

Sustainable Bioplastics Made from Cassava Peel Waste Starch and Carrageenan Formulations: Synthesis and Characterization

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Received March 09, 2023; Accepted December 04, 2023; Available online March 20, 2024

ABSTRACT. Plastic waste has become a global environmental problem as it is difficult to decompose and harms the environment. The alternative solution to overcome this problem is to produce biodegradable plastics made from renewable materials. Cassava peel waste contains starch that can form a thin film but has poor mechanical properties. Therefore, it is necessary to include additional substances to enhance its strength. Carrageenan is a substance that can improve bioplastic mechanical properties. This study aims to determine the characteristics of bioplastic made from cassava peel waste starch and carrageenan formulation. Nine formulations were tested in this study using the randomized complete block design factorial with the two factors of cassava peel starch (S) and carrageenan (C) concentrations, both at three levels and carried out under three replications. Bioplastics characteristics are observed in thickness, swelling, biodegradation, tensile strength, elongation, and elasticity. The formulation S2C1 (starch 20%; carrageenan 5%) produced the bioplastic with the best mechanical properties, including a tensile strength of 1.1 MPa, elongation of 1.33%, and elasticity of 0.87. Meanwhile, the formulation S3C1 (starch 30%; carrageenan 5%) had the best results for swelling, thickness, and biodegradability, with a swelling value of 57.79%, thickness of 0.36mm, and 11-day biodegradability.

Keywords: bioplastic, carrageenan, cassava peel, formulation, starch

INTRODUCTION

Plastic waste pollution has become the primary environmental issue and source of pollution that is discussed annually on a worldwide scale. The yearly growth in plastic usage parallels with the expansion of the food sector and the population. In the past three years, from 2020-2022, The Ministry of Environment and Forestry of Indonesia (2022) reported that plastic waste generation in Indonesia gradually increased. It increased by 4.7% from 2020 to 2021, amounting to 5.2 million tons, and then drastically increased by 23.2% in 2022, amounting to around 6.5 million tons. Indonesia is the second largest contributor to global plastic pollution after China (Jambeck et al., 2015) and is currently reported as the top country in the Southeast Asia region for around 30% contribution to land-based marine plastic litter (Gutberlet, 2023). Plastic waste harms the environment because it may impair soil fertility, contaminate the ocean, and threaten the existence of marine life. Furthermore, when plastic garbage is burnt, it releases CO₂ and HCN gas, contributing to global warming and air

pollution (Purwaningrum, 2016). Governments in most countries around the world have been tempted to implement the 3R concept (reduce, reuse, recycle) to minimize plastic waste (Evode et al., 2021; Knoblauch & Mederake, 2021). However, using eco-friendly plastic packaging or bioplastics is one of the options that may be adopted to support solving this issue.

Bioplastics are biodegradable plastics that are readily decomposed by soil microbes. They are made of polysaccharide compounds, such as starch, cellulose, protein, or lipid, which can be degraded by soil microorganisms (Jacoeb et al., 2014). Since bioplastics consist of organic compounds, they can replace synthetic plastic polymers and are more environmentally friendly. Additionally, the raw materials used to make bioplastics are abundant in nature and renewable (Maneking et al., 2020).

Due to its abundance, cassava peel waste has excellent potential as a raw material for bioplastics. FAO (2023) reported that from 2019 to 2021, cassava production in Indonesia showed an average positive

yearly growth of around 4.5%, reaching a production volume of 17.7 million tons in 2021. Cassava is commonly used in industrial production, especially for tapioca. Tapioca production generates solid waste in the form of cassava peel and its pulp during the cassava starch extraction process. Approximately 15-20% of cassava's weight becomes cassava peel waste (Kawijia et al., 2017). However, the current utilization of cassava peel waste is limited, usually used as animal feed or left to pile up, which can lead to unpleasant odors and environmental pollution (Suryati et al., 2017). Cassava peel waste contains high levels of starch, typically around 44-59% (Kawijia et al., 2017; Suryati et al., 2017), which makes it suitable for bioplastic production. Cassava peel waste is also potentially useful as an additional material in paper production (Kurnia et al., 2023). Polysaccharides such as starch and cellulose are essential raw materials for making bioplastics.

The use of cassava peel as a bioplastic has been reported in previous studies. It has been combined with additive materials for fillers, such as eggshell (Berghuis et al., 2022), microcrystalline cellulose (Maulida et al., 2016), CaCO₃ (Abidin, et al., 2021), and avocado seeds starch (Putri, 2019). However, the additives used in these studies result in low mechanical properties and the biodegradability of bioplastic. Also, they did not vary the concentration of cassava starch used as a treatment, making it difficult to determine the optimal concentration. Therefore, using other potential bioplastic additives, such as carrageenan, need to be investigated.

Carrageenan is one of the promising sustainable materials derived from marine red seaweed (*Eucheuma cottonii*) that is suitable for bioplastic application due to its biocompatibility, non-toxic, low-cost, good film-forming, and gelling properties (de Lima et al., 2020; Farhan & Hani, 2017; Ramesh & Velayudhaperumal, 2022; Sedayu et al., 2020a). Abdillah & Charles (2021) reported that the addition of carrageenan significantly improved the mechanical properties and swelling ability of arrowroot starch-based biodegradable film. Based on these studies, the combination of carrageenan and cassava peel waste starch will be potentially promising for producing low-cost bioplastics.

Although the combination of cassava peel starch and carrageenan has been reviewed as a potentially good and promising material for sustainable bioplastics (Fadhallah et al., 2023), further experiments on using those materials with various formulations have yet to be reported. Therefore, further research on the characteristics of bioplastics formulated from a combination of cassava peel waste starch and carrageenan at various concentrations will provide important information on the optimal concentration for using these materials. The purpose of this study was to determine the characteristics of

bioplastic made from cassava peel waste starch and carrageenan formulations.

EXPERIMENTAL SECTION

Materials and Equipment

The materials used in this study were cassava peel (*Manihot esculenta*), carrageenan flour (80 mesh, RP Chemicals), glycerol (RP Chemicals), citric acid, and aquadest. The cassava peel used in this study was the second layer of the outer peel with a white color. It was collected from the waste collection point in the tapioca starch factory located in Bangun Sari Village, Pesawaran, Lampung, Indonesia. The equipment used included a Universal Testing Instrument (Zwick Roel, Austria), digital caliper (RoHS, China), digital stirring hotplate (Cimarec+ Thermo Fisher, US), laboratory drying oven (Memmert UN55, Germany), and laboratory blender (Waring 8010 BU). Supporting tools such as digital scales, Pyrex chemical glassware, glass plates, knives, and a thermometer were also used.

Methods

The research used a two-factor randomized complete block design as the experimental design. The first factor consists of three levels of cassava peel starch (S) concentration (% w/v): 10% (S1), 20% (S2), and 30% (S3). The second factor consists of three levels of carrageenan (C) concentration (% w/v): 5% (C1), 10% (C2), and 15% (C3). Combining these factors resulted in nine formulations carried out under three replications.

Starch Extraction

The cassava peel waste starch was extracted using the wet extraction method, following Hasmadi et al. (2021) with slight modification. Cassava peel waste is cleaned and soaked in clean water, with the water being replaced every 15 minutes until it is clear of any physical contaminant. The peel was then immersed in water for 24 hours to eliminate HCN. The peel was then minced in a blender (the water-to-peel ratio was 2:1) and filtered with a filter cloth. After allowing the filtered water to precipitate for 60 minutes, starch was produced in the sediment. Once the water was removed from the starch, the starch was dried at 70 °C for 30 minutes and weighed with a digital scale. The yield of starch (expressed as a percentage, %) was calculated by dividing the weight of starch by the weight of cassava peel.

Preparation of Bioplastics

The synthesis of bioplastic was carried out using the solution casting method (Jacob et al., 2014). Starch and carrageenan in different formulations (Table 1) were mixed with 5 mL of glycerol, 2 mL of citric acid (1%), and distilled water until the solution reached 100 mL. The mixture was then stirred (150 rpm) and heated in a magnetic stirrer at 70 °C for 30 minutes. The solution was poured into a glass plate (10x10 cm)

and dried at 65 °C for 14 hours until a thin layer was formed. The thin film was placed at room temperature for two hours before being released from the plate. The bioplastic is further investigated, including mechanical characteristics, thickness, swelling, and biodegradation measurement.

Mechanical Properties Measurement

The observed mechanical properties of bioplastic include tensile strength, elongation, and elasticity. Elongation is the proportional change in film length when the film is pulled to break compared to its beginning length. The elasticity is related to elongation, where the higher the elongation value, the more elastic the material is. This elasticity value is calculated from the ratio between tensile strength and elongation of bioplastics (Hidayati et al., 2019). These properties were tested using a Universal Testing Instrument (Zwick Roel, Austria) following ISO 527-1: 2019 standard (ISO, 2019). The instrument will measure the specimen's grip, overall length, and thickness, and then the data will be recorded in the Zwick Roell software. The specimen was placed vertically and grasped on both sides and then tested by pressing the test button in the program on the computer. The test results will be given in MPa (for tensile strength values) and % (for elongation values) on the computer screen. The elasticity value is calculated by dividing the tensile strength value by the elongation value.

Thickness, Swelling, and Biodegradation Test

The thickness of bioplastic was measured at five points: upper right corner, lower right corner, upper left corner, lower left corner, and center using a digital caliper and expressed as mean thickness and standard deviation (Kawijia et al., 2017). The swelling and biodegradation test followed Alfian et al. (2020). Bioplastics were cut into 2x2 cm pieces and weighed their initial weight (W_0). They were then placed in a container containing 15 mL of distilled water for 10 minutes. After removing the sample and wiping the water on the surface with a tissue, the sample was weighed again. Weighing and soaking were repeated until the final weight was constant. The swelling test was calculated by comparing the difference between the initial and final weights with the initial weight and then multiplying by 100 percent. The biodegradation test was conducted to determine the time it takes for bioplastics to decompose. The test involved cutting 3x3 cm of bioplastic and placing them with an EM-4 (bioactivator) in a petri dish. Sample condition changes were observed and recorded daily, from intact until complete decomposition.

Data Analysis

The data obtained are statistically analyzed using the one-way ANOVA test and Duncan's posthoc test with a significance level of 0.05. The analysis was conducted using SPSS software version 23.

RESULTS AND DISCUSSION

Starch Yield

This research involves extracting 7.7 kg of unused industrial cassava peel waste, resulting in a starch yield of 444.59 grams, or approximately 5.77%. The result is similar to other studies by Wahyuningtyas et al., (2019) and Syuhada et al.,(2020), which reported that the yield of extracted starch from cassava peel is 5.97% and 5.71%, respectively. Starch yield extracted from cassava peel has a low value since the peel contains more cellulose, lignin, and hemicellulose than starch. The chemical properties of cassava peel consist of 37.9-40.5% cellulose, 21.4%-23.9% hemicellulose, 7.5-11.7% lignin, and other substances such as protein and minerals (Otache et al., 2017; Widiarto et al., 2019). The cassava peel starch color obtained from this study is light brown or beige. The yellow-brown color of cassava peel results from the enzymatic browning reaction, which occurs when cassava peel polyphenol compounds come into contact with oxygen and are catalyzed by the enzyme polyphenol oxidase (PPO) (Selvarajan et al., 2018).

Thickness

The thickness test is carried out since it correlates with water resistance (swelling) and tensile strength tests. Water resistance and tensile strength will rise proportionally with the thickness of the bioplastic. The thicker the bioplastic produced, the higher the values of water resistance and tensile strength will be, as reported by (Hasri et al., 2021). The thickness value is expressed as average and standard deviation (**Figure 1**). Different letters indicate significant differences based on the Duncan Test at the $\alpha = 5\%$ level. The thickness of S2C1, S2C3, and S3C2 were significantly different from other treatments, while S1C1, S1C2, S1C3, S2C2, S3C1, and S3C3 were not significantly different from each other.

Formulation of cassava peel waste and carrageenan has a significant effect on the thickness value ($\text{sig} < 0.05$). The bioplastic thickness test data shows that the S2C1 treatment has the highest thickness of 0.66 mm. However, the thickness of the bioplastic in this study has yet to meet the standard of JIS Z 1707, which specifies a maximum thickness value of 250 μm or 0.25 mm (Japanese Standard Association, 2019). The formulation of carrageenan and cassava peel starch significantly affects the thickness of the bioplastic, with a higher concentration of carrageenan or starch resulting in a thicker product. This phenomenon is affected by the total amount of soluble solids in the mixed carrageenan solution at the higher concentration, making the solution more viscous. Cisneros-Zevallos & Krochta (2003) explained that the higher viscosity of solution results in the higher thickness of films. As the solution viscosity increases, the flow behavior and film-forming capabilities of the solution change, resulting in the deposition of a thicker solution layer during the casting and drying process. According to Supeni (2012), the thickness of bioplastic

tends to increase with higher concentrations of carrageenan. Manuhara et al. (2016) and Sedayu et al. (2020b) reported the same results that the addition of 1% and 5% concentration of carrageenan significantly increased the thickness of films by 0.02-0.06 mm.

Swelling and Biodegradation

The swelling test was conducted to determine the bioplastics' ability to absorb water, while a biodegradation test was carried out to assess the sample's degradation process (Solekah et al., 2021). The results are presented in **Table 1**.

The results of statistical tests (ANOVA) showed that the combination of starch and carrageenan formulations did not significantly affect the swelling value of bioplastics (Sig > 0.05). The swelling value in this study varied from 28.72-58.09%. This result is similar to Wang et al. (2023), which reported swelling values ranging from 14.67 to 25.09%. Moustafa et al. (2021) also reported that carrageenan-potato starch

blend polymers have swelling value from 33.41 to 54.35%, whereas the higher ratio of starch and carrageenan was not significantly different. Wittaya (2012) stated that the swelling properties of starch-based bioplastics are mainly affected by amylose. It is suspected that each bioplastic formulation in this study contains the same amylose content; hence, the swelling value did not vary significantly. Cassava peel starch previously reported has a low amylose content of around 9.6-21.02% or amylose-amylopectin ratio 1:5 (Fronza et al., 2023; Mudaffar, 2021; Wasistha et al., 2021). Jacob et al. (2014) explained that the thickness and matrix density of bioplastics could influence water absorption, the thicker bioplastics with high matrix density can reduce water vapor transmission, making it more difficult for water to penetrate. A high water absorption value in the swelling test indicates that the bioplastics will decompose more quickly in the environment and dissolve more easily in water.

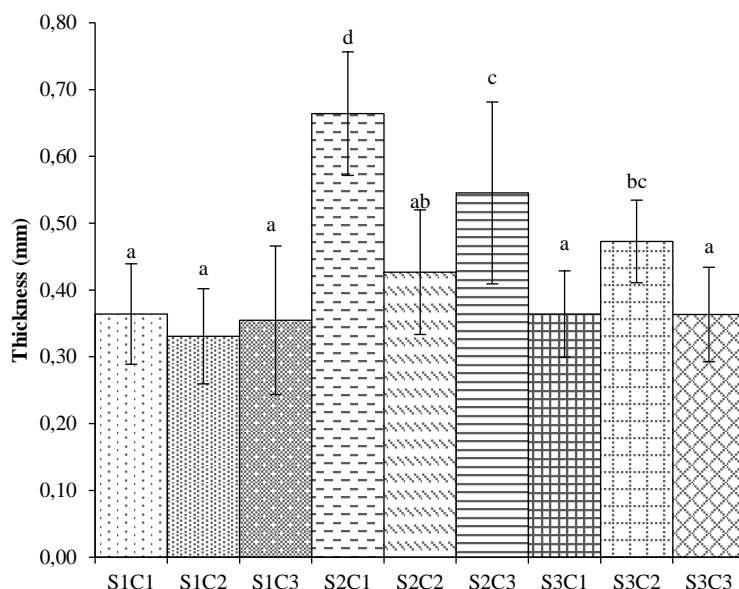


Figure 1. Thickness value of bioplastics from cassava peel starch (S) and carrageenan (C) formulations.

Table 1. Swelling and biodegradation of bioplastics

Formulations	Swelling (%)	Degradation (days)
S1C1 (starch 10%, carrageenan 5%)	32.28±4.56	15
S1C2 (starch 10%, carrageenan 10%)	39.56±6.71	15
S1C3 (starch 10%, carrageenan 15%)	28.72±7.21	11
S2C1 (starch 20%, carrageenan 5%)	33.38±3.80	13
S2C2 (starch 20%, carrageenan 10%)	54.60±4.13	12
S2C3 (starch 20%, carrageenan 15%)	41.19±6.19	12
S3C1 (starch 30%, carrageenan 5%)	57.79±5.12	11
S3C2 (starch 30%, carrageenan 10%)	43.48±3.58	13
S3C3 (starch 30%, carrageenan 15%)	58.09±6.82	15

Based on the results of the biodegradation test, it was found that bioplastics made from cassava peel starch with carrageenan addition were easily and quickly degraded within 11 to 15 days. The S1C3 and S3C1 treatments, treated with EM4 fermentation bacteria, showed the fastest degradation for 11 days. This finding shows a faster degradation compared to another study (Alfian et al., 2020), which reported 35-45 days of degradation of edible film. Syuhada et al. (2020) also reported that bioplastic made from cassava peel and chitosan did not fully degrade in 14 days, with 62.95% degradation in soil media and 53.69% in river water media. The faster degradation time of the cassava peel starch bioplastics indicates that they are more environmentally friendly and easily decomposable. Starch is a natural polymer that is easily degraded by nature and other decaying bacteria, making it a suitable material for producing bioplastics. According to Emadian et al. (2017), the degradation of bioplastic based on organic material, such as starch, can quickly degraded by the soil bacteria by breaking down the polymer chain into its monomers, causing physical changes such as size reduction until it is destroyed. The biodegradability of bioplastics made from cassava peel starch makes them an attractive alternative to traditional plastic materials, known to persist in the environment for a long time and contribute to pollution.

Tensile Strength

The tensile strength test aims to determine the maximum amount of stress a material can resist before breaking. Several factors can influence the tensile strength value, such as starch concentration and glycerol (Arini et al., 2017). The results of the tensile strength test are presented in **Figure 2**. The tensile strength value is expressed as average and standard deviation (**Figure 2**). Different letters indicate significant differences based on the Duncan Test at the

$\alpha = 5\%$ level. The combination of 20% starch and 5% carrageenan (S2C1) showed the highest tensile strength, which was significantly different from all combinations.

Statistical test results (ANOVA) showed that the combination of starch and carrageenan formulations significantly affected the tensile strength of bioplastics (Sig < 0.05). **Figure 3** shows that the tensile strength value increased significantly at the higher starch and carrageenan concentrations. This is due to the formation of a greater film matrix structure due to the interaction of starch gelatinization and carrageenan gelation ability, producing a more robust and extensive matrix that contributes to the overall increased tensile strength. Moreover, Tao et al. (2022) explained that carrageenan contributes to an improvement in blended film's tensile strength via abundant hydroxyl group molecules that form hydrogen bonds from intramolecular and intermolecular with others, such as starch (Cheng et al., 2022), making a greater bonding with carrageenan. Similarly, Abdillah and Charles (2021) reported that adding arrowroot starch in carrageenan films resulted in a significant increase in tensile strength compared with pure carrageenan without starch addition. Suryanto et al. (2019) reported that adding 5% carrageenan increases the tensile strength significantly of cassava starch-based bioplastic made by the extrusion process from 0.22 to 1.02 MPa. Wasistha et al. (2021) used a higher concentration of cassava peel starch (6%) in carboxymethyl cellulose-based films and reported a significant increase of tensile strength from 0.08 MPa (2% cassava peel starch) up to 0.37 MPa. Generally, the tensile strength value of the bioplastics in this study meets the standard of JIS Z 1707, which specifies a minimum tensile strength of 0.392 MPa (Japanese Standard Association, 2019).

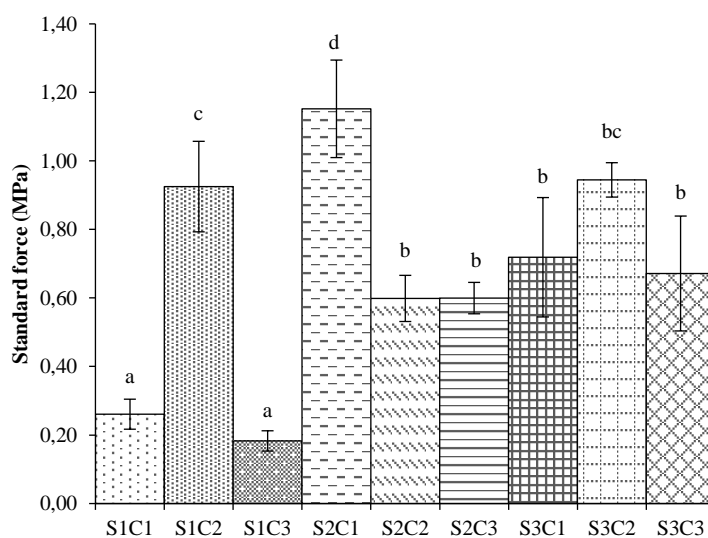


Figure 2. Tensile strength value of bioplastics from cassava peel starch (S) and carrageenan (C) formulations..

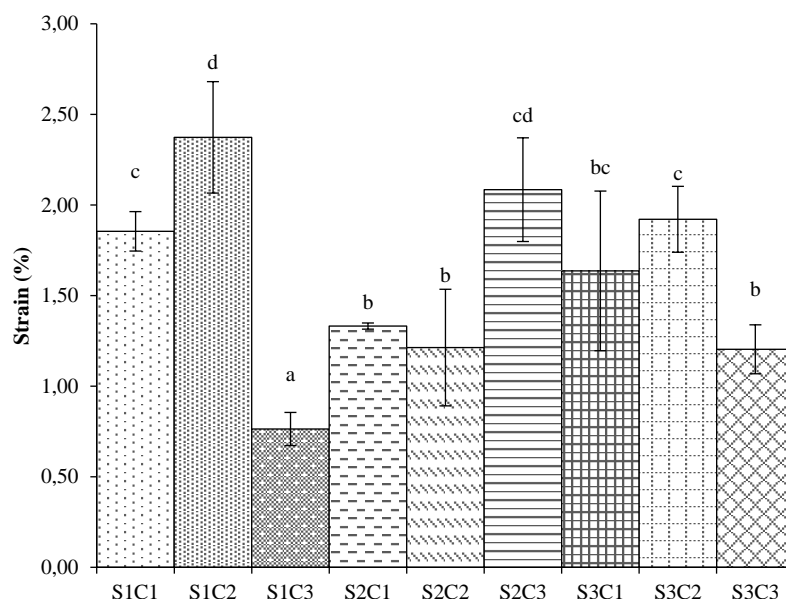


Figure 3. Elongation value of bioplastics from cassava peel starch (S) and carrageenan (C) formulations.

Elongation

The elongation test measures the stretchability of bioplastics before they break (Muhammad et al., 2020). The results of the elongation test are presented in **Figure 3**. The elongation value is expressed as average and standard deviation (**Figure 3**). Different letters indicate significant differences based on the Duncan Test at the $\alpha = 5\%$ level. The combination of 10% starch and 10% carrageenan (S2C1) showed the highest elongation, significantly different from the higher concentration of carrageenan (S1C3), which showed the lowest elongation. A similar phenomenon can be seen on S3C2 and S3C3, where the higher concentration of carrageenan also significantly decreases the elongation.

Figure 3 shows that the concentration of carrageenan and cassava peel starch significantly affects the elongation of bioplastics (Sig < 0.05). The highest elongation value was obtained in treatment S1C2, which consists of 10% starch and 10% carrageenan (2.37%), and the lowest value was found in treatment S1C3, which consisting of 10% starch and 15% carrageenan (0.76%). These results indicate that the formulation of carrageenan with a 1:1 to cassava peel starch ratio has a high elongation value. However, the bioplastic with a higher concentration of carrageenan and starch tends to have a lower elongation value. This phenomenon can happen due to an increase in the number of molecular interactions between the polymer chains at higher concentrations of starch and carrageenan. As a result, a denser and more tightly packed molecular network forms inside the bioplastic, limiting its capacity to stretch or elongate under tensile stress, causing a decrease in the elongation value. In this study, the elongation value tends to be the opposite of the tensile strength

value. According to Putri et al. (2023), starch-based films have high tensile strength but low elongation due to the formation of amylose crystals after starch is gelatinized and retrograded during the drying process. This crystal results in stronger tensile strength but makes it brittle and hard to stretch. Wasistha et al. (2021) also reported a decrease in elongation value from 56.43 to 31.86% using the higher concentration of cassava peel starch. Abdullah et al. (2021) showed the same trends where the elongation of starch-CMC-based films decreased drastically as the carrageenan concentration increased, from 28.89% (0% carrageenan) to 14.78% (10% carrageenan). The elongation value of the bioplastics in this study was categorized as "poor" according to JIS z 1707, since the elongation value of all formulations was below 10%. This result is quite far from the "good" category, which specifies a minimum elongation value greater than 70% (Japanese Standard Association, 2019). Therefore, it needs to be improved using a variation of plasticizer concentration.

Elasticity

The elasticity test determines the elasticity value of a material or bioplastic produced. The elasticity value is obtained by comparing the tensile strength and elongation values (Situmorang et al., 2019). The results or values of the elasticity test are presented in **Figure 4**. The elasticity value is expressed as average and standard deviation (**Figure 4**). Different letters indicate significant differences based on the Duncan Test at the $\alpha = 5\%$ level. The combination of 20% starch and 5% carrageenan (S2C1) showed the highest elasticity, which is significantly different from all other combinations, including S1C1 with the lower starch concentration, which had the lowest elasticity value.

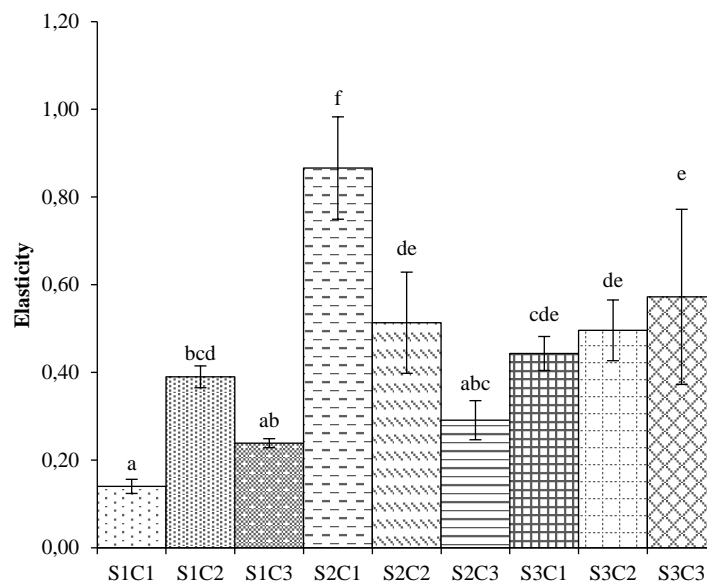


Figure 4. Elasticity value of bioplastics from cassava peel starch (S) and carrageenan (C) formulations.

Figure 4 shows that the concentration of carrageenan and cassava peel starch significantly affects the elasticity of bioplastics (Sig < 0.05). The formulation S2C1, which consists of 20% starch and 5% carrageenan, has the highest elasticity value compared with S1C1 (10% starch and 5% carrageenan), which produces the lowest elasticity value. The difference between these formulations lies in the starch concentration, where a higher concentration results in higher elasticity. Also, the finding of this study shows that the interaction of cassava peel starch and carrageenan tends to result in a higher elasticity value. The higher concentration of starch and carrageenan encourages the formation of more cross-linking sites between the polymer chains, improving the bioplastic's structural integrity. This increased cross-linking allows the material to resist stretching and deformation while maintaining its shape and structure, leading to an overall improvement in the bioplastic's elasticity. Khalil et al. (2017) explained that in carrageenan-based films, the elasticity was increased due to the contribution of carrageenan ability, which tends to create an elastic structure via the aggregate gel process. Moustafa et al. (2021) reported the same trend results, where the blend films of carrageenan and potato starch have a higher elasticity than the carrageenan films without potato starch addition. A similar trend was reported by Suryanto et al. (2019), which shows a significant increase in elasticity as the carrageenan concentration rises, from 0.77 to 5.47 (using 5% carrageenan) and 9.55 (using 10% carrageenan). Abdullah et al. (2021) reported the same phenomenon where the higher concentration of carrageenan results in a significant rise of elasticity value from 4.99 (0% carrageenan) up to 11.89 (15% carrageenan) in a starch-CMC-based

bioplastic. According to JIS Z 1707, the elasticity value of sample formulations with 20% and 30% starch with the variation of carrageenan has met the standard, which specifies a minimum value of 0.35 (Japanese Standard Association, 2019).

CONCLUSIONS

The formulations of cassava peel waste starch and carrageenan concentration significantly affected bioplastic's mechanical properties such as thickness, tensile strength, elongation, and elasticity, but not swelling and biodegradation. Higher starch and carrageenan concentrations increase thickness, tensile strength, and elasticity but decrease elongation value. In this study, bioplastic has a thickness ranging from 0.33 to 0.66 mm, a swelling value from 28.72 to 58.09%, and degrades in 11 to 15 days. Tensile strength ranges from 0.18 to 1.15 MPa, elongation from 0.76 to 2.37%, and elasticity from 0.14 to 0.87. It meets JIS Z 1707 standards for some mechanical parameters (tensile strength and elasticity), indicating potential use for dry seasoning packaging. Further study is needed to assess various plasticizers at optimal concentrations to meet the standard for food packaging criteria.

ACKNOWLEDGEMENTS

The author expresses gratitude to the Institute of Research and Community Services (LPPM) of Universitas Lampung for funding this research through the Basic Research Grant in 2023.

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