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THE EFFECT OF GLYCEROL AND SAGO STARCH ADDITION ON THE CHARACTERISTICS OF BIOPLASTICS BASED ON ORANGE PEEL PECTIN

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SCIENTIFIC PAPER

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Article Highlights

- The high pectin content in orange peel boasts 42.5% pectin, suitable for bioplastics synthesis
- Examining orange peel pectin with glycerol and sago starch for improved bioplastics
- Tensile strength (4.22 MPa) and elongation (24%) in pectin:starch (75:25%) and glycerol (40%)

Abstract

Sustainable food packaging materials have significant interest in addressing environmental issues by making renewable substitutes such as bioplastics based on pectin. Orange peel has a relatively high pectin content of 42.5%, which can be synthesized into bioplastics. Pectin-based bioplastics tend to have limitations in terms of mechanical and physical strength due to the hygroscopic nature of pectin. This study aims to determine the effect of adding glycerol and sago starch on the characteristics of orange peel pectin-based bioplastics. The casting method was used in the bioplastic formulation using the variation of the pectin:starch ratio (75:25, 65:35, 55:45; and 50:50%) and glycerol composition (10, 20, 30, and 40%). Based on the results, the pectin obtained was categorized as low methoxyl pectin and ester pectin. The characterizations of bioplastics achieved the maximum value of the tensile strength of bioplastics was 4.22 MPa, obtained by adding pectin:starch (50:50%) and 10% glycerol. The maximum value of the elongation properties at the break of bioplastics is 24%, obtained by adding the composition of pectin:starch (75:25%) and 40% glycerol. The more additions of the pectin:starch and glycerol composition result in a higher water vapor evaporation rate. Hence, utilizing orange peel pectin-based bioplastics offers a sustainable solution by exploring repurposing waste to create bioplastics for food packaging, thereby contributing to environmental preservation.

Keywords: bioplastics, pectin, starch, orange peel.

Plastics are widely used in the packaging industry, especially in food packaging, so it must come from environmentally friendly biodegradable materials.

Bioplastics are a renewable source of great interest to researchers because provide a solution to the limitations of petroleum-based polymer resources and can reduce environmental pollution [1,2]. In 2021, the global production capacity of bioplastics reached approximately 2.42 million tons, with nearly 48 percent (1.15 million tons) of the volume destined for the packaging market - the largest market segment within the bioplastics industry [3]. Bioplastics can be defined as plastics that are bio-based and biodegradable so that microorganisms can naturally decompose them [4]. Bioplastics are usually derived from natural materials

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such as polysaccharides (carbohydrates), polypeptides, and lipids [5].

Pectin is a natural biopolymer compound with a high molecular weight (ranging from 50 to 150 thousand), primarily found in plants. Galacturonic acid molecules linked with α-(1-4)-glycoside bonds to form polygalacturonic acid are the constituent components of pectin [6,7]. One source of pectin can be reused as raw material for producing edible films that can be used as food packaging with biodegradable and environmentally friendly capabilities, namely waste derived from fruit peels. Pectin is one of the carbohydrates that can be isolated from orange peel, considering that the productivity of orange plants, especially in Sumatera Utara, Indonesia, is relatively high, around 448,476 tons in 2022 [8]. The pectin content in orange peels is also quite large at around 42.5%, so orange peels have the potential to be reused as a source of pectin in the manufacture of bioplastics [9,10]. Pectin and essential oil are the main contents of citrus peel, especially the albedo part. Pectin in orange peel can be isolated using a solid-liquid extraction process. Pectin can make bioplastics more robust, better, and stable because it has a high gel-forming ability. However, too many hydrocolloids and pectin have hydrophilic properties, so the resulting bioplastics will tear easily [11].

Pectin-based bioplastics with starch can improve pectin-based bioplastics' physical and mechanical characteristics. Starch is a glucose polymer consisting of amylopectin and amylose. Starch can be produced by the extraction process of materials containing high carbohydrates, such as corn, sweet potato, cassava, sago, and taro [12]. Starch is a carbohydrate composed of two components, amylose and amylopectin. Linear polymers with α-(1→4) glucose units in starch are called amylose. Plants with a reasonably high starch content, namely sago (*Metroxylon* sp), are 82.94%. Based on information from the Indonesian Ministry of Agriculture in 2021, sago production is estimated to reach 381,065 tons [13]. Using sago starch as a raw material for bioplastics has several advantages, such as an abundant supply of raw materials that can be biodegradable. Bioplastics made from starch also still have some disadvantages, such as being able to absorb water excessively and being rigid [14].

Bioplastics from natural polymers like pectin and starch are generally brittle and rigid. Glycerol is one example of a plasticizer that can be added to produce flexible and mechanical solid properties [15]. Glycerol as a plasticizer is often chosen because it has a high boiling point of 290 °C. This results in no glycerol evaporating during processing, which is more beneficial. In addition, glycerol can reduce intermolecular and intramolecular bonds found in starch and pectin. Adding glycerol concentration will increase the flexibility of starch, pectin, and gelatin-based biocomposites [16]. Glycerol is widely used in the manufacture of bioplastics, as done by Yuniarti *et al.* [17] researched the manufacture and characterization of bioplastics made from sago starch (*Metroxylon* sp) with acetic acid and glycerol, which concluded that the physical and mechanical properties of bioplastics can increase with the addition of glycerol and acetic acid. Darni *et al.* [18] also researched the effect of plasticizers on the properties of pectin-based edible films, showing better mechanical properties and water vapor permeability in glycerol compared to sorbitol.

Based on this explanation, this study aims to make bioplastics on the effect of adding glycerol and sago starch on the characteristics of orange peel pectin-based bioplastics produced. In the growing field of chemical engineering, exploring sustainable alternatives to plastics is a must. This research also contributes by utilizing growing eco-friendly materials, addressing the issue of environmental problems, and encouraging a more sustainable future.

MATERIALS AND METODS

The materials used such as orange peel for pectin and sago starch, were obtained from supermarkets around Medan, Sumatera Utara, Indonesia. Food-grade glycerol was used. Hydrochloric acid (HCl), ethanol (C_2H_5OH), and aquadest were used as pectin isolation materials, as sodium chloride (NaCl), sodium hydroxide (NaOH), and phenolphthalein indicators. The equipment used in this study were analytical balance, three-neck flask, beaker glass, filter paper, measuring cup, bioplastics mold, hot plate, magnetic stirrer, and 100 mesh sieve.

Procedure for pectin isolation from orange peel

Weighed 6 g of orange peel powder and poured it into a three-neck flask, then added 200 ml of 0.02 N HCl solution at 70 °C for 60 minutes. Filtered and added 96% ethanol to the filtrate with a volume ratio of 1:1 while stirring to form a precipitate.Separated the precipitate using filter paper. Filter the precipitate and wash it using ethanol continuously. The precipitate was dried to a constant weight and sieved using a 100-mesh sieve.

Bioplastics preparation procedure

Weighed 3 g of pectin/starch in the ratio of 75:25; 65:35; 55:45; 50:50%, poured into a beaker glass, and added 75 ml of distilled water. Then, heat the solution to 70 °C using a hot plate. Glycerol was added with 10, 20, 30, and 40% variations and then stirred until homogeneous. After mixing evenly, the solution was cooled before molding. The solution was poured into the mold and then dried for 48 hours at room temperature. Then, the bioplastics was removed from the mold.

Pectin Analysis

Equivalent Weight

An amount of 0.5 g of pectin was moistened with 5 ml of ethanol and then dissolved in 100 ml of distilled water supplemented with 1 g of NaCl. Titrated slowly using 0.1 N NaOH with phenolphthalein indicator until it turns pink color and the volume of NaOH used is noted [19]. The equivalent weight of pectin can be calculated by Eq. (1).

$$Equivalent\ weight\left(mg\right)=\frac{samle\ weight\left(mg\right)}{ml\ NaOH\times N\ NaOH}\times 100\% \tag{1}$$

Methoxyl content

A total of 25 ml of 0.25 N NaOH was added to the neutral solution from the determination of equivalent weight, then stirred and allowed to stand for 30 minutes under closed conditions at room temperature. A total of 25 ml of 0.25 N HCl was added and titrated with 0.1 N NaOH with phenolphthalein indicator until it turned pink color, and the volume of NaOH used was noted [20]. Pectin methoxyl content can be calculated with Eq. (2).

$$Methoxyl\ content\left(\%\right)=\frac{ml\ NaOH\times 31\times N\ NaOH\times 100}{sample\ weight\left(mg\right)} \tag{2}$$

The value 31 is the molecular weight of methoxyl which is CH_3O.

Galacturonate content

The volume of NaOH obtained from the determination of equivalent weight and methoxyl content is used to calculate galacturonic content with the following Eq. (3) [20].

$$Galacturonate\ levels=\frac{176\times 0.1z\times 100}{sample\ weight\left(mg\right)}+\frac{176\times 0.1y\times 100}{sample\ weight\left(mg\right)} \tag{3}$$

where, z-volume of NaOH based on equivalent weight; y-volume of NaOH based on methoxyl content; the value 176 is the molecular weight of galacturonate.

Degree of esterification

The methoxyl content and galacturonic content that have been obtained are used to calculate the degree of esterification of pectin using Eq. (4) as follows [20].

$$Degree\ of\ esterification=\frac{176\times \%methoxyl}{31\times \%galacturonate} \tag{4}$$

The value 31 represents the molecular weight of methoxyl; the value 176 represents the molecular weight of galacturonate.

Characteristics and physical properties of bioplastics

The physical strength of bioplastics can be determined based on the tensile strength and elongation at break values of the resulting starch/pectin-based bioplastics. The ASTM 882 standard with the provisions of the Universal Testing Machine (UTM) model was used to test the tensile strength and elongation at break of pectin-based bioplastics at the Laboratorium Polimer, Department of Chemical Engineering, Universitas Sumatera Utara, Medan, Indonesia. Eqs. (5) and (6) calculate the tensile strength and elongation at break, respectively:

$$\sigma=\frac{F_{\max}}{A_0} \tag{5}$$

where σ is the tensile strength (N/m^2), $F_{\max}$ is the tensile force applied to the cross-section of the specimen (N), and A_0 is the initial cross-sectional area of the specimen before force load (m^2).

$$elongation\left(\%\right)=\frac{\Delta l}{l_0}\times 100\% \tag{6}$$

where Δl is the change in length (cm) and l_0 is the initial length (cm).

Analysis Scanning Electron Microscope (SEM)

The morphological of biocomposite was observed by SEM EVO MA 10 ZEISS at magnification ranging from 1000x to 5000x in Laboratorium Penelitian dan Pengujian Terpadu, Universitas Gadjah Mada, Yogyakarta, Indonesia.

Water vapor transmission rate

The film was placed on the mouth of a porcelain dish with a diameter of 7 cm and a depth of 2 cm filled with 10 g of silica gel. The edges of the film and the porcelain cup were glued with glue. Put the porcelain cup into a container containing 40% NaCl (b/v). Calculated the film changes every 1 hour until reaching the 7th hour by Eq. (7) [21].

$$Water-vapor\ transmission\ rate=\frac{\Delta x}{A} \tag{7}$$

where Δx - linear slope (g/h); A - film area (m^2).

RESULTS AND DISCUSSION

Orange peel pectin analysis

Pectin was characterized from orange peel to see the equivalent weight, methoxyl content, galacturonic content, and degree of esterification produced. These values can be seen in Table 1. The yield value and equivalent weight of pectin from orange peel were 25.38% and 510.52 mg, respectively. These results can be compared with other raw materials such as Dragon Fruit (*Hylocereus polyrhizus*) peel has a pectin content of 12% and yields 11% [22] while Grapefruit (*Citrus Maxima*) peel has a pectin content of 40% and yields 27.3% pectin [23]. Considering the use of this fruit peel material as waste is still small so it needs to be encouraged. The content of unesterified free galacturonic acid in the pectin molecular chain is the equivalent weight. Free galacturonic acid that is free of methyl ester (unesterified) is called pectic acid. The equivalent weight results obtained in this study did not meet the standards of the International Pectin Procedures Association (IPPA) (2003), which ranged from 600–800 mg [24]. This is influenced by pectin content, plant type, and extraction method, which can affect the equivalent weight of pectin [19].

Table 1. Characterization results of orange peel pectin.

Characterization	Values	IPPA Standard
Yield (%)	25.38	-
Equivalent Weight (mg)	510.52	600–800
Methoxyl (%)	5.20	>7.12 (pectin with high methoxyl) <7.12 (pectin with low methoxyl)
Galacturonic acid (%)	64.06	60
Degree of esterification (%)	46.09	>50% (high ester pectin) <50% (low-ester pectin)

Methoxyl content is the number of esterified methyl groups in orange peel pectin that can affect gel formation ability. Pectin with methoxyl content >7% is called high methoxyl pectin, while pectin with methoxyl content <7% is called low methoxyl pectin. In this study, pectin was obtained with a methoxyl percentage of 5.20%, so the pectin produced was categorized as low methoxyl pectin [25].

Galacturonic content is an important parameter affecting the pectin gel's structure. Table 1 shows that the galacturonic content obtained has met the IPPA (2003) standard of 64.06% [24]. Galacturonic levels that have met the standards of IPPA (2003) can be concluded to have broken the chain of pectin components against other components, such as hemicellulose contained in orange peel [19]. The degree of esterification is a parameter that states the percentage of esterified carboxyl groups. Pectin with a degree of esterification > 50% is called high methoxyl pectin, while pectin with a degree of esterification < 50% is called low methoxyl pectin. The study obtained pectin with a degree of esterification of 46.09%, so the pectin obtained in this study is included in the category of low-ester pectin [15,25].

Effect of glycerol and sago starch addition on tensile strength value of orange peel pectin-based bioplastics

Fig. 1 shows the effect of glycerol and sago starch addition on the tensile strength value of orange peel pectin-based bioplastics.

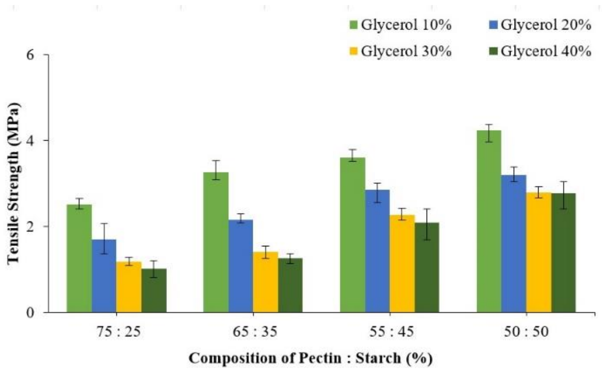
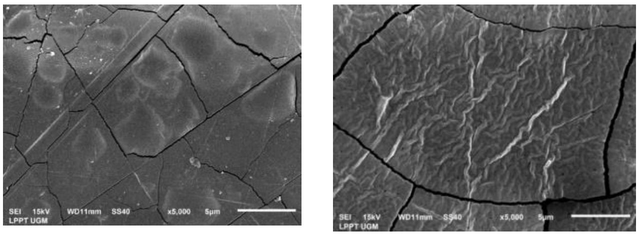


Figure 1. Effect of glycerol and sago starch addition on tensile strength value of orange peel pectin-based bioplastics.

Fig. 1 shows that the maximum tensile strength of bioplastics is 4.22 MPa, which is obtained in addition to pectin:starch composition (50:50%) and 10% glycerol. The minimum value of bioplastics tensile strength is 1.01 MPa in addition to pectin:starch composition (75:25) and 40% glycerol. According to Japanese Industrial Standard 1707:1975, the minimum value for tensile strength for bioplastics is 0.39 MPa [26]. The maximum tensile strength value of bioplastics indicates that the composition of starch has a significant role in the tensile strength value of bioplastics compared to pectin. This is because sago starch is known to have main components in the form of amylose and amylopectin, which will affect the mechanical properties of bioplastics. The more the composition of pectin increases, the tensile strength value of bioplastics will decrease. This is because pectin comprises galacturonic acid molecules that bind to α-(1-4)-glycoside bonds to form polygalacturonic acid. Polygalacturonic acid is an amorphous region with lower stiffness than crystalline regions, such as amylopectin found in starch.

Fig. 1 also shows that the maximum tensile strength value continues to decrease until the addition of 40% glycerol. At the addition of 10% glycerol in the composition of pectin:starch (50:50%), the highest tensile strength value is due to glycerol reducing intermolecular interactions between pectin and starch [27]. This is also supported by the scanning electron microscopy (SEM) analysis results, shown in Fig. 2a, which shows that bioplastics have a more stable surface.



a b

Figure 2. SEM results of pectin:starch (50:50%) composition bioplastics in the addition of (a) 10% glycerol and (b) 40% glycerol.

Fig. 2 shows the scanning electron microscopy (SEM) analysis results of pectin:starch (50:50%) composition bioplastics in the addition of 10% glycerol and 40% glycerol. Bioplastics surfaces must be produced homogeneous and gap-free to maintain the characteristics [28]. However, adding 20%, 30%, and 40% glycerol decreased the tensile strength. This is because the glycerol added is too high, so glycerol disrupts not only intermolecular interactions between pectin and starch molecules but also disrupts intramolecular bonds in starch and pectin, thereby reducing the tensile strength value of the resulting bioplastics as can be seen in Fig. 2b where the resulting surface is less stable and indicated to form a plasticsol film caused by excess glycerol.

Effect of glycerol and sago starch addition on elongation at break value of orange peel pectin-based bioplastics

The effect of glycerol and sago starch addition on the elongation at the break value of orange peel pectin-based bioplastics can be seen in Fig. 3

Fig. 3 shows that the maximum value of elongation properties at the break of bioplastics is 24%, which is obtained by adding pectin:starch composition (75:25%) and 40% glycerol. This is due to the high composition of pectin, which will increase the amorphous area, thereby increasing the bioplastics' flexibility. The Japanese Industrial Standard 1707:1975 for elongation at break value is between 10%–50%[26]. The minimum elongation value at the break of bioplastics is 6.39% in the addition of pectin:starch composition (50:50%) and 10% glycerol. This is due to the difference in polarity between starch, pectin, and glycerol, resulting in a decrease in the elongation at the break value of the bioplastics produced [29]. Hassani *et al.* obtained similar results when observing the decrease in elongation at the break of the nanocomposite bioplastics film of potato starch and gum arabic with the addition of boron oxide nanoparticles and Anise hyssop (*Agastache foeniculum*) essential oil [30].

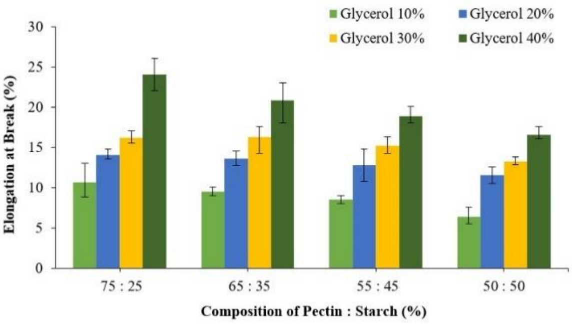


Figure 3. Effect of glycerol and sago starch addition on elongation at break value of orange peel pectin based bioplastics.

Effect of glycerol and sago starch addition on water vapor transmission rate of orange peel pectin-based bioplastics

The effect of the addition of glycerol and sago starch on the water vapor transmission rate of orange peel pectin-based bioplastics is shown in Fig. 4.

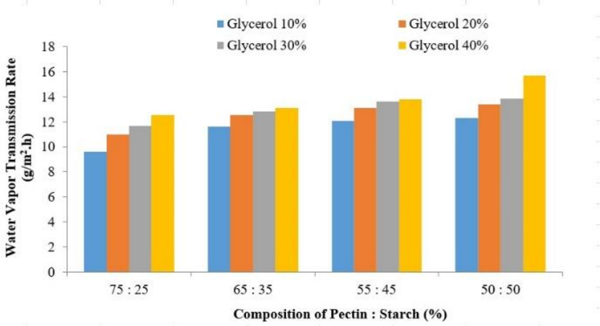


Figure 4. Effect of glycerol and sago starch addition on water vapor transmission rate of orange peel pectin-based bioplastics.

The maximum water vapor evaporation rate is 15.67 g/m^2.h when adding pectin:starch (50:50%) and 40% glycerol. The use of pectin contributes to forming a linked structure in bioplastics that can limit air exchange [31]. Fig. 4 also shows that the more the pectin: starch and glycerol composition is added, the higher the water vapor evaporation rate. This is due to glycerol, which acts as a plasticizer with water-absorbing properties, so the transfer of water vapor from the environment to the surface of bioplastics becomes faster. In addition, glycerol has a small molecule, so it will more easily enter the amorphous network in pectin and starch so that there are more opportunities for water to be absorbed and water transfer in the film [27,32]. Bioplastics protects the product against ambiance risk variations according to the packaging settings [33].

CONCLUSION

Bioplastics based on pectin from orange peel formulated with sago starch and glycerol have been created in this study. Based on the results, the pectin obtained from orange peel were yielding of 25.38% or equivalent weight of 510.52 mg, categorized as low methoxyl pectin (5.20%) and low ester pectin (46.09%). The addition of glycerol and sago starch in formulating orange peel pectin-based bioplastics showed significant changes in the mechanical and physical properties of the bioplastics produced. The maximum tensile strength of bioplastics is 4.22 MPa, obtained by adding pectin:starch composition (50:50%) and 10% glycerol. The maximum value of elongation at the break of bioplastics is 24%, obtained by adding pectin:starch composition (75:25%) and 40% glycerol. The formulation given follows the Japanese Industrial Standard 1707:1975. Scanning electron microscopy confirms the supported analysis of the surface of the bioplastics to the interaction between the variation formulated. The more the addition of pectin:starch and glycerol composition, the higher the water vapor evaporation rate. The utilization of orange peel as pectin also provides an excellent alternative to orange peel waste solutions. The future research direction is to apply food packaging to prevent oxidation by observing changes in food color and texture.

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NAUČNI RAD

UTICAJ DODATKA GLICEROLA I SAGO SKROBA NA KARAKTERISTIKE BIOPLASTIKE NA BAZI PEKTINA OD KORE POMORANDŽE

Održivi materijali za pakovanje hrane imaju značajan interes u rešavanju ekoloških problema stvaranjem obnovljivih zamena, kao što je bioplastika na bazi pektina. Kora pomorandže ima relativno visok sadržaj pektina od 42,5%, koji se može sintetizovati u bioplastiku. Bioplastika na bazi pektina ima ograničenja u pogledu mehaničke i fizičke čvrstoće zbog higroskopne prirode pektina. Ova studija ima za cilj da utvrdi efekat dodavanja glicerola i skroba sagoa na karakteristike bioplastike na bazi pektina od kore pomorandže. Metoda livenja je korišćena u formulaciji bioplastike korišćenjem varijacije odnosa pektin:skrob (75:25, 65:35, 55:45 i 50:50%) i sastava glicerola (10, 20, 30 i 40%). Na osnovu rezultata, dobijeni pektin je kategorisan kao pektin sa niskim sadržajem metoksila i estara pektina. Karakterizacijama bioplastike postignuta je maksimalna vrednost zatezne čvrstoće od 4,22 MPa za bioplastiku dobijenu korišćenjem odnosa pektin:skrob od 50:50% i 10% glicerola. Maksimalna vrednost svojstava elongacije pri lomljenju bioplastike od 24% dobijena je korišćenjem odnosa pektin:skrob 75:25% i 40% glicerola. Više dodataka sastava pektin: skrob i glicerol dovodi do veće brzine isparavanja vodene pare. Stoga, korišćenje bioplastike na bazi pektina od kore pomorandže nudi održivo rešenje istraživanjem otpada za dobijanje bioplastike za pakovanje hrane, čime se doprinosi očuvanju životne sredine.

Ključne reči: bioplastika, pektin, skrob, kora pomorandže.