Tensile Strength and Elongation Testing for Starch-Based Bioplastics using Melt Intercalation Method: A Review

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Abstract. Plastics were commonly used as packaging materials for primary, secondary, and tertiary needs. However, the continuous use of plastic was inadequate for the environment. The research that was developing to address the use of conventional plastics is bioplastics. Bioplastics undergo faster degradation but had low mechanical strength and were hydrophilic. One of the main ingredients of bioplastics was starch. This study aimed to examine the effect of using starch-based materials on the quality parameters of bioplastic tensile strength and elongation quality. The tensile strength and elongation values of bioplastic from various treatments showed a relatively large range of results. Glycerol was the most widely used plasticizer because Glycerol has the best interaction ability compared to other plasticizers when combined with starches with different characters, either by adding various types of fillers or without adding fillers. The types of fillers that were commonly used are chitosan, clay, and ZnO. The use of plasticizers and fillers gives an opposite contribution to the bioplastic quality of tensile strength and Elongation.

1. Introduction

The demand and use of plastics continue to increase now. Plastics were commonly used as packaging materials for primary, secondary, and tertiary needs. The plastic characteristics, which were light in weight, as a packaging that is easy to use, are the advantages of plastic compared to other materials. However, plastic’s continuous use is terrible for the environment because it was difficult to decompose, increasing environmental damage such as soil pollution.

The research that was developing to address the use of conventional plastics was bioplastics. Bioplastics undergo faster degradation but had low mechanical strength and were hydrophilic. It was mixing starch with chitosan, cellulose, gelatin, and other biopolymers, which can correct starch-based plastics’ shortcomings. There are other advantages of using bioplastics, namely bioplastics made from natural polymer materials such as Starch, cellulose, and Poly Lactic Acid (PLA). Bioplastics can accelerate soil fertility when disposed of into nature [1, 2].

One of the main ingredients of bioplastics was starch. Indonesia has a wealth of natural resources that can be a starch source, including corn, sago, cassava, sweet potatoes, sorghum [2, 6, 18]. The unique functional characteristics allow the starch to be used for various purposes, both as food and non-food ingredients. The most significant demand for bioplastics was starch-based. Starch was more economical and competitive than petroleum because it comes from...
renewable ingredients. The process of making bioplastics from starch was more straightforward than other raw materials. Starch can be processed using several methods into bioplastics. The types of starch that are widely used are corn starch and cassava starch [3].

The abundant availability of Starch in Indonesia makes starch the raw material of choice for making bioplastics. Types of Starch used as the primary material for making bioplastics include cassava, potato, and corn starch [1]. Starches from other carbohydrate sources and agricultural waste that could be used as raw material for bioplastics include konjac tuber starch, durian seed starch, and starch from cassava peels [14, 25]. Meanwhile, starch was abundant in nature but was slightly used as the primary material for making bioplastics, including sago starch, sorghum starch, and canna starch [2]. By knowing the characteristics of various Starch types that exist, the bioplastic produced quality characteristics’ potential and impact can be described. In this case, the tensile strength of the bioplastic is the main parameter for plastic products.

2. Research Methods
The method used in this study was a literature review, by collecting various published libraries in 2000-2019 on the scope of starch-based bioplastic production. Data and information in this study were obtained through secondary data in journals, theses, and literature from the internet, whose information sources can be justified. References were obtained from several sources, including Google Scholar, SCOPUS, Science Direct, Springer Link, doaj.org, university repository, etc. Additional more detailed information was sought by referring to certain websites whose information sources can be accounted for, such as data on the astm.org and iso.org websites.

References sources that have been obtained were then selected. If there were references that were not related to bioplastic-based bioplastics, they could not be used. The initial connections sources obtained were then carried out a selection process of at least 30 articles. The next step that was carried out after the selection process of reference(s) sources was to review the literature by analyzing each reference(s) source, criticizing the presentation given, and comparing one literature with another.

3. Results and Discussion
3.1. Characteristics of Starch-Based on Source
Starch is a water-insoluble complex carbohydrate, white powder, tasteless, and odorless, resulting from plant extraction. Starch significantly influences plants as a food reserve, generally found in the tubers, rhizome, fruit, seeds, and grains.

Starch consists of two types of carbohydrates, namely amylose and amylopectin. Starch for every kind of plant has a different composition from one another. Amylose was a straight-chain polysaccharide consisting of \(?-1,4\) glycoside glucose molecules. Amylose was part of starch soluble in water, plastic and a molecular weight between 50,000 and 200,000. Amylopectin was a branched polysaccharide consisting of glucose molecules bound to each other via \(?-1,4\) glycoside bonds and \(?-1,6\) glycosidic. Amylopectin is part of starch insoluble in water, has a molecular weight of between 70,000 - 1,000,000, and gives it a sticky nature [4].

The content of amylose and amylopectin affects starch’s physical and chemical properties, which include the influence of water absorption, solubility, degree of gelatinization, and swelling. Gelatinization was the process of swelling of the starch granules due to heat and water, which results in the starch granules unable to return to their original shape. The constituent compositions of starch generally have amylopectin about 70-85% and amylose of 15-30% [19].

The size of the starch granules was related to the gelatinization temperature. The size of the starch granules ranges from 1 to 150 microns. Starches with large granule sizes tend to have a low gelatinization temperature because their molecular bonds are weaker so that the energy required for the process was lower. On the other hand, starches with small granule sizes tend
to have high gelatinization temperatures because their molecular bonds are more robust. The energy required for the process is also higher [20].

### Table 1. The yield of Starch, Amylose, and Amylopectin Content Based on Starch Source Classification

| Classification     | Type of Starch | Amylose (%) | Amylopectin (%) | Total starch (%) | Ref. |
|--------------------|----------------|-------------|------------------|------------------|------|
| Agricultural waste | Mango seeds    | 14.82       | 44               | 44               | [13] |
|                    | Jackfruit seeds| 16.39       | 58.83            | 74.22            | [9]  |
|                    | Durian seeds   | 22.33       | 54.31            | 76.65            | [10] |
|                    | Banana peels   | 25.77       | 74.35            | 22.6             | [11] |
|                    | Cassava peels  | 27.83       | 72.61            | 60.68            | [14] |
|                    | Potato peels   | 25          |                  |                  | [12] |
| Tubers             | Yam            | 23.7        | 65.26            | 82.15            | [5]  |
|                    | Cassava        | 14.6        | 70.19            | 85.53            | [6]  |
|                    | Taro           | 5.55        | 74.45            | 80.4             | [5]  |
|                    | Potato         | 15.9        | 71.2             | 87.19            | [5]  |
| Grains             | Corn           | 28.5        | 71.5             | 72.81            | [8]  |
|                    | Sorghum        | 25.86       | 74.14            | 69.51            | [8]  |
| Fruits             | Breadfruit     | 26.76       | 73.24            | 76.39            | [7]  |

Table 1 shows that the starch sources can be classified into tubers, starch sources, seeds, fruits, and starch sources from agricultural waste. Sources of Starch from tubers such as potatoes, cassava, sweet potatoes, taro have a starch composition of about 87.19%; 85.53%; 82.15%; and 80.40%, respectively [5, 6]. The source of fruit-based starch was breadfruit with a starch content of 69.00% [7]. Corn and sorghum were included as starch in grains with starch content of 72.81% and 69.51%, respectively [8]. Other materials that can be used as a source of starch come from agricultural waste, such as mango seeds, jackfruit seeds, durian seeds, potato peels, banana peels, and cassava peels with starch content of 70.22% each; 76.65%; 38.79%; 22.60%; and 60.68% respectively [9, 10, 11, 12, 13, 14]. This shows that the starch source material which contains high amounts of starch comes from tubers. This is because the tubers have a low moisture content compared to other starch sources. The lower the water content in the material, the greater the starch content [15, 6].

Potato peels starch has a small starch content of 25.00%. This was influenced by the proportion of potato skins, which is only 11.2% of potato tubers [16]. The lowest starch content was found in banana peels at 22.60% [11]. The research of [11] stated that the level of maturity of banana skin color affects the starch and sugar composition. As indicated by the change in skin color on the banana, the ripe banana fruit lowered the starch content and sugar content contained therein. It shows that low banana starch content is influenced by the level of maturity of the banana fruit.

Starch made from grain has a high amylose content. As a result, separating the elements causes gelatinization to have a higher temperature, around 78 - 86°C. The gelatinization temperature in cereal starch is higher than that of tuber starch sources 69 - 75°C. The difference in gelatinization temperature of the starch source from tubers and Grains was caused by the lower strength of the intermolecular bonds in the starch of tubers than Grains’ starch source. This resulted in a lower gelatinization temperature, more significant development, and Starch dissolution rate [10]. Meanwhile, for fruit peel waste materials such as banana peels, the maturity level will affect starch’s gelatinization temperature. The riper banana peels used, the higher the gelatinization temperature needed [11].
Agricultural waste in the form of jackfruit seeds, durian seeds, mango seeds, and sweet potatoes was a starch source with amylopectin less than 70% (Table 1). This was because the waste fruit seeds contain amylose and amylopectin in proportions that tend not to be different. This condition is related to the function of fruit seed waste, which acts as a regeneration agent in plants to require a certain proportion of compounds for the growth process [20]. The difference in the proportion of amylose present in sweet potato starch to starch’s balance in tubers was very dependent on the different varieties and cultivars in each plant. Planting conditions and treatments also influence the varied proportions of various types of tubers [21].

The starch content in cereal-based starch sources such as corn and sorghum starch had a relatively high amylose content, respectively 28.50% and 25.86% [8]. This material was included in the high amylose starch group (>25%). High amylose levels were also found in breadfruit by 26.76% [7]. The harvest’s age influenced amylose levels in grains and fruits. The longer the harvesting age for grain crops, the higher the starch and amylose content [22].

According to [3], amylose and amylopectin produce bioplastics with different characteristics. High amylose tends to form crystals that have more substantial mechanical properties than amylopectin, which is amorphous. The crystalline nature of amylose causes starch molecules to become brittle when used as raw material for making bioplastics. Thus it is necessary to separate amylose and amylopectin to obtain bioplastics with better results. The separated amylose can be used as a raw material for making films. Films made from pure amylose have better mechanical properties than films using pure amylopectin as raw material. The stability of bioplastics was influenced by amylopectin, while amylose affects the compactness of the material. However, the addition of plasticizers and processing conditions at high humidity increases the crystallinity of bioplastics with high amylopectin starch raw materials and improve their mechanical properties. The addition of plasticizers did not affect the crystallinity of high amylose starch [3].

### 3.2. Effect of Starch, Plasticizers, and Filler Types on the Tensile Strength of Bioplastics

Starch’s use in the manufacture of biodegradable plastics (bioplastics) has a significant effect on physical quality in the form of tensile strength. Sources of Starch used in bioplastics manufacture can be classified into Starch sources from grains, fruit, agricultural waste, and tubers. Table 2 shows the tensile strength of bioplastics based on starch sources classification that tuber-based bioplastics offer the best tensile strength interaction. This can be seen in potatoes, taro, and cassava, which have tensile strength values that meet the standard of bioplastic tensile strength of 10 - 100 MPa [1]. The tensile strength of taro starch has the most extensive range compared to other starch sources, amount 18.49 to 89.32 MPa. This was influenced by the taro root starch’s characteristics, which has the highest amylopectin content than the amylopectin content in other tubers. According to [37] bioplastic stability was influenced by amylopectin, while amylose affects the compactness of the material. The tensile strength quality of starch-based bioplastics from other tubers shows a varied value.

Sweet potato starch was a bioplastic base material that has the lowest tensile strength value of 3.29 MPa. The low tensile strength value in sweet potatoes was due to the high levels of amylose in the ingredients of 23.7%. The sweet potato starch contained the lowest amylopectin compared to other tuber starch content 65.6%. This condition makes the gelatinization process imperfect in starch. High levels of amylose reduce the ability of starch to undergo gelatinization. Another factor that causes the tensile strength value of sweet potato to be lower than other tubers is [34], which states that high amylose tends to form crystals that produce more robust mechanical properties than amylopectin, which is amorphous. The crystalline nature of amylose causes starch molecules to become brittle when used as raw material for making bioplastics, so it was necessary to separate amylose and amylopectin to obtain bioplastics better results.

Bioplastics derived from grain starch consisting of corn starch and sorghum have a tensile strength value of 1.42 to 3.92% and 8.75%, respectively [23,1]. This shows that the grain source
bioplastic’s tensile strength was still within the standard of moderate bioplastic properties, namely 1 - 10 MPa. However, it did not meet the standard of bioplastics (10 - 100 MPa) and conventional plastic Indonesia National Standard (SNI) (24 - 302 MPa). The tensile strength value was influenced by the high amylose content, which exceeds 25% of the starch content with an amylose content of more than 25%, classified as starch with high amylose content.

The value of bioplastic tensile strength from agricultural waste starch sources classification has the lowest average compared to other starch sources. This is because the starch content in the starch source of agricultural waste is less than 75%, which means that it does not meet Indonesian Industrial Standards. However, the types of potato skin starch and cassava peel starch have a tensile strength of 23.39% and 43.22%, which have met the conventional SNI plastic standard of 24-302 MPa. This value is obtained from the contribution of the types of fillers and plasticizers used in making bioplastics [23, 25]. Starch’s choice as a bioplastic material needs to pay attention to the final characteristics expected from the bioplastic produced.

Table 2. The Effect of Starch Source Material, Plasticizer and Filler types on Tensile Strength

| Starch Source                                  | Plasticizers | Type of Filler | Result (MPa)       | Ref.(s) |
|------------------------------------------------|--------------|----------------|--------------------|---------|
| Jackfruit seeds, corn, potato, banana peels, taro, cassava peels | Glycerol     | Non-Filler     | 0.22 – 18.49       | [25, 26, 31, 32, 33, 34] |
| Mango seeds, taro                              | Clay (3 – 4%) |                | 5.65 – 76.57       | [13, 35] |
| Corn, potato peels, banana peels, cassava peels, yam, Jackfruit seeds, cassava | Chitosan (2 – 41.7%), MCC (20%) | | 3.29 – 43.22, 0.63 – 16.70 | [1, 36, 37, 38] |
| Cassava peels                                  | Palmyra fibbers (10%) | | 2.09 | [24] |
| Yam                                            | ZnO (9%)     |                | 6.29               | [38] |
| Yam                                            | Chitosan (9%) + ZnO (1%) | | 3.92 | [38] |
| Taro                                           | Sorbitol    | Clay (4%)      | 89.32              | [35] |
| Sorghum, breadfruit, jackfruit seeds           | Chitosan (2 – 40%) | | 8.75 – 19.36 | [7, 23, 28] |
| Biodegradable plastic standard by SNI 7818:2014 |              |                | Minimum 1.343      | [40] |
| Conventional plastic standard by SNI ISO 17557:2011 |              |                | 24.7 – 3022        | [44] |

Assessment of the quality of the tensile strength of bioplastics was not only influenced by the type of starch source used. Other additives in plasticizers and fillers affect the bioplastic’s final quality on the tensile strength parameter. Table 2 shows that Glycerol was the most widely used plasticizer because Glycerol has the best interaction ability compared to other plasticizers when combined with starches with different characters, either by adding various types of fillers or without adding fillers. Meanwhile, sorbitol in bioplastic production is highly dependent on the characteristics of the starch used. In this case, sorbitol needs to be supported with certain fillers that can increase the tensile strength of the bioplastic produced according to the characteristics of the starch used. Therefore, the interaction of starch molecules with Glycerol is better than the interaction between starch and sorbitol.

Sorbitol plasticizers and fillers as supporting materials for the manufacture of starch-based bioplastics had an essential role in determining the tensile strength value. Based on Table 2 shows that the bioplastic made from taro starch with the addition of sorbitol in the concentration range of 20-30% and the type of Clay filler with a concentration of 4% produces the highest tensile strength compared to the use of sorbitol with chitosan and the use of Glycerol with any filler. This is following [39] stated that fillers’ addition could increase the value of the tensile strength response because of hydrogen bonds formed in the bioplastic so that the chemical bonds will be
Bioplastic made from various types of starch with glycerol plasticizer without filler has a tensile strength range of 0.22 - 18.49 MPa. The tensile strength value was the lowest compared to bioplastics using Glycerol and filler. The low value of bioplastic tensile strength is following [23] states that plasticizers are suitable enough to reduce internal hydrogen bonding to increase the material’s intermolecular distance. Still, plasticizers’ use is not enough, so that a filler is needed as a filler that can increase the stiffness of the bioplastic too much bending, increasing tensile strength.

The types of fillers that were commonly used are chitosan, clay, and ZnO. Meanwhile, Palmyra fibers and microcrystalline cellulose were filler materials that were less commonly used for the manufacture of bioplastics. However, they had a high contribution to increasing tensile strength in bioplastics. Bioplastics with Microcrystalline Cellulose filler with the same concentration, different starches, and different glycerol concentrations also showed significant tensile strength values. The resulting tensile strength value in bioplastics made from jackfruit seed starch with a glycerol concentration of 20% was 0.63 MPa. In comparison, bioplastics made from cassava starch with a glycerol concentration of 25% resulted in a tensile strength value of 16.37 MPa. This difference in value occurs because several materials are not entirely mixed [30].

In the manufacture of bioplastics, using Glycerol as plasticizers and palmyra fiber as a filler produces a tensile strength of 2.09 MPa. This value is more significant than non-filler bioplastics and lower than bioplastics using Microcrystalline cellulose (MCC) filler. The low tensile strength value of bioplastics using palmyra filler is due to the added coir fiber having a different size. The fiber size was uniformed by sieving with a 100mesh sieve, but due to the palmyra fiber’s physical shape, which is hard, stiff, and long, it allows the elongated fiber to escape sieved [24].

The use of chitosan in making bioplastics has concentrations ranging from 2 - 41.7%. This shows that chitosan could fill bioplastics with various types of plasticizers, starch, and different concentration variations. Bioplastics of multiple kinds of starch and plasticizers using chitosan produce tensile strength values that meet bioplastic standards, Indonesia National Standard for conventional plastics, and moderate properties of bioplastic standards. However, incorporating chitosan and ZnO fillers in making bioplastics did not produce optimal tensile strength values. The chitosan added by ZnO had a tensile strength value of 3.29 MPa. This value was not more significant than the chitosan filler bioplastic without ZnO or vice versa. This can happen because each filler could not fill the layers with one another. The result was that the increase in ZnO and clay concentrations causes the stiffness of the plastic produced.

Based on the tensile strength test results on bioplastic from various Starch types, most bioplastic meets the minimum standard of the biodegradable plastic tensile strength of 1.343 MPa (SNI 7818: 2014). However, the bioplastic from jackfruit seeds, banana peels, and cassava peels with the addition of Glycerol and without the addition of fillers showed a lower tensile strength value than the minimum standard tensile strength of biodegradable plastic, respectively 0.94 MPa, 0.228 MPa, and 0.21 MPa [25, 26, 33]. The bioplastics from jackfruit seeds with Glycerol and filler MCC also showed lower tensile strength results than the standard. Due to Glycerol and filler’s intermolecular interactions, MCC was antagonistic, which induces the development of a heterogeneous film structure, figuring discontinuities, thus decreasing the tensile strength of the bioplastic [28].

Furthermore, bioplastics based on cassava peel starch with Glycerol and chitosan, bioplastics based on taro starch with Glycerol or sorbitol, and filler clay produce tensile strength values that conformed to the conventional plastic tensile strength standards. The importance of these treatments was 43.22 MPa, 76.57 MPa, and 89.32 MPa, respectively. This condition indicates an excellent intermolecular interaction between the type of starch, plasticizers, and fillers used [35, 45].
3.3. Effect of Starch, Plasticizers, and Filler Types on the Elongation of Bioplastics

The assessment of the elongation quality in bioplastics is strongly influenced by the type of starch source, starch content, and additional materials such as plasticizers and fillers. Glycerol was the most widely used plasticizer due to the best interaction ability compared to other plasticizers when combined with starch with different characters with or without stuffing. Meanwhile, sorbitol in the production of bioplastics was very dependent on the starch’s characteristics. It is necessary to add certain fillers that can improve the resulting bioplastic’s mechanical quality according to starch’s characteristics.

Bioplastics from several starch sources with the addition of Glycerol in a concentration range of 4 - 43% and without the addition of fillers showed 8.1 - 50.98% elongation results. None of these values comply with the elongation standards for biodegradable plastics. However, bioplastics made from mango seeds, corn, potato peels, sweet potatoes, and banana peels had elongation values that meet the standard for conventional plastics and have the highest elongation values. Compared to the production of sweet potato starch-based bioplastics with the addition of 25% glycerol and 10% chitosan, the elongation value was 38.50% [38].

| Starch Source | Type of Plasticizers | Type of Filler | Result (%) | Reference(s) |
|---------------|---------------------|----------------|------------|---------------|
| Mango seeds, Yam, Corn, Potato peels, Banana peels | Glycerol (4-43%) | Non-filler | 21.11 – 50.98 | [1, 13, 36, 38, 41] |
| Cassava peels, Cassava, Taro, Sago, Banana peels | | Non-filler | 8.1 - 18.77 | [24, 25, 33, 34, 39, 43] |
| Banana peels | | Chitosan (30%) | 24.85 | [37] |
| Avocado seeds, Jackfruit seeds | MCC (10-40%) | 13.36 – 15.67 | [28, 42] |
| Durian seeds | Sorbitol (40%) | Chitosan (10%) | 48.68 | [10] |
| Biodegradable plastic standard by SNI 7818:2014 | | | 400 – 1,220 | [40] |
| Conventional plastic standard by SNI ISO 17557:2011 | | | 21 - 220 | [44] |

At the same proportion of materials and composition, bioplastics without using fillers resulted in a more excellent elongation value of 43%. However, the elongation value from bioplastics with chitosan filler or without filler has met conventional plastic standards’ elongation value. This shows that the increasing number of fillers can cause the elongation value at the bioplastic break to decrease [10]. Due to the growing density of intermolecular bonds in bioplastics, the increase in hydrogen bonds when chitosan was added, thus the bioplastic formed was increasingly strong and less elastic. The type and concentration of plasticizers play a significant role in Elongation when the bioplastic breaks [10]. Without plasticizers, amylose and amyllopectin will form a film structure with an inhomogeneous composition distribution, where the interaction between amylose and amyllopectin molecules makes the bioplastic film brittle and stiff.

Bioplastics with Glycerol and filler MCC’s addition produced the lowest elongation value compared to other variables. The use of a glycerol concentration of 30% and MCC concentration of 40% for durian seed starch resulted in an elongation of 15.67% [42]. This value was higher than the bioplastics, which added 3% glycerol and an MCC concentration of 10% [28]. These results indicate that the higher the MCC plasticizer concentration, the higher the elongation value.

The addition of plasticizers was directly proportional to the percentage of Elongation. The addition of plasticizers can reduce the brittleness and increase the polymer film’s flexibility by breaking the hydrogen bonds between adjacent polymer molecules. Meanwhile, the addition of filler resulted in a decrease in the elongation value. Many chitosan fillers cause a reduction in
the intermolecular bond distance; therefore, the plasticizer molecules were in separate regions outside the polymer phase. The decrease in the bond distance was due to the increasing number of hydrogen bonds formed between chitosan molecules with amylose and amylopectin. The bioplastic becomes stiffer and less elastic [1]. Bioplastics based on durian seed starch with a sorbitol concentration of 40% and a chitosan filler of 10% resulted in an elongation value of 48.68%. Compared with the tensile strength of the same material, the value is 19.36 MPa [10]. From these results, the value of Elongation and tensile strength is the best. Making bioplastics cannot form the same high Elongation and tensile strength because of their opposite properties. Thus, it is necessary to know in advance whether the objective of making bioplastic was to obtain a higher elongation value or tensile strength.

4. Conclusion
The tensile strength and elongation values of bioplastic from various treatments showed a relatively large range of results. Glycerol was the most widely used plasticizer because Glycerol has the best interaction ability compared to other plasticizers when combined with starches with different characters, either by adding various types of fillers or without adding fillers. The types of fillers that were commonly used are chitosan, clay, and ZnO. The use of plasticizers and fillers gives an opposite contribution to the quality of tensile strength and Elongation.

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