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PHYSICOMECHANICAL PROPERTIES OF BIOPLASTICS FROM KAPPA-CARRAGEENAN AND CASSAVA PEEL STARCH

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Abstract

Kappa-carrageenan, derived from red seaweed, is well-known for its excellent film-forming properties and is widely used as a bioplastic material. It is possible to improve the physical and mechanical properties of bioplas-tics by mixing kappa-carrageenan with cassava peel starch and polyvinyl alcohol (PVA) as a reinforcement. The goal of this study is to find the best combination of kappa-carrageenan and cassava peel starch for bio-plastic based on its thickness, tensile strength, elongation, and water vapor transmission rate (WVTR), as specified in JIS Z 1707. A completely randomized block design (CRBD) was employed with varying ratios of kappa-carrageenan and cassava peel waste starch. The findings show that the mixtures of kappa-carrageenan and cassava peel starch had a big effect on all of the bioplastic's physical and mechanical properties, except for its density. All bioplastic formulations met the JIS standard for tensile strength and elongation. Higher starch con-centrations significantly improved the barrier properties by reducing WVTR and water absorption. However, increasing starch concentration enhanced elongation while decreasing thickness, tensile strength, and Young's modulus, resulting in slower biodegradation. The best mix, which had 4% starch and 1% kappa-carrageenan, was thickest at 0.35 mm, a tensile strength of 1.14 MPa, an elongation of 25.78%, and a WVTR of 18.47 g/m^2/day (Grade 3). The results show that kappa-carrageenan and waste starch from cassava peel can be used to make bioplastics that meet the standards for physical and mechanical properties. This could also help reduce plastic pollution in the future.

Keywords: barrier properties, biodegradable, mechanical properties, tensile strength, transparency

Sifat Fisikomekanik Bioplastik dari Campuran Kappa-Karagenan dan Pati Limbah Kulit Singkong

Abstrak

Kappa-karagenan yang berasal dari rumput laut merah dikenal bersifat membentuk film yang sangat baik dan banyak digunakan sebagai bahan bioplastik. Formulasi kappa-karagenan dan pati limbah kulit singkong yang diperkuat dengan polivinil alkohol (PVA) berpotensi menghasilkan bioplastik dengan sifat fisikomekanik yang lebih optimal. Penelitian ini bertujuan untuk menentukan formula terbaik kappa-karagenan dan pati limbah kulit singkong sebagai bioplastik berdasarkan sifat fisikomekaniknya yang meliputi ketebalan, kuat tarik, elongasi, dan laju transmisi uap air dengan mengacu pada standar JIS Z 1707. Penelitian menggunakan rancangan acak kelompok lengkap (RAKL) dengan variasi rasio kappa-karagenan dan pati limbah kulit singkong. Hasil menunjukkan bahwa formula kappa-karagenan dan pati limbah kulit

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singkong secara signifikan memengaruhi seluruh sifat fisikomekanik bioplastik, kecuali densitas. Seluruh formula memenuhi standar JIS untuk kuat tarik dan elongasi. Formula yang menggunakan pati limbah kulit singkong dengan konsentrasi yang lebih tinggi secara signifikan memperbaiki sifat penghalang bioplastik dengan menurunkan laju transmisi uap air dan penyerapan air. Peningkatan konsentrasi pati meningkatkan elongasi film secara signifikan, tetapi mengurangi ketebalan, kuat tarik, dan modulus Young, serta menghasilkan biodegradasi yang lebih lambat. Formula terbaik diperoleh pada rasio 1% kappa-karagenan: 4% pati dengan ketebalan 0,35 mm, kuat tarik 1,14 MPa, elongasi 25,78%, dan laju transmisi uap air 18,47 g/m2/hari. Hasil ini menunjukkan bahwa formulasi kappa-karagenan dan pati limbah kulit singkong dapat menghasilkan bioplastik dengan sifat fisikomekanik yang memenuhi standar dan berpotensi mengurangi polusi plastik secara keberlanjutan di masa depan.

Kata kunci: biodegradabel, kuat tarik, sifat mekanis, sifat penghalang, transparansi

INTRODUCTION

The global environmental crisis caused by plastic waste has intensified into one of the most pressing challenges facing humanity. Annually, plastic production has surged to over 400 million metric tons, a significant portion of this turning into waste in natural environments (Pilapitiya & Ratnayake, 2024). This plastic waste has persisted for centuries, posing severe threats to ecosystems, economies, and sustainability efforts. Marine life is particularly affected, as species suffer from ingestion and entanglement in plastic debris, while microplastics infiltrate the food chain, raising serious concerns about human health (Pandey *et al*., 2023; Thushari & Senevirathna, 2020). The economic impact is equally profound, with billions spent on cleanup efforts and lost revenue in sectors like tourism and fisheries (Raes *et al*., 2022). The enduring resilience of conventional plastics in the environment highlights the urgent need for alternative materials to decompose more readily and reduce our dependency on fossil fuels. In response to this escalating crisis, the scientific community has concentrated on developing bioplastics—materials derived from renewable sources that offer a more sustainable alternative to traditional plastics. Starch has emerged as a promising candidate due to its natural abundance and biodegradability. Starch is a polysaccharide found in various plants, and its ability to form films makes it an attractive and sustainable material for bioplastic production and prospective use in the future (Kumar *et al*., 2022). Starch-based bioplastics are better than synthetic plastics because they come from renewable resources, have less of an effect on the environment, and are in line with the ideas of a circular economy, which means that resources are reused and waste is kept to a minimum (Kumar *et al*., 2024; Rosenboom *et al*., 2022; Zhao *et al*., 202).

Cassava peel waste, a byproduct of the cassava processing industry, is particularly intriguing as a starch source (Fronza *et al*., 2023). In many regions, cassava serves as a staple crop, and its processing generates substantial amounts of peel waste, which accounts for 5–30% of total waste (Olukanni & Olatunji, 2018). Despite its high starch content, this waste is often discarded or used as low-value animal feed (Bayata, 2019). Recent studies have explored the potential use of cassava peel starch in the production of bioplastic. The results look good (Maulida *et al*., 2016; Syuhada *et al*., 2020) because it is cheap, safe, and easy to break down (de Lima Barizão *et al*., 2020). Bioplastics made from cassava peel starch, on the other hand, tend to be brittle and have low tensile strength. They also tend to absorb water easily (Abe *et al*., 2021; Oluwasina *et al*., 2019), which makes them much less useful in real life. To address these limitations, researchers have explored the incorporation of other natural polymers, such as kappa-carrageenan, into starch-based bioplastics.

Kappa-carrageenan is a red seaweed-derived polysaccharide widely used in the food industry for its stabilizing, thickening, and gelling properties (Siregar *et al*., 2016). Kappa carrageenan works as a film-forming humectant (Sujuliyani *et al*., 2021). It can also make edible coatings stronger and more stable (Nurdiani *et al*., 2022). Kappa carrageenan is a galactose polysaccharide compound that is easily hydrolyzed in acidic solutions and is stable in an alkaline atmosphere (Sandria *et al.*, 2017). It is also biodegradable and renewable, making it a valuable addition to bioplastic formulations (Ramadas *et al.*, 2024). When combined with starch, kappa-carrageenan can enhance the flexibility and mechanical strength of the resulting bioplastic (Favian & Nugraheni, 2023; Roy & Rhim, 2021). However, its effectiveness is sometimes limited by its sensitivity to moisture, which leads to a significant drawback for practical applications, such as decreased mechanical performance under humid conditions (Cheng *et al.*, 2022; Hanani & Husna, 2018). Adding polyvinyl alcohol (PVA), a man-made polymer known for its great film-forming and sticking abilities, is another way to improve the physicomechanical properties of bioplastics.

A lot of research has shown that PVA can make bioplastic films stronger, more biodegradable, better at letting water vapor through, and less likely to get wet (Chan & Tang, 2022; Hanani *et al.*, 2023; Liu *et al.*, 2022). Due to its non-toxic and safe nature (Abedi-Firoozjah *et al.*, 2023), the Food & Drugs Administration (FDA) has approved the use of PVA as a material for food packaging (Gómez-Aldapa *et al.*, 2020). In our previous study (Fadhallah *et al.*, 2024), a bioplastic made from cassava peel starch combined with carrageenan displayed poor elongation that doesn't meet the standard for food packaging film as specified by the Japanese Industrial Standard (JIS) (Japanese Standard Association, 2019). It is expected that adding PVA to bioplastics made from kappa-carrageenan and cassava peel waste starch will improve their mechanical properties, making them better for a wider range of uses. However, the use of cassava peel starch and kappa-carrageenan mixtures with PVA reinforcement in bioplastic has not yet been studied. This study aims to find the best combination of kappa-carrageenan and cassava peel starch for bioplastic based on its thickness, tensile strength, elongation, and water vapor transmission rate (WVTR). JIS Z 1707 recommends doing this. Furthermore, this study is expected to provide valuable insights into the potential use of agricultural waste materials, such as cassava peel, as a renewable and sustainable material for bioplastic production.

MATERIALS AND METHOD

Starch Extraction from Cassava Peel Waste

The extraction procedure for starch from cassava peel waste followed the method outlined by Fronza *et al.* (2023). Cassava (*Manihot esculenta*) peel waste was collected from an unprocessed solid waste site in a cassava chip factory in Bandar Lampung, Indonesia. It was placed into a plastic bags and transported to the laboratory. The outer brown layer of the peel was removed, leaving only the white inner layer (cortex). The cortex was thoroughly washed under running water to remove physical contaminants and soaked in water for 24 hours. After soaking, the cortex was drained for 10 minutes and cut into pieces (approximately 3×3 cm). Consequently, it was blended with water at a ratio of 2:1 (water:cortex). The mixture was filtered to separate the solid residue (pulp) from the liquid fraction. The liquid fraction was left to settle for 24 hours to allow the starch to precipitate. The precipitated starch was collected and dried in a dehydrator at 50°C for 4 hours. The dried starch was then ground into a fine powder using a blender (Waring 8010S, 22,000 RPM) and sieved through an 80-mesh sieve to obtain uniformly sized starch powder.

Preparation of Bioplastic

Bioplastics in this study were prepared using a casting method based on the previous study (Fadhallah *et al.*, 2024). Cassava peel starch (starch content 21.6%±0.03, amylose 17.02%±0.10, amylopectin 82.98%±0.10) and kappa-carrageenan powder (Indogel SGP-168M, water gel strength 1295 g/cm^2 at 1.5% gel) were weighed according to the formulation (Table 1) and placed in a beaker (Pyrex), followed by the addition of 150 mL of aquadest. The mixture was stirred and heated using a hot magnetic stirrer at 60°C for 20 minutes. Then, PVA (88% hydrolyzed, viscosity 45.8 cps, Chang Chun Petrochemical, Taiwan) and glycerol (pharmaceutical-grade,

Table 1 Formulations of bioplastic
Tabel 1 Formulasi bioplastik

Materials (%)	Formulation ratio of kappa-carrageenan and cassava peel starch (%)					
	5:0 (kC5)	4:1 (S1kC4)	3:2 (S2kC3)	2:3 (S3kC2)	1:4 (S4kC1)	0:5 (S5)
Kappa-carrageenan	5	4	3	2	1	0
Starch	0	1	2	3	4	5
PVA	22	22	22	22	22	22
Glycerol	3	3	3	3	3	3
Aquadest	70	70	70	70	70	70
Total	100	100	100	100	100	100

100% purity, OneMed, Indonesia) were added, and the mixture was stirred for 10 minutes. Once homogenized, the mixture was poured into a 15x30 cm glass plate and allowed to cool at room temperature for 24 hours. The films were dried using a dehydrator at 50°C for 2 hours. Subsequently, it was conditioned at room temperature for 30 minutes before being carefully peeled off for further characterization.

Fourier-Transform Infrared (FTIR) Analysis

The FTIR spectra were analyzed following the ASTM E1252-98 (American Society for Testing and Materials [ASTM], 2021). Potassium bromide (KBr) pellets were prepared before analysis. The FTIR portable instrument (Agilent Technologies Cary 630, US) was configured with a resolution of 2 cm^{-1} and a wavenumber range from 650 to 4000 cm^{-1}. Bioplastic samples (1×1 cm) were pressed with KBr and placed in the sample holder. Scanning was conducted 32 times to enhance spectral accuracy. The resulting spectra were then analyzed to determine the chemical structure and any changes in the functional groups of the samples.

Physical Properties Analysis (Thickness, Density, and Transparency)

The bioplastic film samples were cut into 3×3 cm (A) pieces and weighed (B) using a laboratory digital analytical scale (Osuka, precision of 0.001 g). The thickness was measured using a digital gauge (Digilife, precision of 0.001 mm) at five points: the center, top-right, bottom-right, top-left, and bottom-left. These five measurements were then averaged to determine the thickness (C) in millimeters (mm). The density was expressed as g/cm^3 following ASTM D792 (ASTM, 2020), calculated by dividing the sample's weight (B) by its volume (A×C). The thickness and density data were presented as averages followed by standard deviations.

The transparency of the bioplastic sample was measured following Ulyarti *et al.* (2021). The samples were prepared into 4x1 cm and then placed in a cuvette. The transmittance (T) was measured using a UV-Vis spectrophotometer (Genesys 10S, US) at 600 nm. The percent transparency of the bioplastic films was calculated by dividing the transmittance (T) value by the sample thickness (mm). The transparency data were presented as averages followed by standard deviations.

Mechanical Properties

The sample's mechanical properties, including tensile strength, elongation, and Young's modulus, were tested following the ISO 527-1 standard (International Organization for Standardization [ISO], 2019). The testing machine, Universal Testing Machine (Orientec, UCT-5T, Japan), was configured with a 25 kg load cell, a crosshead speed of 1 mm/s, and a grip separation of 60 mm. The sample films were cut into 4x1 cm and vertically placed in the grips of the testing machine at a temperature of 27°C and 65% relative humidity. The displayed data is then recorded for further calculation. Tensile strength (MPa) was calculated by dividing the force (N) by the cross-sectional area of the sample (mm^2); elongation was calculated by dividing the strain with the initial length, and Young's modulus (MPa) was calculated by dividing the tensile strength by the elongation. Each mechanical property value was reported as the mean, followed by the standard deviation.

Barrier Properties (Water Absorption and WVTR)

Bioplastic samples (2×2 cm) were first weighed to determine their initial weight (W_0). Following ASTM D570 (ASTM, 2022), the samples were immersed at room temperature for 1 hour in 20 mL of distilled water. After immersion, they were allowed to drain for 5 minutes, and their surfaces were gently wiped with a tissue. The samples were reweighed to obtain the final weight (W_1). The water absorption was calculated by dividing the weight difference (ΔW) by the initial weight and then multiplying by 100%.

The WVTR was determined using the gravimetric cup method following ASTM E96 (ASTM, 2011). Bioplastic samples were cut into circular shapes matching the diameter of the test cup. Blue silica gels (10 g) were placed inside the cup and then sealed with the bioplastic sample. All specimens were then maintained at room temperature for 24 hours. The initial and final weights of the sample were recorded once within 24 hours, and the WVTR was calculated in g/m^2/day.

Biodegradability Test

The biodegradability test was performed using the soil burial method (Nissa *et al.*, 2019). Bioplastic film samples were cut into 1.5x1.5 cm and 3×3 cm pieces, and their initial weight (W_0) was recorded. The samples were then buried at a depth of 7.5 cm in mixed ultisol soil. It was collected from the surface at 25 cm depth and 50 cm depth from the Integrated Field Laboratory, Faculty of Agriculture, University of Lampung (Indonesia). The samples were left undisturbed for 30 days. To simulate natural environment conditions, the soil was moistened daily with approximately 30 mL of water (Charles *et al.*, 2022). The samples were retrieved every three days, cleaned of any attached soil, and reweighed (W). The percentage weight loss of the samples was calculated as the biodegradation rate, determined by the difference between the initial and final weights relative to the initial weight.

Experimental Design and Data Analysis

A completely randomized block design (CRBD) was employed in this study with six formulations by varying kappa-carrageenan and cassava peel waste starch concentrations (w/w), which were replicated four times. A total of five percent of cassava peel starch and carrageenan were used to make film-forming mixtures that were tested to find the best mix for bioplastic packaging film uses. FTIR, physical properties (such as thickness, density, and transparency), mechanical properties (such as tensile strength, elongation, and Young's modulus), barrier properties (such as water absorption and WVTR), and biodegradability in soil were all looked at in this study. The obtained data were analyzed statistically through one-way ANOVA followed by Duncan's post-hoc test at a 5% significance level, using IBM SPSS version 25. Meanwhile, the FTIR spectra and biodegradability tests were examined and discussed descriptively.

RESULTS AND DISCUSSION

FTIR Analysis

FTIR spectroscopy was used to look at the differences in structure between bioplastic films made with different amounts of cassava peel starch and carrageenan. The bioplastic's FTIR spectrum is shown in Figure 1. It shows a uniform mix of bioplastic film formulations, as shown by the presence of functional groups from different materials, such as cassava peel starch, kappa-carrageenan, and PVA.

As with other studies (de Jesus *et al.*, 2023; Panatarani *et al.*, 2023), all of the samples showed absorption peaks between

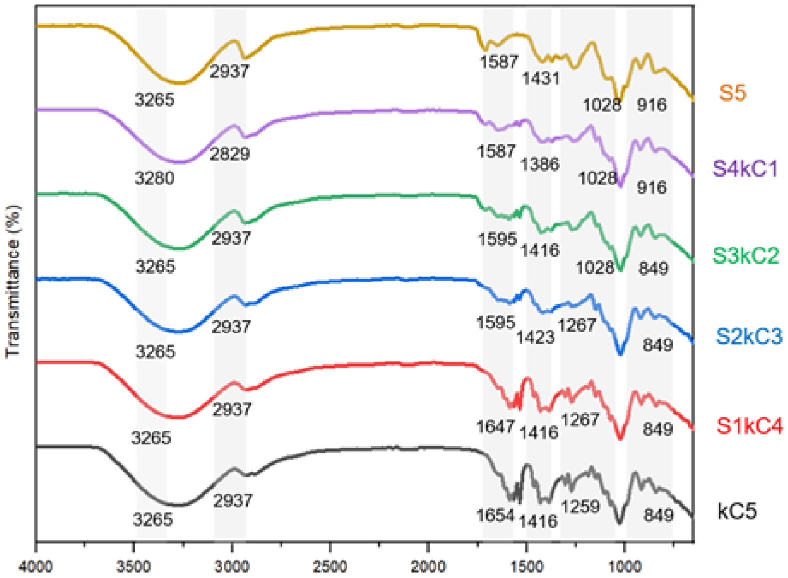


Figure 1 FTIR spectra of bioplastic samples
Gambar 1 Spektra FTIR dari sampel bioplastik

3265 and 3280 cm^{-3}. These peaks showed the -OH vibration of hydroxyl groups in starch, kappa-carrageenan, and PVA. These peaks show that the hydroxyl groups are hydrogen-bonded to each other. Small changes in the wavenumber between formulations show that these interactions are different because the amounts of starch and kappa-carrageenan are distinct. The C-H stretching vibrations were found at around 2829 cm^{-3} and 2937 cm^{-3}. These vibrations mostly came from the starch and PVA hydrocarbon backbones (Lim *et al.*, 2021; Maziad *et al.*, 2018). The appearance of peaks at 1587, 1595, 1654, and 1657, which are attributed to the amide I band, suggests that protein or peptide parts are present, most likely coming from kappa-carrageenan. A noticeable shift in wavenumber is observed within this range, particularly as the ratio of kappa-carrageenan increases. The ʙ-carrageenan sulfate groups and the amide functionalities may have formed a stronger hydrogen bond or ionic interaction. The 1431 cm^{-1} and 1386 cm^{-1} peaks correspond to C-H bending vibrations. The C-O stretching vibrations within the 1028-1267 cm^{-1} region are associated with alcohols, ethers, and esters (Maziad *et al.*, 2018). The hydroxyl groups from starch, kappa-carrageenan, and PVA interact with each other and form intermolecular hydrogen bonds that affect the structural properties of the bioplastics (Lim *et al.*, 2021). This is shown by changes in the peak positions in this area. All of the samples had peaks at about 916 cm^{-3} and 849 cm^{-3}, which means they all have sulfate groups. These peaks correspond to the C-O-C band of 3,6-anhydro-D-galactose, which is unique to kappa-carrageenan, as Santos *et al.* (2023) also said.

Physical Properties

The physical properties of bioplastic as packaging film in this study include thickness, density, and transparency. Table 2 implies that the formulation of cassava peel waste starch and carrageenan significantly affected the film's thickness and transparency ($p<0.05$), while it showed no effect on the film's density ($p>0.05$). A photograph of bioplastic in this study is presented in Figure 2.

The thickness of bioplastic films, which refers to the distance between their outer surfaces, plays a crucial role in determining the physical and mechanical properties of the bioplastic (Shah *et al.*, 2016). Table 2 shows that the combination of starch and kappa-carrageenan formulations significantly influenced the thickness of the bioplastic ($p<0.05$). It varied significantly with the

Table 2 Physical properties of bioplastic films
Tabel 2 Sifat fisik dari film bioplastik

Kappa-carrageenan : starch	Thickness (mm)	Density (g/cm^3)	Transparency (%)
5:0	0.65±0.05^a	1.89±0.017^a	1.49±0.32^a
4:1	0.58±0.02^b	1.78±0.013^a	2.00±0.35^b
3:2	0.39±0.01^c	1.67±0.014^a	2.80±0.17^c
2:3	0.34±0.03^c	1.78±0.019^a	2.91±0.29^c
1:4	0.35±0.03^c	1.74±0.017^a	3.06±0.14^c
0:5	0.25±0.01^d	1.66±0.016^a	3.54±0.27^d

Numbers followed by different letters in the same column indicate a significant difference ($p<0.05$)

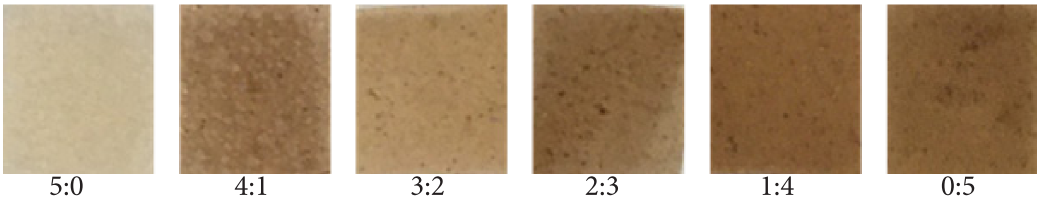


Figure 2 Photograph of bioplastic film from various kappa-carrageenan and cassava peel starch ratio
Gambar 2 Foto bioplastik dari pati limbah kulit singkong dan kappa-karagenan

different starch-to-carrageenan ratios, with the kC5 (5% kappa-carrageenan) being the thickest at 0.65 mm and S5 (5% starch) being the thinnest at 0.25 mm. This trend suggests that as the concentration of cassava peel starch decreases, the thickness of the bioplastic also decreases. Starch molecules, when present in higher concentrations, create a more robust network that contributes to the overall thickness of the film. When the concentration of starch is reduced, polymer chains become less entangled, leading to a thinner film structure. This reduction in thickness is often accompanied by a decrease in mechanical strength and barrier properties, which are critical for applications such as food packaging (Abdullah *et al.*, 2019). The fact that extracted starch from cassava peel is semi-crystalline (Thuppahige *et al.*, 2023) also plays a part. Lower concentrations may not have enough crystalline regions to support the film's integrity, which leads to thinner films. Conversely, as the concentration of kappa-carrageenan increases, the resulting bioplastic becomes thicker. Kappa-carrageenan acts as a gelling agent and can form a more cohesive network when mixed with starch. As the concentration of carrageenan increases, more polymer chains are incorporated to improve the film's structural integrity. According to Sriphochai & Prachayawarakorn (2024), this is important because kappa-carrageenan can hold a lot of water because it is hydrophilic. The absorption and retention of water in the matrix could result in a thicker film. When carrageenan and starch interact, they create a phase-separated structure. The carrageenan matrix strengthens the starch grains, making the film thicker and better at withstanding mechanical forces (Larotonda *et al.*, 2016). The bioplastic thickness trend in this study is consistent with the findings of Manuhara *et al.* (2016), who reported a 20.45% increase in thickness by increasing the carrageenan content by 0.5-1.0%. Also, Zaky *et al.* (2021) found an increase in thickness from 0.025 mm to 0.093 mm with the increased concentration of carrageenan in CMC-based biodegradable films. However, only the S5 formulation has met the JIS Z 1707 standard, which stipulates a maximum thickness of 250 μm or 0.25 mm for food packaging (Japanese Standard

Association, 2019), whereas the higher carrageenan content fails to meet the standard.

Another factor that could influence film thickness is the volume of the bioplastic solution (Figaliah *et al*., 2024). The total amount of materials used in this study stayed the same for all formulations. This included the amount of aquadest used as a solvent for kappa-carrageenan and cassava peel starch. However, discrepancies in solution viscosity may arise due to variations in kappa-carrageenan concentrations. Abdou & Sorrour (2014) illustrated that elevated carrageenan concentrations notably elevate solution viscosity. For instance, a solution containing 1.25% starch and 0.5% carrageenan displayed a viscosity of 0.1 Pa.s, contrasting with 0.6 Pa.s for a solution comprising 1.25% starch and 0.75% carrageenan. Even though viscosity wasn't measured in this study, it's possible that higher concentrations of kappa-carrageenan caused viscosity to rise, which made it harder for the solution to spread evenly during casting. This may have led to a slightly thicker liquid layer on the casting plate, contributing to the observed increase in film thickness after drying. Sedayu *et al.* (2019) emphasized that carrageenan is highly effective as a base material for biodegradable films due to its hydrocolloid properties, which can form a strong matrix, increasing the film's thickness.

The formulation of cassava peel starch and kappa-carrageenan did not significantly affect the density of the bioplastic (p>0.05). The density values ranged from 1.66 to 1.89 g/cm^3 (Table 2). Based on what Nugrahanto *et al.* (2021) found, the density of biodegradable films made from tapioca starch and low-quality milk casein was between 1.5 and 1.8 g/cm^3. The relatively consistent density across different formulations suggests that the internal structure or compactness of the film matrix remains stable, despite variations in thickness. This consistency is crucial because it shows that the molecular interactions between starch, kappa-carrageenan, and glycerol are balanced. This keeps the structure of the film from being too porous, even when the thickness changes a lot. The type and concentration of plasticizers significantly influence the density of bioplastic. Edhirej *et al.* (2017) varied the type of plasticizers and revealed that the fructose-plasticized film produces the highest density. Nigam *et al.* (2021) used different amounts of PEG600 as a plasticizer in cellulose-based bioplastic. As the plasticizer concentration went up (10–50%), the density went down (1.54 to 1.33 g/cm^3). Tarique *et al.* (2021) reported that an increase in glycerol concentration (15 to 45%) slightly decreased the density (1.47 to 1.33 g/cm^3). Furthermore, Rahmatullah *et al.* (2022) observed that bioplastics produced with glycerol exhibited lower density (0.836 g/cm^3) compared to those with sorbitol (0.941 g/cm^3).

Glycerol, used as a plasticizer in this study, plays a crucial role in maintaining these balanced interactions. As a low-molecular-weight plasticizer, glycerol reduces the intermolecular forces between polymer chains, enhancing flexibility and reducing brittleness (Tarique *et al*., 2021). However, unlike other plasticizers, glycerol's hydrophilic nature and compatibility with starch and carrageenan likely contribute to maintaining uniform density throughout formulations. Plasticizers are additive bioplastic materials with a lower molecular weight than the polymer matrix (Tyagi & Bhattacharya, 2019). The addition of more plasticizer leads to a decrease in the average density of the bioplastic, as it increases the proportion of lower-density plasticizer in the composite. In the current study, the consistent density values across varying formulations suggest that the glycerol content used was sufficient to maintain a uniform film structure. This consistency in density is crucial when considering other film properties, such as mechanical strength and water vapor transmission rates, where a denser film could offer better barrier properties and enhanced durability.

Table 2 revealed significant differences among bioplastic transparency in various formulations (p<0.05). Bioplastics with a higher starch content showed greater transparency, while those with higher kappa-carrageenan content tended to be less transparent. The highest transparency value, 3.54%, was found in S5 (5% starch). Conversely, the kC5 (5% kappa-carrageenan) exhibited the lowest transparency values, measuring 1.49%. This result was in line with another study by de Lima Barizão *et al.* (2020), who reported that films with 75% carrageenan have a lower transparency (0.67%) than 25% carrageenan (0.86%). Dmitrenko *et al.* (2023) showed a more transparent visual of bioplastic film in a carrageenan/starch ratio of 1:2 rather than 2:1. The increase in transparency with higher starch content can be related to starch gelatinization, where starch absorbs water and forms a more transparent gel, which plays a crucial role in this phenomenon. Li (2022) explained that during high-temperature heating, gelatinization causes the initially opaque starch suspension to become clear, resulting in a more homogeneous and transparent film structure. This enhanced clarity is related to the swelling capacity and amount of starch granules; the higher the starch presence, the greater the swelling capacity, resulting in a clearer bioplastic. However, the possibility of starch browning due to the heating process is also considered to affect the film's transparency. Browning can occur due to Maillard reactions that lead to the darker color of the film. Interestingly, in this study, despite the visible brown color found in bioplastic with higher starch content (Figure 2), the transparency value increased. This means that browning doesn't really affect the optical clarity of bioplastic. Instead, the gelatinization of starch granules is probably what makes it clear.

The amylopectin content in cassava peel starch also influences the transparency properties of the bioplastic. Due to its long chains, amylopectin can lead to poor chain rearrangement and less compact starch matrix formation, resulting in a more transparent film (Domene-López *et al.*, 2019). Interestingly, this study reveals a correlation between transparency and thickness. Thinner films, like the S5 sample, tend to have a higher transparency due to less material obstructing light passage. Additionally, the smoother and more uniform surface of carrageenan-dominant films contributes to this increased transparency. This surface uniformity enhances transparency and suggests better film formation, which may positively impact mechanical properties such as tensile strength and elongation.

Mechanical Properties

This study examined the mechanical properties, including tensile strength, elongation, and Young's modulus. Table 3 shows that cassava peel starch and carrageenan formulations significantly affected all parameters of the bioplastic's mechanical properties.

The mechanical properties of the bioplastic, including tensile strength, elongation at break, and Young's modulus (Table 3), were significantly influenced by the varying concentrations of starch and carrageenan (p<0.05). The tensile strength of the films exhibited a clear correlation with the starch and kappa-carrageenan concentrations. The highest tensile strength value (1.91 MPa) was recorded for the kC5 (5% kappa-

Table 3 Mechanical properties of bioplastic films
Tabel 3 Sifat mekanis dari film bioplastik

Kappa-carrageenan : starch	Tensile strength (MPa)	Elongation (%)	Young's modulus (MPa)
5:0	1.91±0.002^{a}	23.70±0.003^{d}	806.54±2.01^{a}
4:1	1.33±0.001^{b}	25.41±0.001^{c}	522.58±3.57^{b}
3:2	1.28±0.002^{c}	25.61±0.002^{cd}	496.68±2.70^{c}
2:3	1.20±0.001^{d}	25.91±0.001^{bc}	463.17±1.04^{d}
1:4	1.14±0.001^{e}	25.78±0.004^{abc}	440.10±2.26^{e}
0:5	1.10±0.001^{f}	26.03±0.003^{a}	420.34±0.94^{f}

Numbers followed by different letters in the same column indicate a significant difference (p<0.05) carrageenan), while the lowest (1.10 MPa) was observed in the S5 (5% starch). This trend aligns with de Lima Barizão *et al.* (2020), who reported that films without carrageenan produce lower tensile strength (6.53 MPa) than those with 50% carrageenan (17.29 MPa) of starch/PVA-based film. Another study by Favian & Nugraheni (2023) also reveals that a 0.5% higher concentration of carrageenan significantly increases the tensile strength of chitosan-based bioplastic by 47%. Bioplastics with higher concentrations of kappa-carrageenan exhibit a higher tensile strength than those with higher starch. This inverse relationship between tensile strength and starch concentration aligns with the understanding that starch, with its robust intermolecular hydrogen bonding, contributes significantly to the mechanical strength of the film. As the concentration of starch decreases, the film matrix loses some of its rigidity, resulting in reduced tensile strength. Conversely, kappa-carrageenan's presence in higher concentrations increased tensile strength due to its ability to create a more cohesive and elastic matrix. The addition of carrageenan in the bioplastic strengthens the intermolecular interactions, enhancing the film's tensile strength (Adam *et al.*, 2022; Huang *et al.*, 2020). The tensile strength values observed in this study meet the Japanese International Standard (JIS) for plastic packaging, which requires a minimum tensile strength of 0.392 MPa (Japanese Standard Association, 2019). The inverse relationship between tensile strength and transparency is also noteworthy as the starch content increases, resulting in higher transparency and decreasing tensile strength.

The elongation at break, which measures the film's ability to stretch before breaking, showed an opposite trend to tensile strength (Table 3). The highest elongation value (26.03%) was observed in the S5 (5% starch) and the lowest (23.70%) in the kC5 (5% kappa-carrageenan), indicating that films with higher carrageenan content are less flexible and unable to stretch further before breaking. The results agree with de Lima Barizão *et al.* (2020), who revealed a significant decrease in starch/PVA-based bioplastic using 25% of carrageenan (21.60%), while zero concentration of carrageenan exhibits the highest elongation at break (67.65%). Additionally, Suryanto *et al.* (2019) also report the same trend where increasing the amount of carrageenan (0%, 2.5%, 5%) decreases the elongation of starch/carrageenan blend films (28.69%, 20.22%, 19.48%). The increase in elongation with higher starch content, as found in the S5 formulation, is attributed to the absence of kappa-carrageenan, allowing starch to form a more flexible and elastic network. This trend indicates that the flexibility of the films enhances as the matrix transitions from a carrageenan-dominated structure to a starch-dominated one. Furthermore, the higher elongation observed in the thinner, more transparent S5 film supports the notion that increased flexibility correlates with greater transparency. The elongation value in this study showed a negative correlation with film thickness, indicating that thinner films displayed higher elongation (Table 1). Thinner films typically possess a less dense structure, which allows for greater polymer chain mobility and leading to higher elongation and flexibility, as demonstrated by Menzel (2022) and Hidayati *et al.* (2021). This finding is significant as it highlights the inverse relationship between elongation and tensile strength; as the film becomes more flexible, its tensile strength decreases. The elongation values observed in this study fall within a fairly good range, as per the Japanese Standards Association (JIS), where an elongation below 10% is considered poor and above 50% is considered good (Japanese Standard Association, 2019).

Young's modulus, which indicates the material's stiffness, showed significant variation with the different starch and kappa-carrageenan formulations. The highest Young's modulus was recorded for the kC5 (5% kappa-carrageenan) with 806.54 MPa, while the lowest was observed in the S5 (5% starch) with 420.34 MPa (Table 3). These results demonstrate that as the kappa-carrageenan content increases, the films become stiffer, while higher starch content results in more elastic and less rigid films. This trend is consistent with the properties of materials such as kappa-carrageenan, known for its strong gel-forming ability and contributes to a more rigid structure (Tasende & Manrique-Hernandez, 2016; Yahaya *et al*., 2023). At the same time, starch, with its flexible polymer chains, imparts greater elasticity to the films (Xu *et al*., 2023). The decrease in Young's modulus with an increasing starch content underscores the greater flexibility of starch-based films, making them less stiff but more adaptable to stretching. The relationship between Young's modulus, tensile strength, and elongation is evident; as Young's modulus and tensile strength decrease, elongation increases, indicating a more elastic and flexible material.

In addition, the type of plasticizer used plays a significant role in determining Young's modulus. Plasticizers interact with polymer chains by reducing intermolecular forces and enhancing chain mobility, which in turn decreases stiffness and lowers Young's modulus (Garavito *et al*., 2024). Tarique *et al*. (2021) compared Young's modulus of bioplastics plasticized with glycerol and sorbitol at the same concentration. Their results showed that bioplastics with glycerol exhibited a lower Young's modulus (31.546 KPa) compared to those with sorbitol (72.614 KPa). This suggests that sorbitol contributes to a stiffer structure than glycerol, likely due to its lower molecular mobility and stronger interactions with polymer chains, which restrict movement and increase rigidity. In this study, glycerol was preferred as the plasticizer because it produces a more flexible film.

Our findings corroborate those of Abdullah *et al*. (2021), who observed a positive correlation between carrageenan concentration (0% to 15%) and Young's modulus (4.99 MPa to 11.89 MPa). This is attributed to kappa-carrageenan's tendency to form rigid gel networks. Furthermore, de Lima Barizão *et al*. (2020) demonstrated a significant reduction in Young's modulus of bioplastic when the starch-to-kappa carrageenan ratio increased. For instance, 100% carrageenan exhibited a Young's modulus of 59.64 MPa, whereas a 75:25 carrageenan-to-starch ratio yielded a value of 27.03 MPa.

Barrier Properties

This study evaluates the bioplastic's barrier properties, including the water absorption and water vapor transmission rate (WVTR). The various formulations of cassava peel starch and kappa-carrageenan significantly affected these barrier properties ($p<0.05$) (Table 4).

The water absorption analysis of the bioplastic reveals a clear trend where an increase in the starch content results in a significant decrease in water uptake ($p<0.05$), with 5% kappa-carrageenan formulation exhibiting the highest absorption at 189.94% and 5% starch the lowest at 53.55% (Table 4). This trend is aligns with the understanding that highly hydrophilic kappa-carrageenan facilitates

Table 4 Barrier properties of bioplastic films
Tabel 4 Sifat penghalang dari film bioplastik

Kappa-carrageenan : starch	Water absorption (%)	WVTR (g/m^2/day)
5:0	189.94±0.08^a	26.51±0.52^a
4:1	155.44±0.04^b	23.22±0.78^b
3:2	149.55±0.07^b	23.54±0.87^b
2:3	126.67±0.02^c	21.87±0.40^c
1:4	71.46±0.08^d	18.47±0.81^d
0:5	53.55±0.07^e	13.97±0.68^e

Numbers followed by different letters in the same column indicate a significant difference ($p<0.05$)

water absorption through to its sulfate groups, which attract water molecules. Additionally, its more amorphous structure (Selvin *et al.*, 2018) increases water absorption. The higher water absorption in kC5 was attributed to the extensive network of kappa-carrageenan's hydrophilic chains that readily interact with water, leading to significant swelling. As the proportion of cassava peel starch increases, the water absorption decreases, likely due to the reduced hydrophilicity of starch compared to kappa-carrageenan. When incorporated into the matrix, starch granules restrict water absorption by swelling less and form more compact structures that impede water penetration. Starch also exhibits a lower affinity for water due to its crystalline regions (Mohamed, 2021), which have less capability to interact with water molecules. This is supported by de Lima Barizão *et al.* (2020), who reported a significant reduction in swelling degree, from 2002% in 100% carrageenan bioplastics to 391.6% in 100% starch bioplastics. The thickness data in this study (Table 1) supports this interpretation, with thicker films, such as kC5, absorbing more water, potentially due to a higher carrageenan content, leading to more significant swelling. As mentioned by Hanry & Surugau (2020), thicker films generally indicate a higher content of hydrophilic materials, which can absorb more water, as the larger volume provides more sites for water interaction. Favian & Nugraheni (2023) observed a significant increase in solubility from 7.29% to 36.98% as carrageenan content increased. Wahyuningtiyas *et al.* (2018) reported moisture absorption ranging from 18.94% to 44.44% with increasing starch content, further validating these findings, demonstrating that higher starch or lower kappa-carrageenan levels correlate with reduced water-related properties. Islamiyah *et al.* (2022) also reported the same trend of gradual water absorption decrement (132.78% to 50.96%) as the starch concentration rises (0, 1, 1.5, and 2 g) on modified starch-carrageenan-based biodegradable films. These results suggest that bioplastic films with a higher starch content may be more suitable for applications with lower water absorption, such as packaging materials requiring moisture resistance.

The study's findings on the WVTR of bioplastic packaging film formulated with varying proportions of kappa-carrageenan and cassava peel starch demonstrate a clear trend in which the WVTR values decrease significantly ($p<0.05$) as the proportion of cassava peel starch increases. Specifically, the kC5 formulation, which contains 5% kappa-carrageenan without cassava peel starch, exhibits the highest WVTR at 26.51 g/m^2/day. In contrast, the lowest (13.97 g/m^2/day) was found in the S5 formulation, consisting of 5% cassava peel starch without kappa-carrageenan (Table 4). The intermediate formulations (S1kC4, S2kC3, S3kC2, and S4kC1) showed a consistent pattern where WVTR decreases with the addition of cassava peel starch. This observed trend can be attributed to the distinct material properties of kappa-carrageenan and cassava peel starch. Kappa-carrageenan, being more hydrophilic and possessing a less dense structure, is inherently more permeable, allowing for greater moisture passage, which contributes to higher WVTR values. This characteristic is particularly evident in the kC5 formulation, where the thicker film correlates with a higher WVTR due to the porous nature of kappa-carrageenan's sulfate groups, which attract water molecules, thus increasing hydrophilicity and WVTR (Muthukumar *et al.*, 2021). Conversely, cassava peel starch, composed predominantly of amylose and amylopectin, forms a dense and compact structure that effectively restricts water vapor diffusion. Amylose, a linear polysaccharide, contributes to forming a tightly packed matrix with minimal free volume, which reduces water vapor permeability. Amylopectin, with its branched structure, provides flexibility to the film, further enhancing the film-forming capabilities of starch (Amalia *et al.*, 2021). The crystalline nature of cassava peel starch (Fronza *et al.*, 2023), which increases during aging (Wang *et al.*, 2021), likely enhances the barrier properties of the bioplastics by reducing the available pathways for water vapor transmission. The findings in this study are consistent with those reported by de Lima Barizão *et al.* (2020), who found that pure starch films exhibit a 15% lower

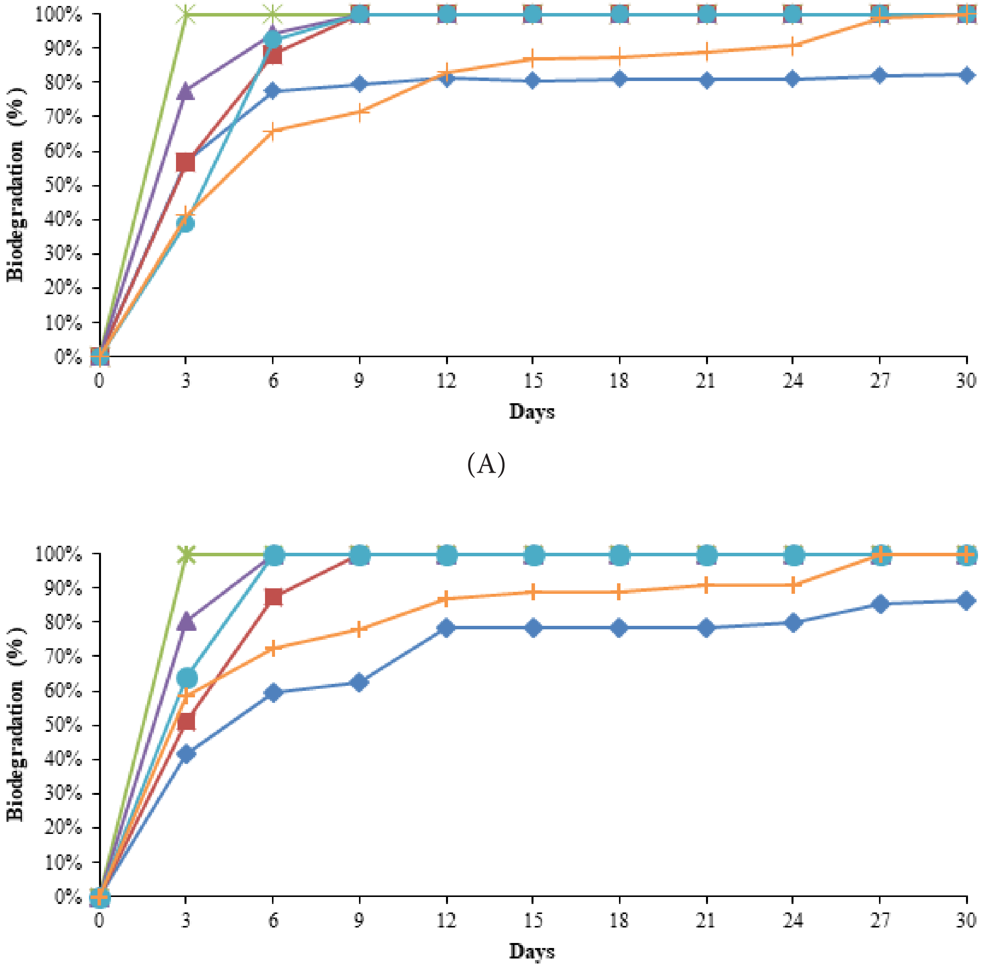
WVTR than pure carrageenan. Another study by Panatarani *et al.* (2023) found that incorporating cassava starch into pure carrageenan films at a 1:1 ratio significantly reduced WVTR by 20% compared to pure carrageenan films. The WVTR values from the present study are generally higher than those reported by Abdou & Sorour (2014), who achieved WVTRs ranging from 3.6 to 4.6 g/m²/day for starch-carrageenan films. However, it is lower compared with Giyatmi *et al.* (2018), who reported that increasing starch concentration from 20% to 80% in arrowroot starch-carrageenan films resulted in a gradual reduction in WVTR from 99.26 to 85.12 g/m²/day, highlighting starch's effectiveness in improving bioplastic's moisture barrier properties.

JIS (2019) classified the food packaging films based on WVTR values into 5 grades: Grade 1 (≤1 g/m²/day), Grade 2 (>1–5 g/m²/day), Grade 3 (>5–20 g/m²/day), Grade 4 (>20–100 g/m²/day), and Grade 5 (>100 g/m²/day). Notably, the WVTR values of the formulations in this study predominantly fall within Grade 4. However, two formulations, S4kC1 and S5, fall within Grade 3Based on these classifications, Grade 4 indicates moderate barrier properties, making it suitable for applications requiring controlled moisture permeability, such as breathable food wraps. On the other hand, Grade 3 represents improved moisture resistance, making them more suitable for packaging materials that require better protection against humidity, such as extended food shelf-life packaging and pharmaceutical coatings (Liu *et al.*, 2023). These results suggest that while some formulations demonstrate relatively improved moisture barrier properties, further optimization is required to achieve lower WVTR values for better performance in applications requiring stronger moisture resistance. A higher WVP value in food packaging may result in food spoilage and degradation (Sohany *et al.*, 2021). The WVTR values in this study showed a positive correlation with film thickness, as presented in Table 2. Specifically, thinner films exhibited lower WVTR values. This finding is consistent with the results reported by Charles *et al.* (2022), who observed a similar relationship between film thickness and WVTR. In their study, a 32% reduction in film thickness resulted in a significant 77% decrease in WVTR, highlighting the strong influence of film thickness on moisture barrier properties.

Biodegradability

The soil burial method was used for 30 days as part of the biodegradation test to see how well bioplastic films made of cassava peel starch and kappa-carrageenan broke down. Figure 3 shows the percentage of biodegradation for different formulations during the testing period, which used two sample sizes.

The results demonstrate that the samples exhibited a progressive increase in degradation over time. Formulation S2kC3 (2% starch: 3% kappa-carrageenan) had the highest biodegradability. Both sample sizes broke down completely in 6 days, giving it a biodegradation value of 100%. In contrast, kC5 (5% kappa-carrageenan) exhibited the lowest biodegradability, with a value of 82.22% (3x3 cm) and 86.47% (1.5×1.5 cm) sample after 30 days, indicating that the sample had not fully degraded (Figure 3a). It was seen that S3kC2 (3% starch: 2% kappa-carrageenan) and S4kC1 (4% starch: 1% kappa-carrageenan) broke down faster than the bigger sizes, and they were completely gone in 6 days (Figure 3b). Higher starch content in the bioplastic typically leads to increased degradation ability. In contrast, higher kappa-carrageenan content is associated with a slower degradation process. We can correlate this relationship to thickness, with kC5 displaying the highest thickness (Table 2). As demonstrated by Charles *et al.* (2022), thicker films degrade more slowly in both seawater and soil environments. The increased mass and structural complexity limit the penetration of crucial environmental factors essential for biodegradation, such as moisture, oxygen, and microbial activity. Additionally, the dense structure of thicker films acts as a barrier, restricting the diffusion of these elements into the material's core. Therefore, the enzymes and chemical processes that break down polymer chains are not as effective because the breakdown starts



(A)

(B)

Figure 3 Biodegradation of bioplastic with different sample sizes (A) 3×3 cm (B) 1.5×1.5 cm
Note: (–◆–) 5% kappa-carrageenan : 0% starch; (–■–) 4% kappa-carrageenan : 1% starch; (–✕–) 3% kappa-carrageenan : 2% starch; (–▲–) 2% kappa-carrageenan : 3% starch; (–●–) 1% kappa-carrageenan : 4% starch; (–+–) 0% kappa-carrageenan : 5%

Gambar 3 Biodegradasi bioplastik pada beberapa ukuran sampel (A) 3×3 cm (B) 1,5×1,5 cm
Keterangan: (–◆–) 5% kappa-karagenan: 0% pati, (–■–) 4% kappa-karagenan: 1% pati, (–✕–) 3% kappa-karagenan : 2% pati, (–▲–) 2% kappa-karagenan : 3% pati, (–●–)1% kappa-karagenan : 4% pati; (–+–) 0% kappa-karagenan : 5% pati

on the outside and moves inside over time (Uzamurera *et al*., 2023; Akhlaq *et al*., 2024).

It was found by Masahid *et al.* (2023) that microbes in the soil can break down the amylopectin part of starch's acetal bonds more easily than the stiffer glycosidic bonds in carrageenan. Moreover, as the burial time increases, the bioplastic material undergoes accelerated degradation, influenced by environmental factors such as soil moisture and microbial activity (Adhikari *et al*., 2016). The higher microbial activity in the soil, particularly in formulations with higher starch content, significantly accelerates the degradation rate. This is consistent with Syuhada *et al.* (2020), who found that starch/chitosan-based bioplastics with a higher starch ratio tend to degrade more rapidly in soil environments. Hypothetically, the abundant microbial population in the soil can metabolize the simpler and more accessible polysaccharides in starch. Furthermore, the size and surface area of the samples play a critical role in the degradation process. A separate study observed that smaller samples (1.5×1.5 cm) degraded more quickly than larger samples (3×3 cm). This is probably because the smaller samples have more surface area compared to volume, which makes them more vulnerable to breaking down agents like enzymes and microbes. Furthermore, films with a higher starch content exhibited faster biodegradation than those with more kappa-carrageenan. This is because both materials have hydroxyl groups (-OH) that help them absorb water and encourage microbial activity, making hydrolysis easier (Onovo *et al.*, 2022). These biodegradability results are crucial for making materials that are better for the environment. The ability to tailor the degradation rate by adjusting the starch and kappa-carrageenan content allows for the design of a bioplastic that balances performance with environmental impact. Formulations with higher starch content are beneficial in applications where rapid degradation is crucial, such as single-use packaging. Conversely, increasing the kappa-carrageenan content might be more suitable for applications requiring longer-lasting materials, considering the mechanical properties.

CONCLUSIONS

This study identified the best formulation for a bioplastic film derived from kappa-carrageenan and cassava peel waste starch. The best formulation, comprising 1% kappa-carrageenan and 4% starch, met the JIS standards for key physicomechanical properties. The results show that mixing cassava peel starch and carrageenan can be changed to make bioplastics that work in certain situations. These bioplastics could be a good alternative to regular plastics and help the environment by using fewer resources that don't grow back.

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