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The Influence of Carrageenan and Sodium Tripolyphosphate Addition on the Characteristics of Cassava Peel Starch Biodegradable Films

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Abstract:- Plastics, the most widely used packaging material, have become a major environmental concern due to their non-biodegradability and adverse effects on ecosystems. To address this issue, biodegradable films made from natural starch, such as cassava peel starch, have emerged as eco-friendly alternatives. This study investigates the impact of incorporating carrageenan and sodium tripolyphosphate (STPP) into cassava peel biodegradable films. Carrageenan enhances tensile strength, while STPP improves the film matrix. Tensile strength increases with higher starch concentrations but decreases elongation. FTIR and SEM tests confirm the formation of new functional groups and denser structures, meeting quality standards. These findings provide a foundation for optimizing cassava peel starch edible film formulations. The research utilized a variety of tests and analyses, including tensile strength, elongation, FTIR, and SEM, to explore the mechanical properties and structural changes in cassava peel starch biodegradable films. The results demonstrated the potential for these films to serve as sustainable alternatives in packaging and various applications.

Keywords:- *Biodegradable Films, Starch-based Films, Cassava Peel Waste, Carrageenan, Sodium Tripolyphosphate (STPP), Tensile Strength, Elongation, FTIR, SEM.*

I. INTRODUCTION

Plastic is the most widely used packaging material, and its use continues to increase with the growing population. Plastic possesses several advantages, including flexibility, durability, cost-efficiency, transparency, heat resistance, stability, and the ability to be combined with various materials [1]. However, after use, plastic waste is often disposed of improperly, leading to environmental pollution. Modern plastics, primarily derived from petroleum, are challenging to degrade quickly [2]. The accumulation of plastic waste on the ground can result in soil pore clogging, hindering water absorption and potentially causing flooding. Moreover, plastic waste accumulation negatively impacts marine ecosystems and, aesthetically, creates an unpleasant environment [3].

The escalating environmental pollution caused by non-degradable plastics has sparked an interest in finding eco-friendly alternatives. One solution is the use of biodegradable films made from biopolymers such as starch. Biodegradable films serve various functions, including inhibiting vapor and gas exchange, preventing fat migration, aroma retention, improving physical characteristics, and carrying additives [4]. Several polysaccharides that can be used to produce biodegradable films include root tuber starch, pectin, alginates, carrageenan, and cellulose.

Starch is one of the biopolymers that has gained significant attention for developing environmentally friendly plastic films. Starch exhibits attractive properties, such as good film-forming ability and strong barrier properties [5]. In Indonesia, cassava (Manihot esculenta) is a widely available tuber. However, cassava peel waste from tapioca flour industries often becomes an environmental pollutant if not properly processed. This waste contains starch and can potentially be used as a raw material for producing biodegradable films.

Biodegradable films derived from natural starch tend to have hydrophilic properties, affecting their mechanical characteristics, such as a tendency to hydrate, easy expansion, fragility, and mechanical weakness [6]. To improve the mechanical properties of the films, some research has explored blending with other materials, chemical modification, or a combination of various techniques.

Carrageenan, sourced from red seaweed, is one of the polymer compounds that have the potential to enhance the mechanical properties of starch-based biodegradable films. Carrageenan possesses a strong gel structure, which is expected to improve the tensile strength and elongation of films [7]. Previous research has shown that the addition of carrageenan can enhance the mechanical properties of starch films.

In addition to carrageenan incorporation, starch modification through cross-linking using sodium tripolyphosphate (STPP) can also affect the mechanical properties of films. STPP can increase the tensile strength of the films, viscosity, and pasta stability, resulting in a stronger film matrix [8].

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Although much research has explored various aspects of biodegradable films, the impact of carrageenan addition and starch modification with STPP on cassava peel biodegradable films has been underexplored. Therefore, this study aims to investigate the effects of carrageenan addition and starch modification with STPP on cassava peel biodegradable films. Carrageenan addition is expected to enhance film tensile strength and elongation, while starch modification with STPP is expected to yield a stronger film matrix.

II. MATERIALS AND METHODS

A. *Materials*

The materials used in the research consist of raw materials and chemicals, along with their specifications, source, and functions, as presented in Table 1.

Table 1 Materials and Specifications

Materials	**Specification**	**Origin/Brand**	**Function**
Cassava peels	Fresh cassava peels	Ngemplak Village, Margoyoso District, Pati Regency, Central Java	Material
Carrageenan	Kappa carrageenan, semirefined	IndoGum Indonesia	Gelling agent
Glycerol	Colorless, liquid, 99% putity	Indrasari Chemist Shop, Semarang, Jawa Tengah	*Plasticizer*
STPP	Food grade	Aditya Birla Chemicals, Thailand	*Crosslinking agent*
Distilled water	Mineral free	Purelizer	Solvent

B. *Equipment*

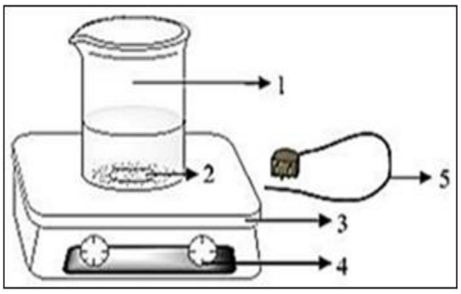

Fig.1 Equipment Setup

➢ *Description:*

- *Beaker glass*
- *Magnetic stirrer*
- *Heaters*
- *Stirrer speed button*
- *Cable*
- *Thermometer*

C. *Variable*

➢ *Fixed Variables:*

- Gelatinization Temperature: 80°C
- Solvent Type: Distilled water
- Glycerol Volume: 2.5 ml

➢ *Independent Variables:*

- Starch Concentration: 5; 10; 15 g/100 ml of solvent
- Carrageenan Concentration: 0.5; 1; 1.5%
- STPP Concentration: 0.5; 1; 1.5%

D. *Research Procedure*

➢ *Production of Cassava Peel Starch*

The production of cassava peel starch was carried out based on the research by Wasistha et al [9]. 100 grams of cassava peels were washed with running water and soaked in water, which was changed every 15 minutes until the soaking water became somewhat clear. Subsequently, they were soaked in water for 24 hours. The purpose of washing and soaking was to remove impurities attached to the cassava peels, reduce the HCN content in the peels, and reduce the adhering latex. The cassava peel was then crushed using a blender, with the addition of 100 ml of water to facilitate the crushing process. The resulting cassava peel slurry was filtered and left for 30 minutes to obtain the precipitate from the cassava peel slurry. After 30 minutes, the precipitate was separated from the water, dried in an oven, and then crushed using a blender and filtered.

➢ *Production of Biodegradable Starch Film – Carrageenan*

Gelatinization was prepared by dissolving cassava peel starch according to the concentration and carrageenan according to the concentration in 100 ml of distilled water. The mixture was heated using a magnetic stirrer to reach 80°C for 30 minutes. Glycerol (2.5 ml) was added to the solution and stirred until homogenous. The solution was then cooled to the desired operating temperature of 50°C. Each film solution was cast on an acrylic glass and dried for 14 hours in an oven at 60°C. Subsequently, mechanical properties such as tensile strength, elongation, thickness, and solubility were analyzed. For comparison, biodegradable pure starch films were also made without the addition of carrageenan and STPP under the same conditions.

- *Production of Biodegradable Starch Film – STPP*

Gelatinization was prepared by dissolving cassava peel starch according to the concentration in 100 ml of distilled water. The mixture was heated using a magnetic stirrer to reach 80°C for 30 minutes. Glycerol (2.5 ml) was added to the solution and stirred until homogenous. The solution was then cooled to the desired operating temperature of 50°C. Subsequently, the mixture was added to STPP according to the concentration and homogenized, maintaining the specified operating temperature for 10 minutes. Each film solution was cast on an acrylic glass and dried for 14 hours in an oven at 60°C. Further characterization of the film included tensile strength, SEM, and FTIR analysis for the formulation with the highest tensile strength.

E. Results Analysis

After the process of producing biodegradable plastic is complete, several characteristic tests on the plastic are conducted.

- *Tensile Strength Analysis*

The analysis of mechanical properties of the material consists of tensile strength and elongation to measure the tensile strength of the biodegradable film products produced using an Instron Universal Testing Machine. The method used to analyze the tensile strength and elongation values follows the research by Putra et al [10]. Samples, cut to a length of 8 cm and a width of 3 cm, are placed on the machine, specifically under the top plate, and clamped in the holding grips. The samples are secured by rotating the handwheel to ensure that the clamping is secure. The Universal Testing Machine is then activated, and it pulls the sample until it breaks. The tensile test is performed by pulling the film from two directions, causing it to lengthen and narrow. The force and length increase are recorded during the test. Tensile strength indicates the maximum force produced when a tensile test is conducted. Tensile strength is the maximum weight the film can withstand, according to the equation (1) below.

$$TS = \frac{F}{A} \tag{1}$$

TS = Tensile Strength
F = Maximum force
A = Initial surface area

- *Elongation Analysis*

The percentage of elongation represents the film's elasticity when subjected to tensile forces until it breaks, resulting in the maximum change in length [11]. The measurement of elongation or percentage elongation is also performed by pulling both ends of the film using the Instron Universal Testing Machine, causing the length to increase. The difference between the initial and final lengths divided by the initial length of the film yields the percentage elongation of the film, with the following mathematical calculation (2).

$$\%Elongation: \frac{L - L0}{L} \; x \; 100\% \tag{2}$$

L = Initial length
L0 = Final length

- *FTIR Analysis*

The FTIR analysis is conducted to identify the functional groups present in the biofilm. FTIR testing is performed using infrared spectroscopy. The samples are placed in a set holder, and the corresponding spectrum is obtained. The result will yield a diffractogram showing the relationship between wavenumber and intensity.

- *SEM Analysis*

The SEM (Scanning Electron Microscopy) analysis is conducted to observe the morphology of the produced biodegradable film.

III. RESULTS AND DISCUSSIONS

A. Tensile Strength Analysis

Tensile strength is a physical property of the biofilm that is related to its strength in resisting film damage during packaging. A higher tensile strength value is expected to withstand physical damage, minimizing product damage during packaging [12].

Table 2 Biofilm Tensile Strength Value

Sample Code	Concentration			Responds
	Starch (gr/100 ml)	Carrageenan (%)	STPP (%)	Tensile strength (Mpa)
P1K1	5	0,5	0	2,33
P1K2	5	1	0	2,37
P1K3	5	1,5	0	2,42
P1S1	5	0	0,5	2,24
P1S2	5	0	1	2,27
P1S3	5	0	1,5	2,21
P2K1	10	0,5	0	2,57
P2K2	10	1	0	2,69
P2K3	10	1,5	0	2,85
P2S1	10	0	0,5	2,43
P2S2	10	0	1	2,51
P2S3	10	0	1,5	2,33

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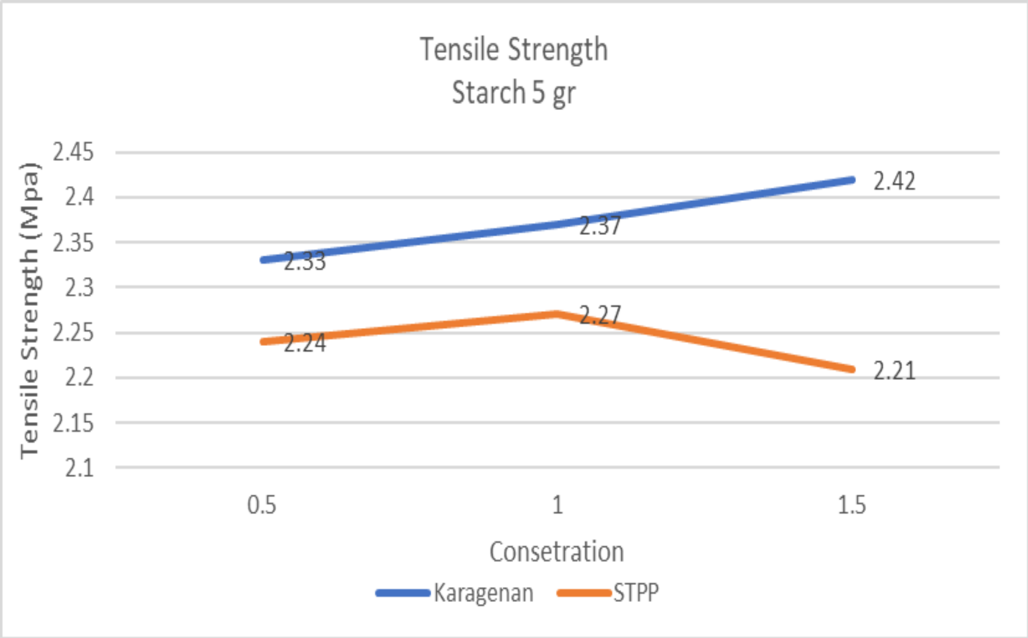
Sample Code	Concentration			Responds
	Starch (gr/100 ml)	Carrageenan (%)	STPP (%)	Tensile strength (Mpa)
P3K1	15	0,5	0	2,97
P3K2	15	1	0	3,09
P3K3	15	1,5	0	3,17
P3S1	15	0	0,5	2,61
P3S2	15	0	1	2,74
P3S3	15	0	1,5	2,48

At different starch concentrations, different tensile strength values were obtained. The higher the starch concentration, the higher the tensile strength of the produced biofilm. This is because as the starch concentration increases, the amylose content in the biofilm solution also increases. This leads to a greater number of polymer chains in the matrix formation and stronger polymer interactions, resulting in a higher tensile strength.



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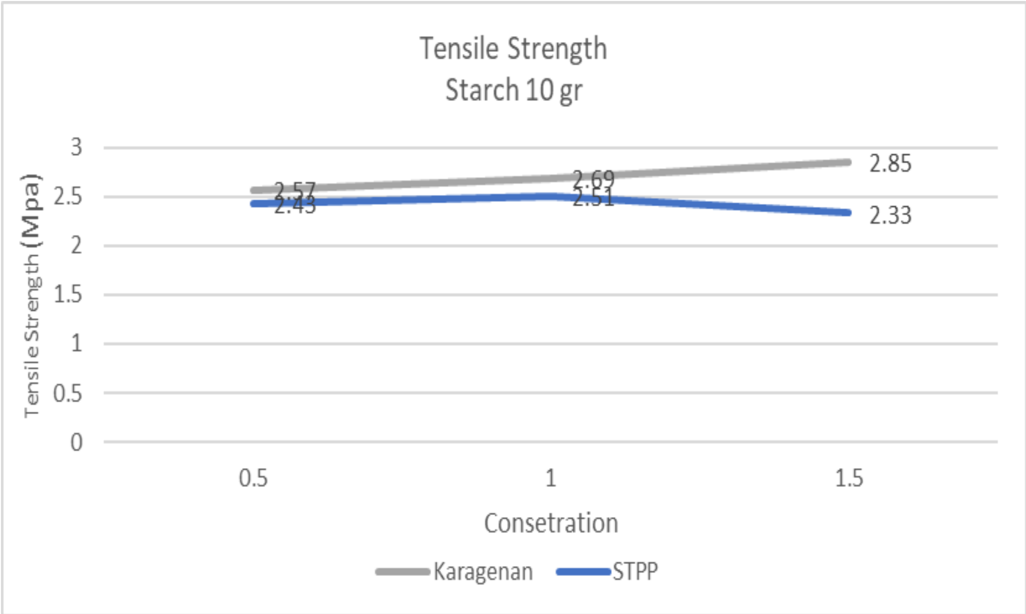

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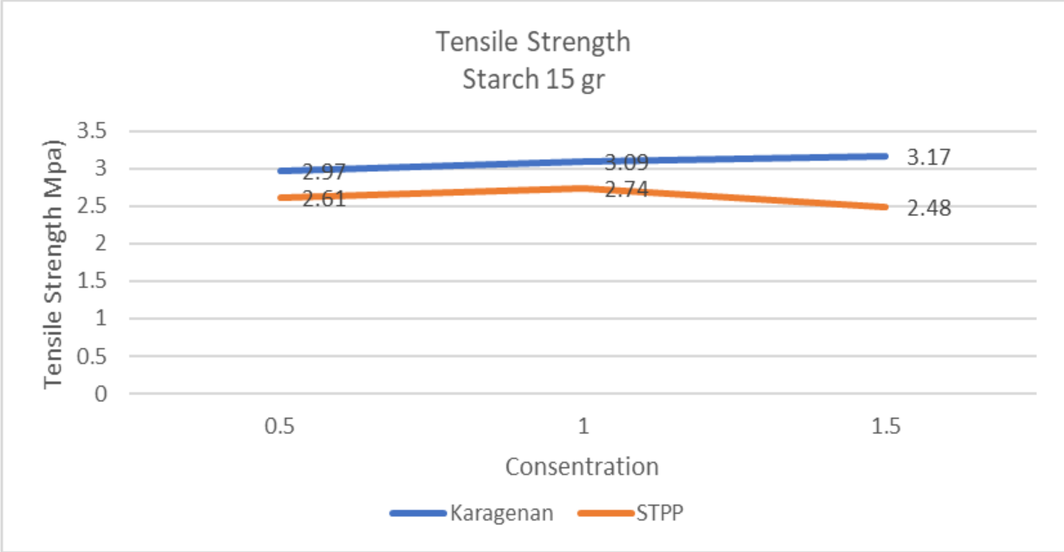

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Fig. 2 Graph of Tensile Strength Values of Starch Biofilm (a) 5 grams (b) 10 grams (c) 15 grams

The tensile strength is directly proportional to the amount of carrageenan added. This is because increasing the amount of carrageenan adds structural integrity to the film matrix and strengthens intramolecular forces, thus requiring greater force to break the film. The addition of carrageenan in the film solution enhances the intermolecular bonds, resulting in a more complex film solution with higher tensile strength. Carrageenan has the capability to form a strong polymer matrix, reinforcing the intermolecular tensile strength in the film [13].

The increase in tensile strength of the film is achieved through cross-linking density, as the reaction between hydroxyl groups and cross-linking agents forms hydrogen bonds [14] [15]. Similar results were reported by Kaur et al. [16] in which the tensile strength of modified corn starch edible films was higher compared to unmodified corn starch. Technically, the optimal concentration of cross-linking agents is essential in determining the mechanical characteristics of modified starch films. Low concentrations of cross-linking agents are insufficient for intermolecular cross-linking, and high concentrations result in excessive cross-linking that limits the mobility of starch molecules, potentially reducing tensile strength [17] [14] [18].

Comparing this research to Wasistha et al.'s [19] study on biodegradable films made from cassava skin starch, the tensile strength in their study ranged from 0.08 to 0.37 MPa. Fathanah et al. [20] also conducted research on bioplastic made from cassava skin starch and chitosan with sorbitol as a plasticizer, achieving the highest tensile strength of 1.37 MPa. According to the Japan Industrial Standard [21], the minimum tensile strength for biofilms is 0.39 MPa. The results of the tensile strength in this study meet the standards and are higher than previous research.

B. Elongation Analysis

Elongation is the percentage change in length of a film when it is stretched until it breaks [13]. The measurement of elongation is conducted to determine the flexibility of the resulting film. A higher elongation value indicates greater flexibility in the use of the film for product packaging.

Table 3 Biofilm Elongation Values

Sample Code	**Concentration**			**Responds**
	Starch (gr/ 100ml)	**Carrageenan (%)**	**STPP (%)**	**Elongation (%)**
P1K1	5	0,5	0	49,8
P1K2	5	1	0	45,5
P1K3	5	1,5	0	42,4
P1S1	5	0	0,5	65,7
P1S2	5	0	1	63,1
P1S3	5	0	1,5	67,4
P2K1	10	0,5	0	43,3
P2K2	10	1	0	40,1
P2K3	10	1,5	0	37,5
P2S1	10	0	0,5	60,7
P2S2	10	0	1	58,4
P2S3	10	0	1,5	63,5
P3K1	15	0,5	0	38,9
P3K2	15	1	0	35,3

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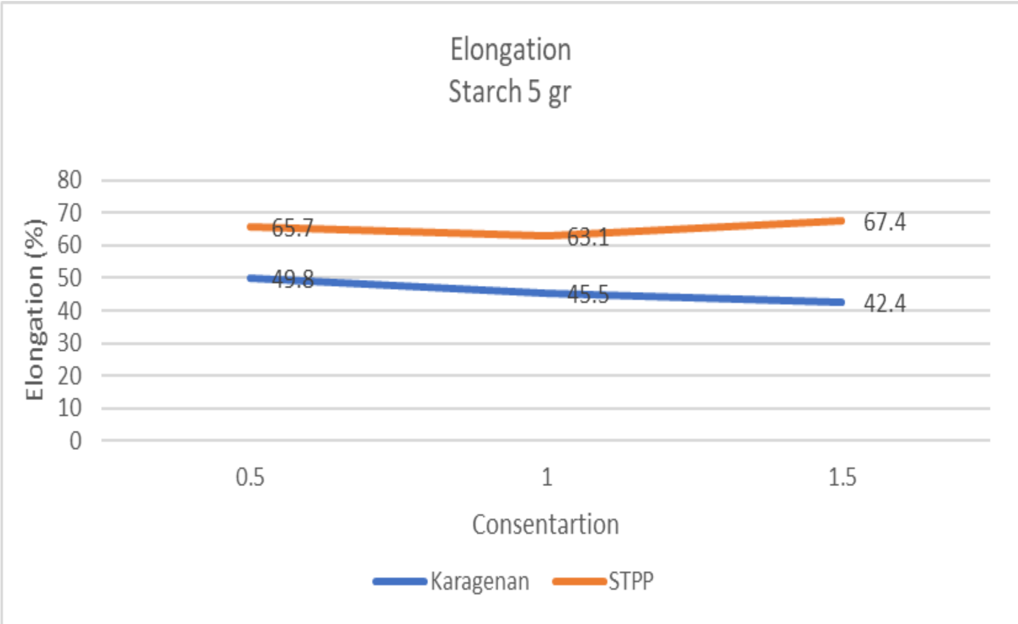
Sample Code	Concentration			Responds
	Starch (gr/ 100ml)	**Carrageenan (%)**	**STPP (%)**	**Elongation (%)**
P3K3	15	1,5	0	33,6
P3S1	15	0	0,5	55,7
P3S2	15	0	1	53,5
P3S3	15	0	1,5	58,9

The results indicate that elongation is inversely related to tensile strength. A higher tensile strength value will lead to a smaller elongation. Conversely, a decrease in tensile strength will increase elongation [22]. This observation aligns with Dick et al. [23], who reported that a decrease in tensile strength from 17.75 MPa to 9.44 MPa resulted in an increased elongation value from 1.93% to 10.78%. The amylose content in starch contributes to the elongation value of the film [24]. A high percentage of elongation signifies that the film has greater flexibility, making it easy to apply and extending its shelf life.



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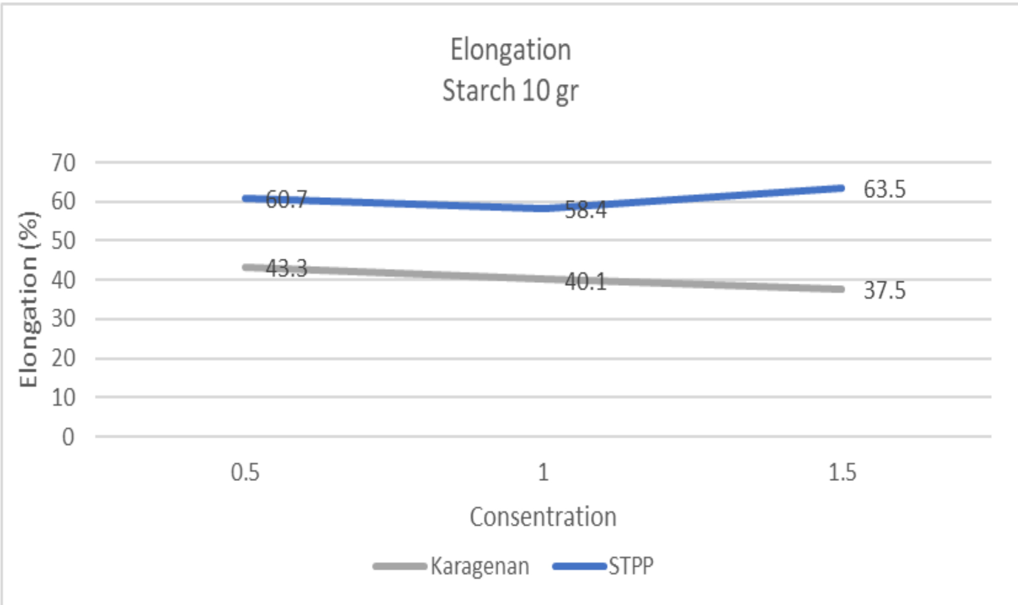

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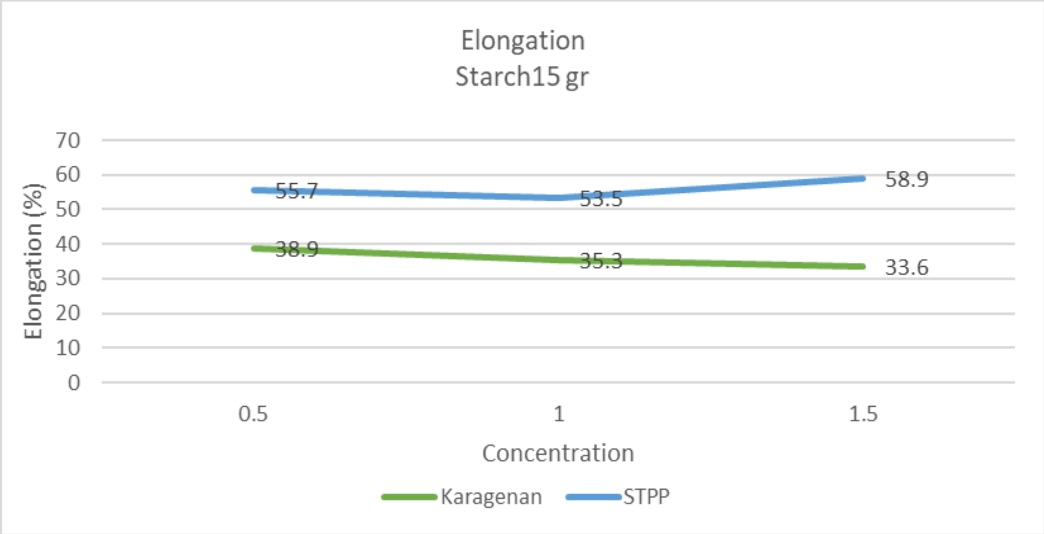

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Fig.3 Graph of Starch Biofilm Elongation (a) 5 grams (b) 10 grams (c) 15 grams

Increasing the concentration of starch reduces the elongation of the film because the film becomes stronger and more brittle. This is consistent with previous research. A high concentration of carrageenan makes the film non-elastic and difficult to stretch due to tight molecular bonds. The addition of STPP does not significantly affect elongation due to reactions between starch molecules. Despite the decrease in elongation, the values in this study still meet the quality standards for the film.

C. FTIR Analysis

The purpose of characterizing biofilms using FTIR is to investigate the influence of carrageenan and STPP. FTIR analysis is employed to determine the presence of functional molecular groups within the biofilm.

Based on the FTIR spectrum, several peaks are observed at specific wavenumbers, and the identification results of the functional groups in each fraction are compared to reference data. The samples analyzed include pure starch, carrageenan, and STPP. The FTIR analysis results indicate the successful identification of functional groups with similar characteristics. This suggests that the fractionation of amylose with the influence of three different concentrations of butanol does not significantly alter the functional groups, as the functional groups of amylose and amylopectin are identical.

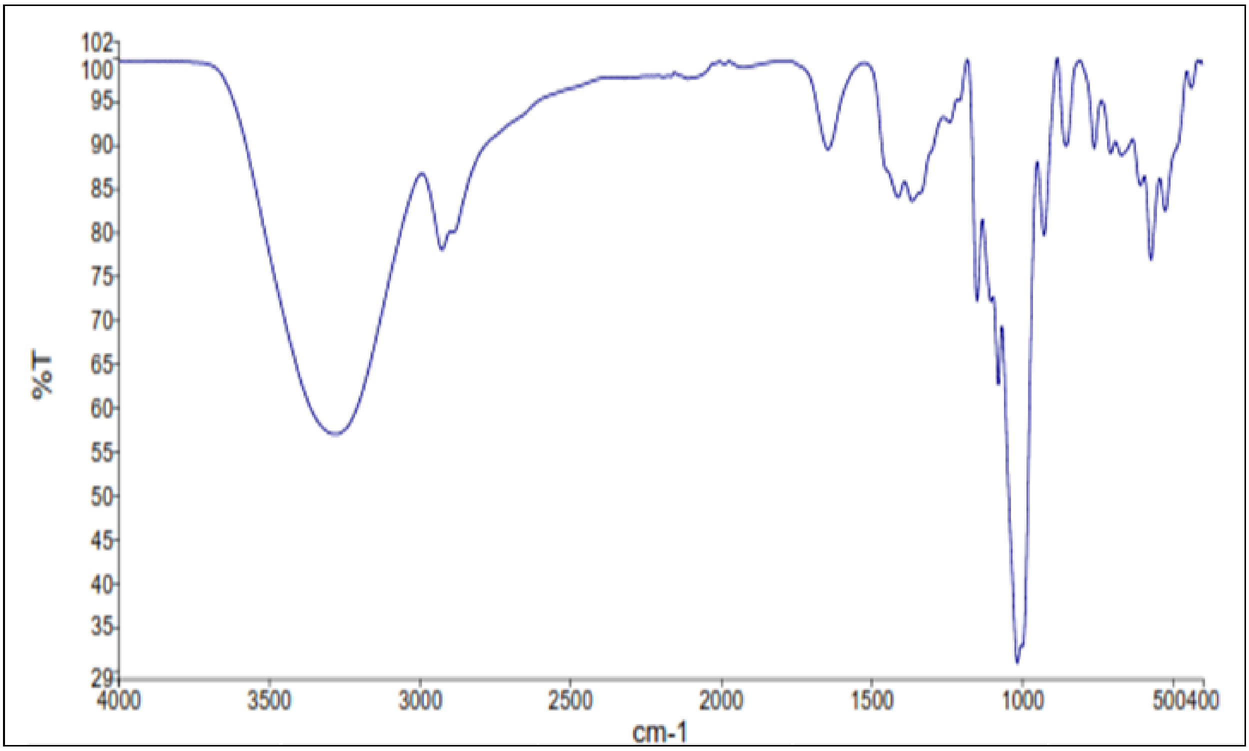


Fig 4 FTIR Spectrum of Cassava Peel Starch

Based on Figure 4, the results of functional group identification using FTIR show absorption patterns at various wavenumbers. The functional groups identified in cassava peel starch include the O-H alcohol group at the wavenumber of 3289.99 cm^{-1}, the long-chain hydrocarbon C-H alkane group at the wavenumber of 2928.54 cm^{-1}, reinforced by the identification of bending vibrations at the wavenumbers of 1412.72 cm^{-1} and 1366.11 cm^{-1}, indicating the presence of C-H alkane groups. Furthermore, functional groups identified are at the wavenumbers of 1241.51 cm^{-1} and 1149.90 cm^{-1}, signifying the bending vibration of C=O ether, and at the wavenumber of 998.85 cm^{-1}, indicating the type of C-C vibration. Based on the FTIR spectrum results, it can be concluded that cassava peel starch contains O-H, C-H, C=O, and C-C functional groups.

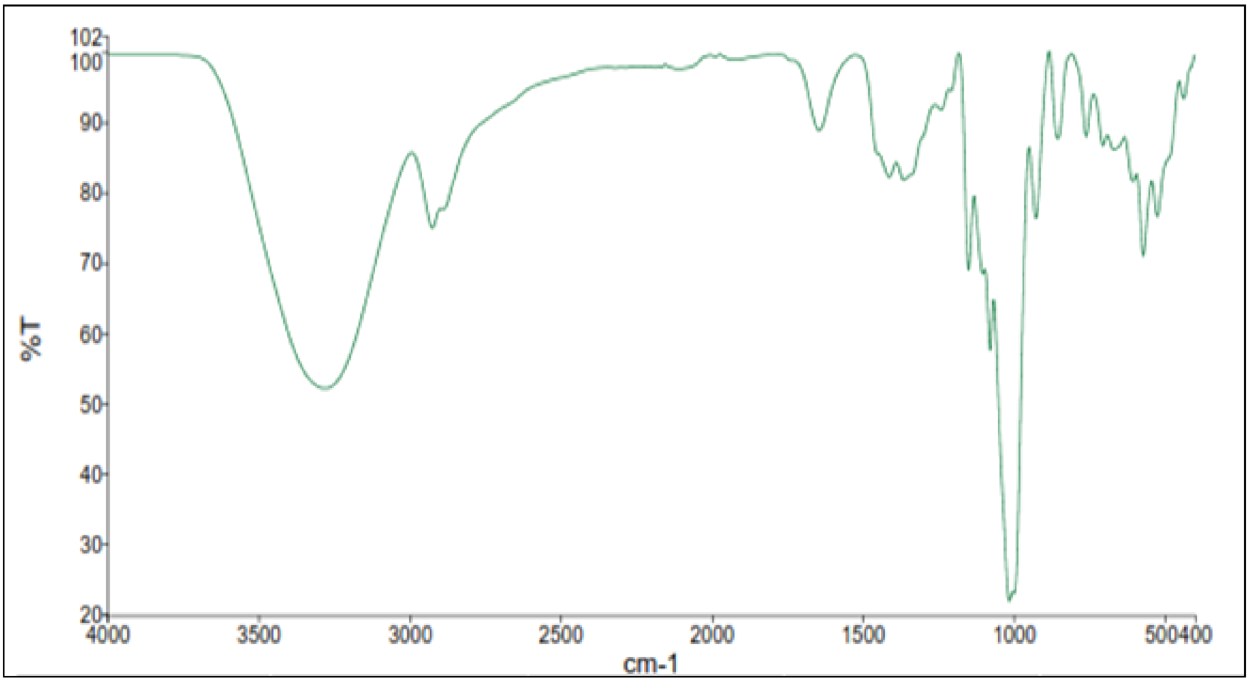


Fig 5 FTIR Spectrum of Cassava Peel Starch Biofilm with the Addition of Carrageenan

According to Figure 5, the results of functional group identification using FTIR display absorption patterns at various wavenumbers. The functional groups identified in the cassava peel starch biofilm with the addition of carrageenan are the O-H alcohol group at a wavenumber of 3290.04 cm^{-1}, the long-chain hydrocarbon C-H alkane group at a wavenumber of 2928.02 cm^{-1}, further confirmed by the identification of bending vibrations at wavenumbers of 1413.32 cm^{-1} and 1365.15 cm^{-1}, indicating the presence of C-H alkane groups. The addition of carrageenan is characterized by the presence of ester sulfate functional groups at a wavenumber of 1241.51 cm^{-1}, as well as glycosidic bonds at wavelengths of 1078.46 $cm^{-1}$and 1014.99 cm^{-1} [25].

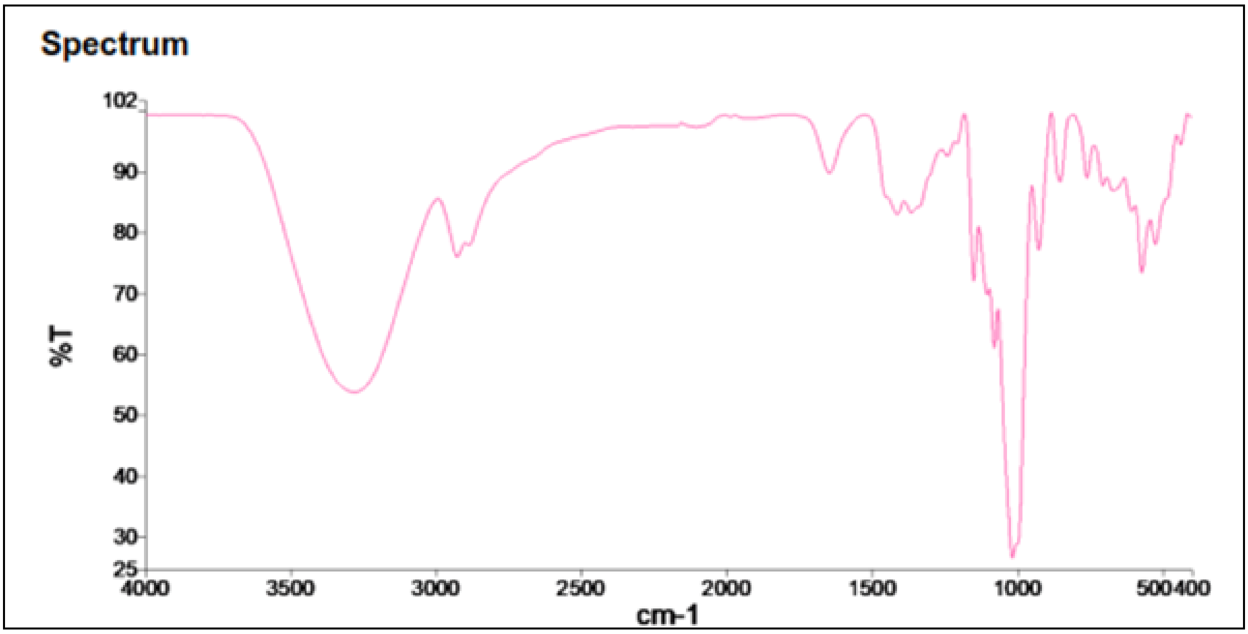
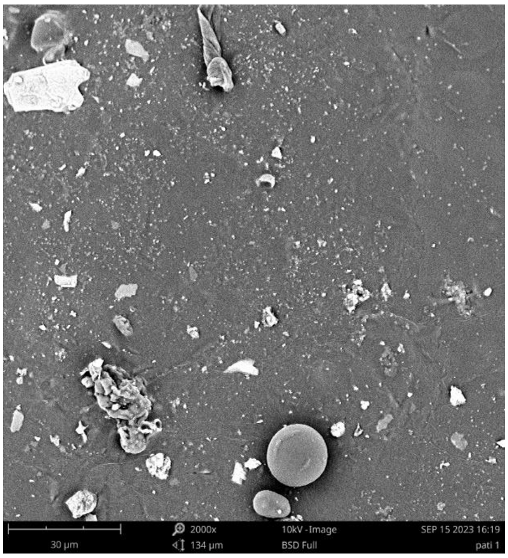


Fig 6 FTIR Spectrum of Cassava Peel Starch Biofilm with the Addition of STPP

Based on Figure 6, the results of functional group identification using FTIR reveal absorption patterns at various wavenumbers. The functional groups identified in the cassava peel starch biofilm with the addition of STPP are the O-H alcohol group at a wavenumber of 3275.50 cm^{-1} and the long-chain hydrocarbon C-H alkane group at a wavenumber of 2929.18 cm^{-1}. Furthermore, functional groups identified include vibrations at wavenumbers of 2105.01 cm^{-1}, signifying C≡C vibration, 1647.34 cm^{-1}, indicating C=C vibration, 1150.01 cm^{-1}, 1104.12 cm^{-1}, and 1078.89 cm^{-1}, representing C-O vibrations, and 1016.52 cm^{-1}, indicating P-O-C vibration. This wavenumber originates from the cross-linking agent added, which is STPP, replacing O-H groups in the starch molecules. This indicates the addition of new functional groups and the occurrence of a crosslink reaction between cassava peel starch and STPP.

D. SEM Analysis

The SEM analysis test is employed to determine the morphological characteristics of pure starch biofilm, starch with the addition of carrageenan, and starch with the addition of STPP. The morphological results of the biofilm can be observed in the figure.



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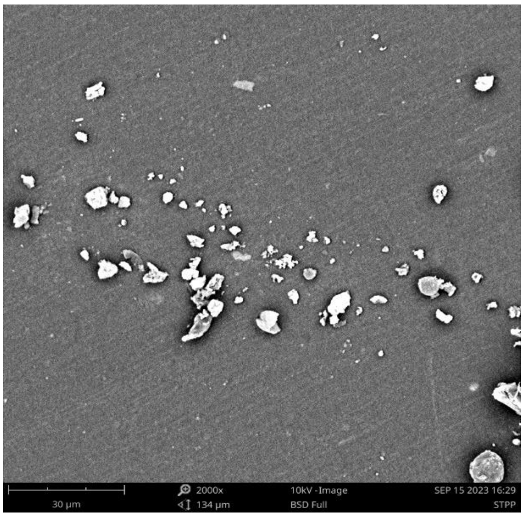

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Fig 7. Morphology of Cassava Peel Starch Biofilm (a) with Addition of Carrageenan (b) and STPP (c)

From Figure 7, it can be observed that the morphological structure of cassava peel starch appears as polygonal granules. According to the study by Koo et al. [26], polygonal starch granules have distinct edges, while cross-linked starch shows a slightly rough surface with dark areas. The dark areas indicate some fragmentation and the formation of grooves within the starch granules. The morphology of cassava peel starch biofilm with the addition of STPP, as shown in image c, exhibits a somewhat rough texture, which aligns with the findings of Singh and Nath [27], who stated that modified starch granules lose their smoothness and structural integrity. This suggests that the rough and compact surface is due to the influence of crosslinking using STPP.

The SEM results in Figure 7 reveal a highly rough surface and even cracking. The absence of glycerol can affect these cracks, as glycerol, being a plasticizer, binds components and enhances elasticity. Cracking can also increase water absorption [28]. SEM analysis shows that the texture of cassava peel starch biofilm with the addition of carrageenan in image b and with the addition of STPP in image c has fewer discontiguous zones compared to pure starch. According to Herliany et al. [29], a high number of discontiguous zones indicates that the biofilm is less compact and dense, resulting in lower tensile strength. The modification of the biofilm with the addition of carrageenan and STPP has proven to enhance the internal structure, as evidenced by the reduced discontiguous zones, making the structure more compact and dense.

IV. CONCLUSIONS

Cassava peel starch has the potential to be developed as a raw material for producing biodegradable films due to its abundant availability, cost-effectiveness, and easy accessibility. The addition of carrageenan and STPP at the appropriate concentrations can improve the physical characteristics, such as tensile strength and elongation, of biodegradable films made from starch. The concentration of starch and carrageenan shows a positive correlation with the tensile strength of the biofilm but a negative correlation with

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its elongation. The concentration of STPP affects an increase in tensile strength at concentrations of 0.5% and 1%, while a decrease occurs at a concentration of 1.5%. The elongation values tend to remain stable with the addition of STPP. This is further supported by FTIR and SEM tests, which reveal the formation of new functional groups after the addition of carrageenan and STPP, indicating the bonding between starch and carrageenan as well as STPP. In SEM testing, the morphology of biofilms with the addition of carrageenan and STPP displays a denser structure compared to pure cassava peel starch biofilm. The tensile strength and elongation values in this study meet the Japan Industrial Standard (1975) requirements, with a minimum tensile strength of 0.39 MPa and an elongation value exceeding 50%. The highest tensile strength is achieved at a starch concentration of 15 grams and a carrageenan concentration of 1.5%, which is 3.17 MPa, while the highest elongation value is obtained at a starch concentration of 5 grams and an STPP concentration of 1.5%, which is 67.4%.

Based on the conducted research, the findings can serve as a basis for optimizing the formulation of cassava peel starch edible films with the addition of carrageenan and STPP.

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