superior local citrus variety, namely Gunuang Omeh Siam orange (*Citrus nobilis* Lour.), which is well known for its quality and distinctive aroma. The peel of Gunuang Omeh orange can be extracted to get its essential oil. Essential oil from orange peel has been reported to contain limonene and has been successfully used as an additive in starch and cellulose solutions [11]. However, the volatile compounds in essential oils can easily evaporate when exposed to hot temperatures. In the case of developing essential oil-based films, the film drying temperature can make changes in the functional properties of the resulting film. So the optimal drying time needs to be studied to obtain a film with the best functional properties.

As far as we know, research about active film from starch and bacterial cellulose incorporated with essential oil from the local products of West Sumatra is still limited. The use of Gunuang Omeh orange peel essential oil on the biopolymer films was never studied before. Besides that, the previous report about the drying temperature effect on sago starch films was also limited. Whereas drying temperature is an important factor that can influence how much essential oil remains in the film after the drying process, as well as the functional properties of the films.

Based on this background, the aim of the study is to fabricate packaging films with sago starch and bacterial cellulose with the addition of Gunuang Omeh orange essential oil. The films will be dried under various drying temperatures in a ventilated oven. Functional properties of films, such as water solubility, moisture absorption, mechanical properties, and antimicrobial properties will be measured and evaluated. The results of this study can provide scientific contributions in the development of eco-friendly packaging films based on local agriculture product in West Sumatra.

2. Materials and Methods

2.1. Materials

Sago (*Metroxylon sagu*) starch was purchased from a traditional producer who processed it without chemicals. Fresh Gunuang Omeh orange peels (*Citrus nobilis* Lour.) were obtained from local farmers in Gunuang Omeh, West Sumatra. Bacterial cellulose (BC) pellicles were obtained from a home-based industry in Padang, West Sumatra. BC pellicles were boiled to remove bacterial residue. Boiled BC pellicles then dried in a ventilated oven at 50ºC for 1 day. After drying, BC was

ground to reduce its size and followed by sieving through a 100-mesh sieve to obtain fine particles. Distilled water and Tween 80 were brought from the local chemical store.

2.2. Film preparation

Orange peel essential oil (OPEO) emulsion was formulated using Abedi [12] method with little modifications. The OPEO and Tween 80 in a ratio of 1:1, namely 0.25 g OPEO and 0.25 g Tween 80, were mixed to 300 g of distilled water and then emulsified using a B-One Em-300D homogenizer for 10 minutes at 3000 rpm. Besides that, a total of 30 g of sago starch, 9 g of glycerol, and 0.24 g of BC powder were added to 300 mL of distilled water. The solution was heated on a hotplate with a magnetic stirrer at a speed of 600 rpm at 90 °C. The mixture was stirred until gelatinization occurred.

About 100 g of essential oil emulsion that had been prepared was mixed with starch-bacterial cellulose gelatinized solution. The mixture was homogenized using a B-One Em-300D homogenizer for 2 minutes until a homogeneous suspension was formed. After that, 100 g of film solution containing essential oil emulsion was poured into a 15 cm diameter petri dish. The casted solutions were then dried in a ventilated oven at 40 °C, 45 °C, and 50 °C for ±24 hours. Films without essential oil were also prepared for each drying temperature for comparison purposes. The samples were coded with their drying temperature for non-essential oil films and drying temperature + EO for samples with essential oil.

2.3. Water Solubility Test

Water solubility value was measured with the procedure described by Enwere et al [13], films were cut into 7 mm diameter circular samples. The samples were dried in an oven at 85 °C for 24 hours. The dried samples were then submerged in 50 mL of aquadest for 24 hours. Then, the water was removed from the surface of film, and it was dried again in at 85 °C for 24 hours. Water solubility, expressed as a percentage, was obtained using the Eq. (1).

$$\text{Water solubility (\%)} = \frac{\text{Starting mass} - \text{Final mass}}{\text{Starting mass}} \text{ x } 100\% \quad (1)$$

2.4. Moisture Absorption Test

Biocomposite films were cut into 7 mm diameter circular samples and then dried at 50 °C for 24 hours. The dried film samples were weighed to determine the starting dry mass before being placed in a closed container containing a saturated NaCl solution (75% relative humidity) at 25 °C ± 2 °C [14]. All specimens were then weighed every 30 minutes to get the final mass. The moisture absorption percentage was calculated using Eq. (2).

$$\text{Moisture absoprtion (\%)} = \frac{\text{Final mass} - \text{ Starting dry mass}}{\text{Starting dry mass}} \text{ x } 100\% \quad (2)$$

2.5. FTIR Spectra

The spectra of FTIR were recorded using a Thermo Fisher Nicolet iS5 instrument. The resulting films were analyzed with a wavenumber range of 4000–650 cm^{-1} at a resolution of 4 cm^{-1}.

2.6. Antimicrobial Test

Disk diffusion method was choose to obtain antibacterial properties of active films. Bacterial suspensions of *Staphylococcus aureus* and *Escherichia coli* were adjusted to a turbidity standard of 0.5 McFarland, then 100 µL was inoculated onto the surface of Mueller Hinton Agar (MHA) and evenly spread using a sterile spreader. Biocomposite film samples were cut into discs with a diameter of 7 mm, and the film discs were placed on the surface of the bacteria-inoculated medium. Iodine 10% was used as a positive control and films without essential oil addition as a negative control. Petri dishes were incubated at 37 °C for 24 hours. The clear inhibition zones formed around the film discs were measured using a caliper and classified based on inhibition zone diameter. Inhibition zones with diameters ≤ 5 mm were categorized as weak inhibition, 6–10 mm (moderate), 11–20 mm (strong), and ≥ 21 mm (very strong).

2.7. Mechanical Properties

Tensile strength, elongation at break, and elasticity modulus, were determined using a RTI-1310 Universal Testing Machine (A&D Company, Japan) according to ASTM D-882-91, with film specimens cut into strips of 15 × 65 mm and tested at a 5 mm/min crosshead speed.

2.8. Color

Color measurements were conducted using a portable CHNSpec CS-10 colorimeter under standard D65 lighting and an observation angle of 10°, and L*, a*, and b* values were recorded using the CIELab scale.

3. Results and Discussion

3.1. Water Solubility

The water solubility values at different drying temperatures, with and without essential oil addition, are presented in Fig. 1. The water solubility of the films ranged from 22.45% to 23.84%. The addition of essential oil at 2.5% caused a slight increase in water solubility compared to films without oil at all drying temperatures (40–50°C). The highest increase occurred at 50°C, reaching 1.14%. This likely occurred due to the essential oil is volatile during the drying process, leaving small pores in the film structure. These pores facilitate the entry of water molecules into the film matrix. Starch contains many hydroxyl (–OH) groups in its molecular structure that easily bind with water, causing starch-based films to readily absorb water [15]. Nevertheless, the differences in

drying temperatures did not have a significant effect on water solubility. This indicates that these temperatures are within a safe range and do not significantly affect the film's water resistance.

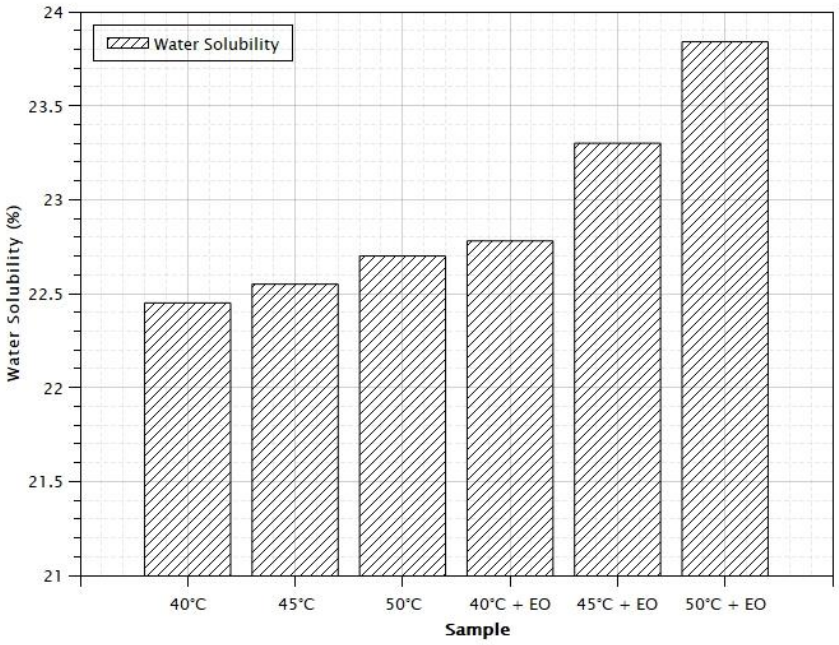

Fig. 1. Water solubility of resulting films

3.2. Moisture Absorption

The moisture absorption of the biocomposite films was presented in Fig. 2. The observation results showed that moisture absorption of the bioplastic film increased with increasing observation time from the beginning to the third hour, indicating a gradual diffusion of water molecules into the film matrix until approaching equilibrium conditions. This property is commonly found in starch-based films due to the hydrophilic hydroxyl (–OH) groups occurrence, they can bind water molecule to construct hydrogen bonds, facilitating the film absorb the moisture from the environment [16]. When comparing drying temperatures, films dried at higher temperatures showed lower moisture absorption, especially in the early stages of observation. This indicates that higher drying temperatures may strengthen the microstructure of the starch matrix, which limits the amount of water molecules that diffuse into the matrix [17]. Moreover, differences in drying temperature can affect the barrier characteristics of biopolymer films, as temperature variation can alter film morphology and matrix structure, thereby influencing barrier properties against water vapor [18]. Films with essential oil also showed lower moisture absorption compared to the control at the same drying temperature. The phenomenon was clearly represented at the 40°C film and the 40°C+EO film.

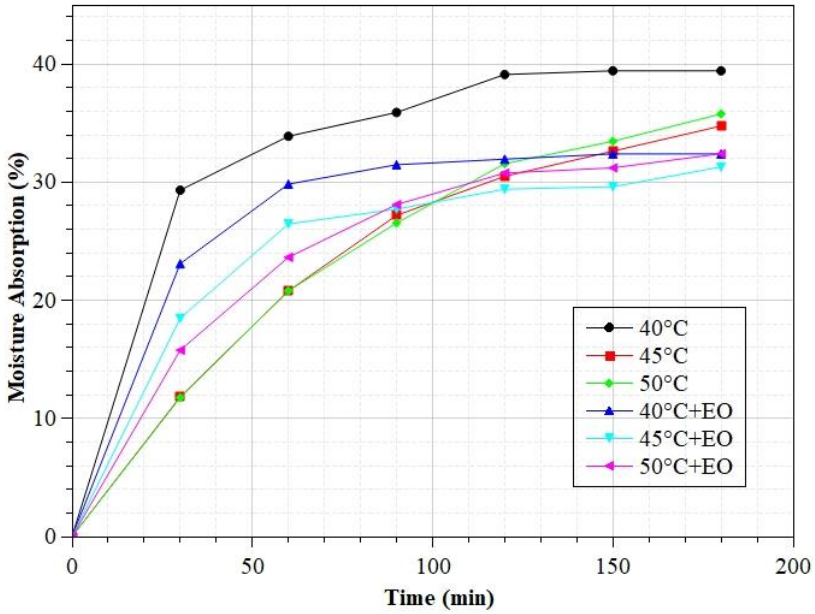

Fig. 2. Moisture absoroption of resulting films

3.3. FTIR Results

FTIR spectra of the resulting film are shown in Fig. 3. All films show similar peaks, which exhibit the profile of the sago starch and bacterial cellulose. Broad peaks at 3295–3296 cm^{-1} representing the O-H stretching. This peak came from hydroxyl groups from glucose units in starch and cellulose and indicated that the films tend to bind water molecules within the matrix. A peak appears around 2929–2931 cm^{-1}, corresponding to C–H stretching, originating from the C–H stretching vibrations of methyl and methylene groups on the polysaccharide chains. Additionally, a peak appears in the region of 1010–1016 cm^{-1} corresponding to a C–O stretching.

Differences in drying temperature (40, 45, and 50°C) result in slight changes in the wavenumber and intensity of peaks. The changes probably occurred because of differences in water evaporation rates during drying, driven by different drying temperatures. The sample dried at 50°C, which normally has the highest evaporation rate of water, showed minor shifting in wavenumber. It indicates the high evaporation rate caused minor changes in matrix density. This is consistent with research by Aguirre-Alvarez et al. [19], who examined the effect of drying temperature (20, 40, and 60°C) on gelatin films, found that at high temperatures (above 40°C), molecular motion in the film increases. This faster motion weakens intermolecular interactions (such as hydrogen bonds). This can cause changes in the FTIR spectrum results, as seen in the data in this study, especially at a drying temperature of 50°C.

The addition of Gunuang Omeh orange essential oil slightly modifies the FTIR spectra, indicating interactions between the OPEO components and the starch matrix. The C–O stretching peak remains detectable, although its position shifts slightly, such as to 1010.15 cm^{-1} in the

40°C+EO sample, demonstrating that the chemical environment of the C–O group changes due to interactions with the oil. This finding is consistent with Abedi et al [12], who reported that the incorporation of cinnamon essential oil into emulsion-based films resulted C–O stretching vibrations of the aldehyde functional group, a characteristic feature of cinnamaldehyde. The O–H stretching peak also experiences a minor shift, for example, from 3295.53 cm^{-1} (40°C) to 3289.67 cm^{-1} (40°C+EO), indicating the occurrence of hydrogen bonding between the polymer hydroxyl groups and polar components within the essential oil. This shows that the essential oil interacts not only physically but also chemically with the polymer matrix, potentially influencing the film's functional properties, such as moisture absorption and antibacterial activity.

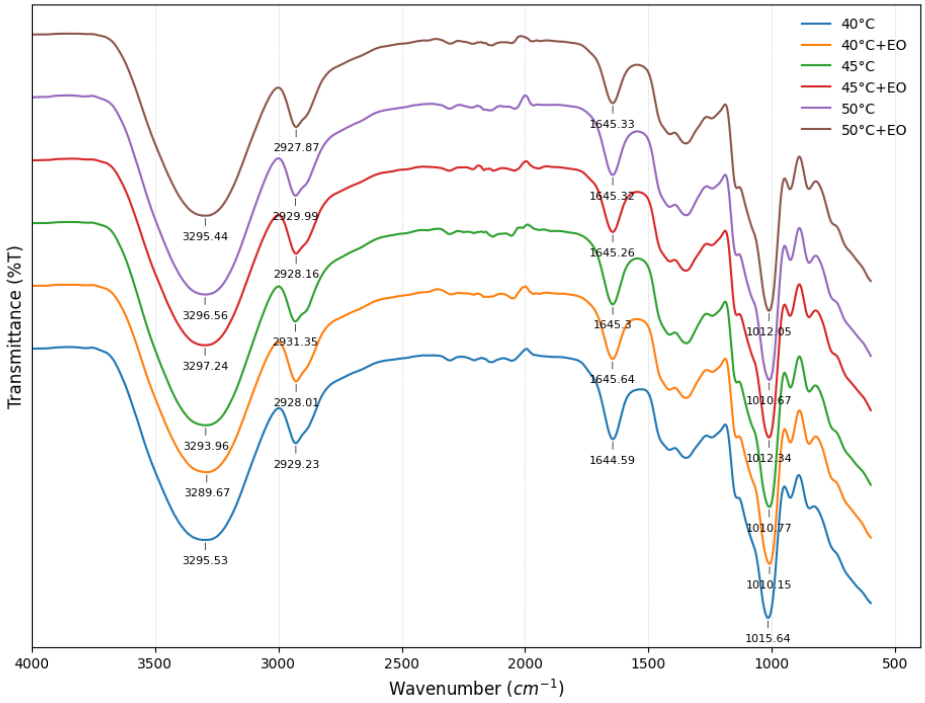

Fig. 3. Fourier Transform Infrared Spectroscopy

3.4. Antimicrobial Activity

The effects of drying temperatures and the addition of Gunuang Omeh OPEO on antibacterial activity were investigated, and the resulting inhibition zone diameters are shown in Table 1.

Table 1. Inhibition zone diameters of biocomposite films

Sample	Inhibitory Zone Diameter (mm)	
	E. coli	S. aureus
40ºC	5.10	3.72
45ºC	6.15	4.44
50ºC	8.27	4.88
40ºC + EO	7.70	4.83
45ºC + EO	10.70	5.65
50ºC + EO	15.77	5.75

In films without essential oil addition, differences in drying temperature (40, 45, and 50°C) showed an increase in antibacterial inhibition, especially against *E. coli*. The inhibition zone diameters were 5.10 mm, 6.15 mm, and 8.27 mm, respectively, all still in the moderate category. In contrast, against *Staphylococcus aureus*, the increase in temperature only increased the inhibition zone from 3.72 mm to 4.88 mm and remained categorized as weak. This indicates that the base film does not yet contain strong antibacterial components to combat Gram-positive bacteria. The addition of 0.25 g Gunuang Omeh orange essential oil significantly increased antibacterial activity. When tested with *E. coli*, the inhibition zone increased to 7.70 mm (40°C), 10.70 mm (45°C), and reached 15.77 mm (50°C), which falls into the strong category. This increase indicates that the essential oil acts as the main antibacterial agent in the active packaging system. Compounds such as limonene are hydrophobic and capable of damaging bacterial cell membranes, thereby increasing microbial growth inhibition effectiveness [20]. For *S. aureus*, EO addition also increased activity, but not as much as for *E. coli*. The maximum inhibition zone was only 5.75 mm (moderate category) at 50°C+EO. As reported by previous research, this difference has a correlation with the limonene mechanism as the major compound in the citrus essential oil. This chemical compound hase been proven to be able destroys the cellular integrity of *E. coli* by damaging the cell membrane, triggering the leakage of intracellular materials (proteins and nucleic acids), while disrupting the DNA transcription process [21]. On the other hand, the lower inhibitory power against S. aureus is strongly suspected due to the presence of a thick peptidoglycan layer and teichoic acid which make up its cell wall. This protective structure is believed to act as a physical shield that reduces the penetration efficiency of hydrophobic molecules from essential oils at the given dose [22].

3.5. Color

Color characteristics are an important quality attribute of bioplastic films, as they influence the visual appearance and consumer acceptance of packaging materials. In this study, the color values were evaluated using the CIE L*a*b* color system to determine the effects of different drying temperatures and the incorporation of Gunuang Omeh orange peel essential oil. The resulting L*, a*, and b* values of the resulting films, are presented in Table 2.

Table 2. Color values of biocomposite films

Sample	Color value		
	L*	a*	b*
40ºC	29.84	35.27	-7.05
45ºC	43.77	5.97	35.47
50ºC	46.13	6.71	34.05
40ºC + EO	35.30	36.81	-4.70
45ºC + EO	45.38	7.97	33.01
50ºC + EO	45.67	8.28	32.89

Color measurement results showed that drying temperature had a significant effect on the visual characteristics of the biocomposite film. The lightness value (L*) increased with increasing drying temperature from 40°C to 50°C, indicating that the film became brighter at higher temperatures. Meanwhile, the addition of orange peel essential oil at a concentration of 0.25 g showed a significant change in lightness value from 40°C to 40°C+EO, the value increased by about 18.30%. The increase in color value after adding essential oil was caused by the yellow color present in the essential oil [22].

3.6. Mechanical Properties

Mechanical properties are critical parameters for evaluating the performance and applicability of bioplastic films, as they represent the tensile strength, elasticity, and resistance to mechanical stress during handling and use. The effects of different drying temperatures and the presence of Gunuang Omeh orange essential oil on the tensile strength (TS), elongation at break (EB), and Young's modulus (TM) were measured. The mechanical properties of the resulting films are shown in Table 3.

Table 3. Mechanical properties of biocomposite films

Sample	Mechanical Properties		
	TS (MPa)	TM (MPa)	EB (%)
40ºC	1.67	124.24	2.40
45ºC	2.38	98.04	5.10
50ºC	2.24	123.50	3.76
40ºC+EO	2.02	102.40	3.63
45ºC+EO	2.34	101.80	3.97
50ºC+EO	1.98	99.99	3.66

For films without essential oil, drying temperature affected tensile strength. At 40°C, the film had a tensile strength of 1.6675 MPa, which was the lowest value among the treatments. Increasing the drying temperature to 45°C significantly increased tensile strength to 2.4435 MPa, indicating that this temperature provided the optimal drying condition for forming a strong film structure. Higher temperatures promote the formation of tighter intermolecular bonds between starch and bacterial cellulose, thereby increasing matrix cohesion. However, when the drying temperature was increased to 50°C, tensile strength slightly decreased to 2.3386 MPa. This is in line with the research of Qin et al. [21], who examined the effect of drying temperature on glucomannan films using temperatures of 40, 50, 60, 70, and 80°C, found that the best drying temperature in this study was 60°C, which means that when the drying temperature is above the optimal temperature, the mechanical properties of the film are reduced. Tensile strength may decrease with increasing drying temperature due to the breaking of chemical bonds in film molecules.

After the addition of 0.25 g of essential oil, changes in tensile strength occurred at all drying temperatures. At 40°C, tensile strength increased from 1.6675 MPa to 2.3842 MPa, but remained lower than the maximum tensile strength of films without oil at 45°C. Conversely, at 45°C, tensile

strength decreased from 2.4435 MPa to 2.0095 MPa, and at 50°C, it decreased from 2.3386 MPa to 1.9751 MPa. This reduction indicates that the essential oil tends to disrupt polymer chain interactions within the film matrix. Hydrophobic compounds in essential oil can reduce intermolecular attraction between starch and bacterial cellulose molecules, making the film more flexible but less resistant to tensile loads. The addition of essential oils can produce varied and even negative effects on film mechanical properties depending on the type and composition of the materials used [23]. Elongation at break of films without essential oil also depended on drying temperature. At 40°C, elongation was 2.3996%, the lowest value. When the drying temperature increased to 45°C, elongation increased to 3.7624%, and slightly increased again at 50°C to 3.9655%. Higher drying temperatures improve intermolecular interactions and matrix structural order, producing a more homogeneous morphology. This more uniform structure allows more even stress distribution during stretching, enabling the film to elongate further before breaking.

After adding 0.25 g essential oil, elongation increased significantly, especially at 40°C, from 2.3996% to 5.103%, which was the highest among all treatments. At 45°C, elongation slightly decreased from 3.7624% to 3.6274%, while at 50°C it slightly decreased from 3.9655% to 3.66%, but remained higher than films without oil at lower temperatures. Essential oil was incorporated as an emulsion using Tween 80, which may lead to physical instability and changes in droplet size and distribution during film formation. Emulsion system characteristics, such as emulsion type, droplet size, and emulsifier properties, can influence the structure, functionality, and performance of active films. Regarding elasticity, films without essential oil dried at 40°C had a Young's modulus of 124.24 MPa, and at 45°C the value was similar at 123.5 MPa. However, at 50°C the modulus decreased to 101.795 MPa. This decrease indicates that higher temperatures may cause internal structural changes in the film, such as micro-pore formation or reduced matrix uniformity due to excessively rapid water evaporation. Increased drying temperature has been reported to influence the structure and mechanical properties of biopolymer films, for example increasing the elasticity modulus up to an optimum limit due to internal reorganization of the film matrix, followed by a decrease at higher temperatures [24].

4. Conclusions

This study successfully developed biocomposite films based on sago starch and bacterial cellulose incorporated with Gunuang Omeh orange essential oil, highlighting the synergistic role of formulation composition and drying temperature in determining film performance. Drying temperature exerted a significant influence on film microstructure, intermolecular interactions, and functional properties, with 45°C identified as the optimal condition for forming a compact and homogeneous polymer matrix. This temperature promoted balanced mechanical strength (tensile

strength: 2.38 MPa), reduced water vapor absorption, and stable molecular interactions, as confirmed by FTIR spectral shifts and improved barrier characteristics.

The incorporation of orange essential oil effectively imparted antimicrobial functionality to the films, particularly against Escherichia coli, confirming its role as the primary bioactive component within the film matrix. However, the essential oil also altered polymer–polymer interactions, resulting in a trade-off between tensile strength and flexibility. Hydrophobic compounds in the essential oil reduced intermolecular attraction between starch and bacterial cellulose molecules, making the film more flexible but less resistant to tensile loads. These findings underscore the importance of precisely controlling drying conditions to minimize volatile compound loss and structural heterogeneity while preserving bioactivity.

Overall, this research provides a scientifically robust strategy for developing starch–cellulose–based active packaging using locally sourced materials from West Sumatra, contributing to sustainable packaging innovation and plastic waste reduction. Future research should focus on optimizing essential oil loading levels, enhancing emulsion stability through nanoemulsion technology, and evaluating real-food applications to comprehensively assess the industrial feasibility and functional performance of the developed packaging system.

Data availability statement

The availability of the data must be requested through the corresponding author.

CRediT authorship contribution statement

Maulana Yuda Anantama: Methodology, Investigation, Visualization, Formal Analysis, Writing – Original Draft, and Writing – Review and Editing. **Fadli Hafizulhaq:** Conceptualization, Data Curation, Supervision, Validation, Writing – Review and Editing. **Andasuryani Andasuryani:** Conceptualization, Supervision.

Declaration of Competing Interest

The authors stated no conflicts of interest regarding the research, authorship, or publication of this article.

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