

MECHANICAL AND BIODEGRADATION PROPERTIES OF CORNSTARCH-BASED BIOPLASTIC MATERIAL

A.B.D. Nandiyanto^{1*}, M. Fiandini¹, R. Ragadhita¹, A. Sukmafitri¹, H. Salam²,
F. Triawan³

¹Departemen Kimia, Universitas Pendidikan Indonesia, Jl. Dr. Setiabudi no 229, Bandung, Indonesia

²Departemen of Mechanical Engineering Education, Universitas Pendidikan Indonesia,

Jl. Dr. Setiabudi no 229, Bandung, Indonesia

³Department of Mechanical Engineering, Faculty of Engineering and Technology, Sampoerna University, Jl.

Raya Pasar Minggu No. 16, Jakarta, Indonesia

*e-mail: nandiyanto@upi.edu

Abstract. The purpose of this study was to investigate mechanical and biodegradation properties of bioplastic from pure cornstarch (with no additional reinforcing components). The following fabrication procedure was done: (1) diluting cornstarch in water, (2) making homogeneous mixture of the diluted cornstarch, glycerol, and acetic acid by heating at less than 100°C, (3) molding process, and (4) drying process to get a solid bioplastic. The present bioplastic had good biodegradability properties, degraded easily within 2-week soaking in water, confirmed by the weight loss and appearance of fungi on its surface. Good mechanical performance was obtained although it is still low compared to the standard bioplastic in moderate grade. Indeed, additional reinforcing components (e.g., co-polymer or additive) is required for improving the mechanical properties.

Keywords: bioplastics, cornstarch, education, tensile strength, Young's modulus

1. Introduction

Plastic is one of the most used materials, which mostly can be found in food packaging materials (food industry) and even household materials (non-food industries) [1-4]. The excellent performance of plastic (such as lightweight, relatively inexpensive, flexible, and waterproof [3,4]) makes this material inseparable from daily life. However, global uses of plastics face great issues for the environment. The waste of plastic continuously increases from time to time, creating problems in the increases in a huge amount of plastic waste [5-7]. Plastic waste has properties that are very difficult to be degraded by microbes in the environment since most plastics are made from synthetic or semi-synthetic materials such as polypropylene, polystyrene, and poly (vinyl chloride). Indeed, in nature, the degradation of plastics prepared from these raw materials takes a long time until hundreds or even thousands of years to break the carbon chain [1,8].

Efforts have been made to handle plastic waste issues. Strategies have been applied from the recycling process to the most extreme process (i.e., burning process) [8]. Although the method can reduce the number of waste, both methods still create new problems. For instance, recycled plastic can cause side effects on contaminating the product, especially when it is used as a food packaging material due to the presence of certain potentially carcinogenic substances [9]. Burning plastic waste can pollute the air because the smoke

produced contains hazardous chemicals such as dioxin [8]. Since plastic cannot be replaced by other materials, some researchers suggested developing bioplastics that are certainly more environmentally friendly [1,10]. Bioplastics are plastics made from natural polymeric materials such as starch, vegetable oil, cellulose, lignin, and also materials derived from animals such as proteins and lipids. Bioplastics are easily degraded by microbes, and the degradation process does not take a long time [5,8]. Starch is one of the most common raw materials used for bioplastic fabrication in replacing plastic polymers [11]. The bioplastic produced from starch has a high biodegradability (quickly decomposes) in the soil. Further, in agricultural countries such as Indonesia, starch is largely available and relatively inexpensive [5,12].

Many reports on the successful synthesis of bioplastic from starch have been documented. Current reports on the synthesis of bioplastic are presented in Table 1. Although strategies have shown a great impact on the successful synthesis of bioplastic, for some cases information about mechanical properties as well as biodegradability of bioplastic is not well included. Specifically, reports on the pure bioplastic without additional reinforcing components are still rare. In fact, information on properties of bioplastics with no additional reinforcing component is important for understanding further development of bioplastics, including knowing what additive is fit with pure bioplastics.

Table 1. Example of the current reports on the synthesis of bioplastic

Type of carbohydrate	Raw material	Results	Ref.
Cassava starch	Cassava starch, fiber, chitosan, and polystyrene foam	Foam tray from cassava starch with fiber and chitosan had the same mechanical properties as polystyrene foam	[2]
	Tapioca starch, cornstarch, polylactide, and methylene diphenyl diisocyanate (MDI), glycerol	The effect of adding MDI increased the tensile strength of bioplastics and great thermal properties. When without adding MDI, bioplastic surfaces were evenly distributed. On the contrary, the addition of MDI allowed the formation of aggregates on bioplastics surface	[6]
	Cassava starch, polyvinyl alcohol (liquid), talc powder, glycerin, urea, and water	Bioplastics from cassava starch had the highest tensile strength than polyvinyl alcohol and paper. Biodegradability of bioplastic is slower than that of paper, which is because the paper is more organic than bioplastics	[8]
	Cassava starch, glycerol, yerba mate extract	The addition of yerba mate extract accelerated the biodegradability of bioplastic in the soil	[9]
	Cassava starch, glycerol, and water	The mechanical properties of bioplastics were influenced by glycerol and starch content. Glycerol content improved the elongation but it decreased the tensile strength and Young's	[10]

		modulus. Conversely, starch content increased the tensile strength and Young's modulus. Bioplastics with higher glycerol and starch content had a tensile strength of 7.0 MPa and elongation of 132.1%	
	Tapioca starch, glycerol, potassium hydroxide, sodium hydroxide, sodium chloride, acetic acid, sulfuric acid, and Cellulose Nanofibers	The addition of cellulose nanofibers as a reinforcing component increased tensile strength, thermal stability, absorption properties, and crystallinity	[13]
	Cassava starch, sorbitol, low-density polyethylene (LDPE) and montmorillonite (MTT)	Additional LDPE provided good mechanical properties. LDPE allowed uniform and homogeneous morphologies compared to MTT	[14]
	Tapioca starch, zinc oxide as repair agents, glycerol, Escherichia coli, and staphylococcus aureus	The addition of 2% ZnO increased tensile strength and elongation. The thickness of biofilms that met Japanese Industrial Standard (JIS) is 0.1087 mm	[15]
Potato starch	Potato starch, yam starch, hydrochloric acid, and sodium hydroxide	Bioplastics made from potato starch and yam starch had a tensile strength of 0.6 MPa and 1.9 MPa. They experienced 50% of weight loss at temperatures of 250°C and 310°C. Biodegradation in the soil takes 1 week	[16]
Cassava peel	Cassava peel, sorbitol, and microcrystalline cellulose (MCC)	The effect of additional microcrystalline cellulose increased tensile strength. Bioplastics with the addition of 6% MCC had the highest tensile strength values. However, the addition of MCC decreased elongation, density, and water absorption	[5]
Avocado seed	Avocado seed, microcrystalline cellulose, glycerol, hydrochloric acid, potassium hydroxide, Sodium hypochlorite, sugar palm fibers, and water	Bioplastics from avocado seeds with the addition of microcrystalline cellulose obtained the best mechanical properties with a ratio of 7: 3 ratio with 2.74 MPa tensile strength and an extension of 3.61%. Bioplastic surface morphology is uneven and hollow	[4]
Durian seed	Durian seeds, ultrapure water, chitosan, acetic acid (0.1%), and lime water	Bioplastic from durian seeds had a tensile strength of 0.1158 MPa compared to the standard value, elongation of 2.1875%, and Young's modulus of 4.1515 MPa. The test results of bioplastics mechanical properties compared to bioplastic	[17]

		moderate are still far from the standard	
Cornstarch	Cornstarch, sodium benzoate, dimethylsulfoxide, and photosensitizer	The best condition was obtained by adding 0.75% of sodium benzoate. The surface morphology is uniform and homogeneous	[7]
	Cornstarch, glycerol, and sorbitol	Bioplastics by casting method had a lower thickness and had a greater tensile strength compared to that by pressing and blowing method	[11]
	Cornstarch, polylactide, lysine diisocyanate (LDI)	Cornstarch decreased the thermal stability of bioplastic. LDI increased the temperature of thermal degradation (compared to bioplastic without LDI) and decreased biodegradability. LDI allowed a homogeneous surface morphology	[12]
	Cornstarch, potassium hydroxide, formaldehyde, resorcinol, and <i>Saccharum spontaneum L.</i> (SS)	Addition of SS as an amplifier improved mechanical properties and thermal stability. Biopolymer degradation time with the amplifier requires 60 days	[18]
	Cornstarch, taro starch as, glycerol, and distilled water	Taro starch improved mechanical properties and thermal stability	[19]
	Cornstarch, banana pseudostem fiber (BSF), and glycerol	10% of BSF increased tensile strength and Young's modulus. The surface morphology of the fill particle in the aggregate bioplastic matrix	[20]
	Cornstarch, polydimethylsiloxane (PDMS), and Acetoxypolysiloxane (Acetoxy-PDMS; Elastosil E43)	Bioplastic prepared with PMDS has good elongation and tensile strength compared to that without PMDS. SEM Surface morphology was uniform and homogeneous	[21]
	Cornstarch, glycerol, white vinegar, titanium dioxide nanoparticles (TiO ₂) as reinforcing agents	TiO ₂ increased the tensile strength of bioplastics from 3.55 to 3.95 MPa, decreased elongation from 88 to 62%, and increased decomposition temperature. Morphology was a homogeneous surface with fewer cracks	[22]
	Cornstarch, distillers dried grain (DDGS), and phenolic resin	DDGS and phenolic resins improved mechanical properties. 25% of DDGS concentration gave good tensile strength. Up to 75% of DDGS increased biodegradability from 0 to 38% and reduced surface hardness	[23]

	Cornstarch, polypropylene (PP), calcium carbonate, Maleic anhydride grafted polypropylene (MAPP), Curauá fibers (CF), and potassium hydroxide	The addition of CF increased hardness, tensile strength, and Young's modulus, as well as decreased biocomposite extension. Bioplastic had a homogeneous surface with the addition of CF and MAPP	[24]
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2. Material and Method

Preparation of Cornstarch-based Bioplastic Material. Several chemicals used in this study were micron-sized cornstarch (purchased from PT Egafood, Jakarta, Indonesia), acetic acid 25% (purchased from Sakura Medical Stores, Bandung, Indonesia), and glycerol 95% (purchased from Sakura Medical Stores, Bandung, Indonesia).

Bioplastic fabrication was carried out through the following steps. In the initial stage, cornstarch was mixed with glycerol with a composition ratio of 1:1. Then, a mixture of cornstarch and glycerol was then mixed with 25% of acetic acid. The mixture was then homogeneous stirred manually. At the same time with the manual mixing process, the mixture was heated at 60°C for 20 minutes using an electrical heater to obtain a viscous product. The viscous product was then molded and dried at room temperature for more than 24 hours until it forms a solid white film.

Physicochemical properties. The morphology of the prepared samples was analyzed using a Digital Microscope (BXAW-AX-BC, China) and a Scanning Electron Microscope (SEM; JSM-6360LA; JEOL Ltd., Japan). To support the analysis, we conducted characterizations using a Fourier Transform infrared (FTIR-4600, Jasco Corp., Japan) to the prepared film.

Mechanical properties. To measure the mechanical properties, the formed film sample was sliced into size of about 5 × 5 mm. To ensure the measurement reliability, we also tested the samples with various thicknesses. In short, the mechanical tests were carried out by performing tensile and hardness tests to the sliced samples [25]. The tensile properties were tested out on a Control Universal Testing Machine at a crosshead speed of 2 kg/min with the load cell of 50 kN. The hardness Shore D tests were carried out by Mitutoyo Shore D hardness tester.

Tensile strength is defined as the maximum force that can be held by the specimen when stretched or pulled before the material is broken. Tensile strength was calculated with the following Eq. (1) [26].

$$\text{Tensile strength} = \frac{F_M}{A_0} \quad (1)$$

F_M is the maximum stress (N), A_0 is the material cross-sectional area (mm²), and the unit of tensile strength is MPa.

Elongation at break is an extension of a material when tested tensile until finally fracture. The elongation at break is quantified by Eq. (2) [26].

$$\text{Elongation at break} = \frac{L - L_0}{L_0} \cdot 100\% \quad (2)$$

where L is the final elongation of the material (mm), and L_0 is the initial length of the material (mm).

Young's modulus is a measure of stiffness to elastic deformation under load. Young's modulus (in MPa) was calculated with the following Eq. (3) [26].

$$\text{Young's modulus} = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \quad (3)$$

where ε_1 and ε_2 are the condition of relative elongation at 0.05 and 0.25%, respectively. σ_1 and σ_2 are the stress that occurs at ε_1 and ε_2 , respectively. The Young's modulus unit is MPa.

Biodegradability. The biodegradability tests were conducted by slicing the prepared bioplastics with sizes of about 5×5 mm and then immersing it into ultrapure water. The weight losses of the sample were measured at the interval time of one day. In line with this test, during the immersing process, it was also visually observed the change of color.

3. Results and Discussion

Figure 1(a) shows the photo image of the sliced as-prepared bioplastic. The bioplastic was white, solid, and dense. Figure 1(b) is the appearance of bioplastic after immersing for 1 week in water. The color of the bioplastic started to change from white into yellowish-white. Cracks were found, which is due to the swelling phenomenon. Figure 1(c) is the photo image of bioplastic after 2-week immersion in water. Different from the image in Fig. 1(a), the color of 2-week immersion bioplastic was yellowish-brown. Severe cracks were obtained, which are worse than the 1-week immersion sample.

To clarify the structure of the bioplastic, SEM analysis was conducted (see Figs. 1(d) and (e)). The results in Fig. 1(d) showed that the as-prepared bioplastic was dense, which was confirmed by the high-magnified SEM image in the insert. The surface of the sample contained a packed ball-like structure, in which this is believed from the micron-sized starch. Different from Fig. 1(d), bioplastic after 2-week immersion shows different surface structures. A worm-like structure was obtained on the surface of bioplastic (see insert image in Fig. 1(e)).

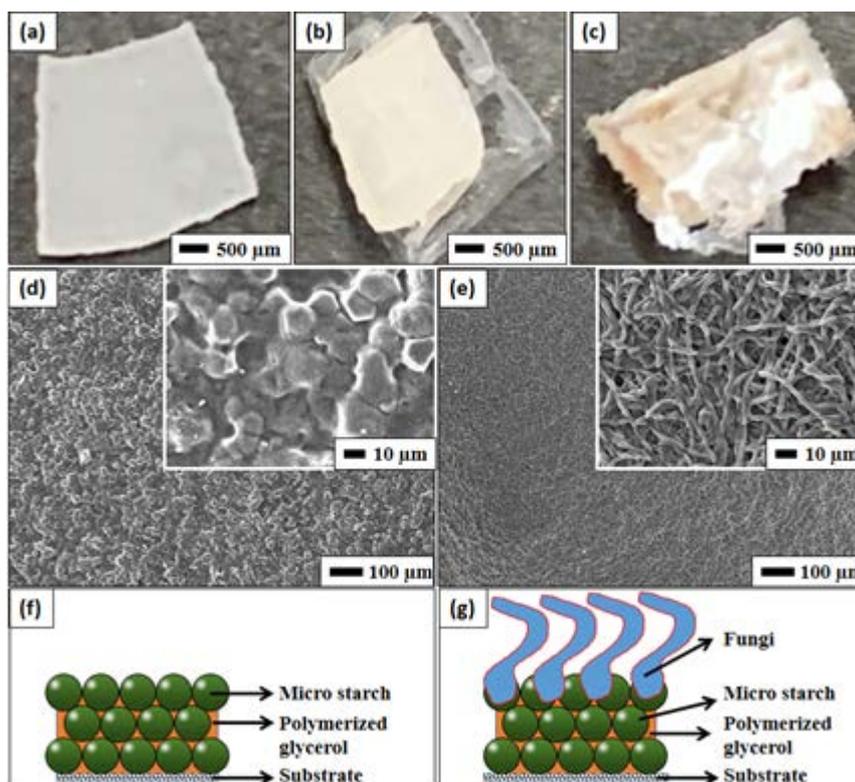


Fig. 1. Photograph (a-c) and SEM (d, e) images of bioplastics. Figures (a) and (d) are the images of as-prepared bioplastics. Figure (b) is the image of bioplastics after 1-week immersed in water. Figures (c) and (e) are the images of bioplastics after 1-week immersed in water. Insert image in the top right in Figures (d) and (e) is the high-magnified SEM image

To understand the structure of the SEM image, illustrations models of samples before and after 2-week immersion are shown in Figs. 1(f) and (g), respectively. The as-prepared sample contained packed balls-like structures (see Fig. 1(f)), in which the micron balls were from micron starch. The structure is dense and the area between micron balls was filled by

polymerized glycerol. Then, when the sample was immersed after several days, the fungi started to form (see Fig. 1(g)), in which this is from the decomposition of micron starch. This is confirmed by the existence of a worm-like structure without the appearance of micron balls on the surface of the film (see Fig. 1(e)).

To confirm the phenomena during the immersion process, Fig. 2 shows the results of the FTIR analysis of various materials, including glycol, corn powder, as-prepared bioplastic, 1-week immersed bioplastic in water and the surface of 1-week immersed bioplastic sample. Several identical peaks were identified at the wavelength of 1035, 1740, and 3313 cm^{-1} [14,20-22], confirmed hydrocarbon structure peaks [27]. The identical peaks indicate that there is polymerization of the corn powder monomer. No additional peaks were detected in the as-prepared bioplastics compared to glycol and corn powder, informing that formation by bioplastic from the polymerization reaction only.

The comparison of the FTIR peaks for bioplastics before and after 1-week immersion in water confirms that the biodegradability in water was the only dilution of the outer component on the bioplastics. The reaction between water and bioplastics involved the dilution process and did not interfere with the complicated reaction [20].

We also found that 2-week immersion results in the appearance of fungi on the surface of bioplastic. The analysis of the surface of bioplastic showed that the fungi degrade the bioplastics, changing the chemical structure of bioplastic into fungi's structure (see the red dashed area in Fig. 2) [20].

To confirm the weight losses during the immersion process, we analyzed the mass of bioplastic as a function of the day (see Table 2). Table 2 shows the results of bioplastic weight loss carried out for a week. The results showed that bioplastics' weight decreased for 3 days immersion in water.

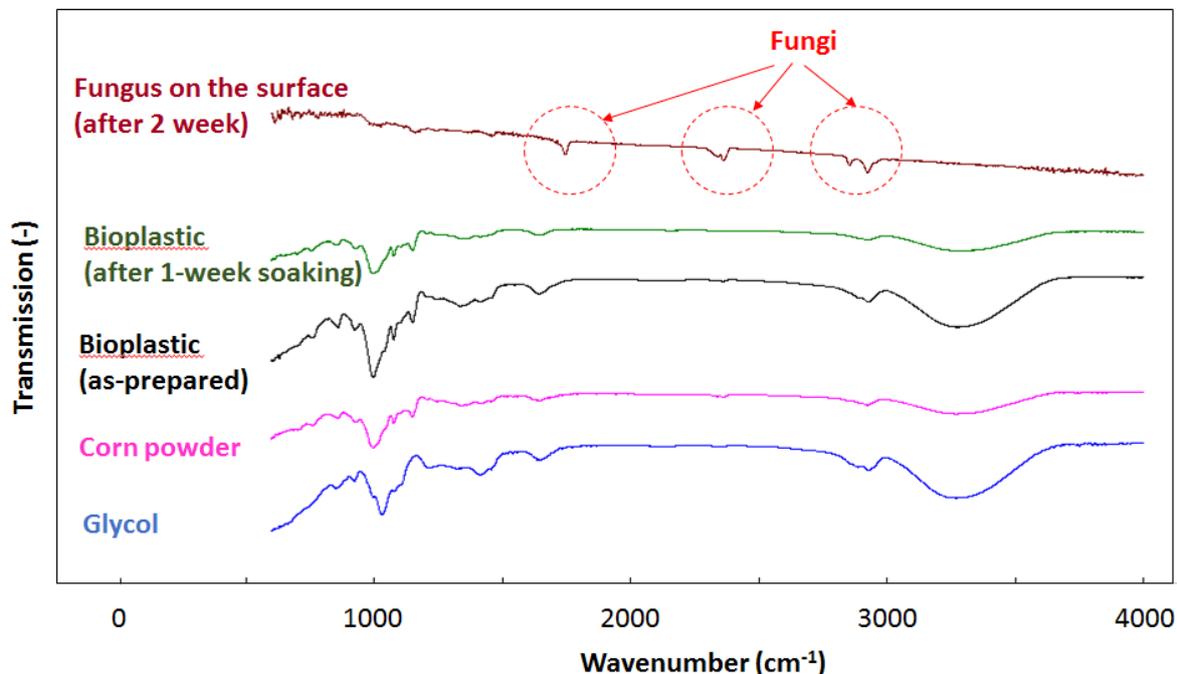


Fig. 2. FTIR analysis results of glycol, corn powder, as-prepared bioplastic, 1-week immersed bioplastic in water and fungus on the 2-week immersed sample

Table 2. Weight loss bioplastics during the immersion process

Days	Initial Dimension, cm ²	Initial mass, g	Mass after Immersion, g	Mass loss, wt%	Decay dimension, g/cm ²
1	0.788	0.040	0.036	10	0.005
2	0.927	0.054	0.050	7	0.004
3	0.916	0.049	0.035	29	0.015
4	0.819	0.041	0.031	9	0.004
7	0.768	0.034	0.027	34	0.018
8	0.866	0.040	0.036	10	0.005
9	0.860	0.034	0.031	18	0.007
10	0.908	0.027	0.019	30	0.009

Figures 1(f) and (g) show the illustration of the biplastic before and after the degradation, respectively. This illustration shows the good correlation between the above FTIR analysis in Fig. 2 and the weight loss analysis in Table 2. The possible weight loss during 1-week immersion is because the bioplastics' outer surfaces were diluted in water, confirmed by the identical FTIR patterns. This result is different for 2-week immersion biplastic, in which the mass loss was followed by the appearance of fungus (see Fig. 1 (e)) and fungus chemical structure (see Fig. 2). The present bioplastics were made from cornstarch, making microorganisms to more easily break the polymer chain inside the bioplastics themselves [14-16,20].

Figure 3 shows the proposal formation mechanism of bioplastics. In short, the polymerization was started from the interaction between micron starch and glycerol (see route R1). Then, adding heat treatment and catalyst (such as acetic acid), the interaction continues to the formation of glycerol-starch bonding (by releasing OH group). When there are other movements of other micron starch (see route R2) to the surface of the glycerol-starch component (see route R3), other polymerization happened. This makes the final component contained packed balls-like structure, as confirmed by the SEM image in Fig. 1(d) and illustration model in Fig. 1(f).

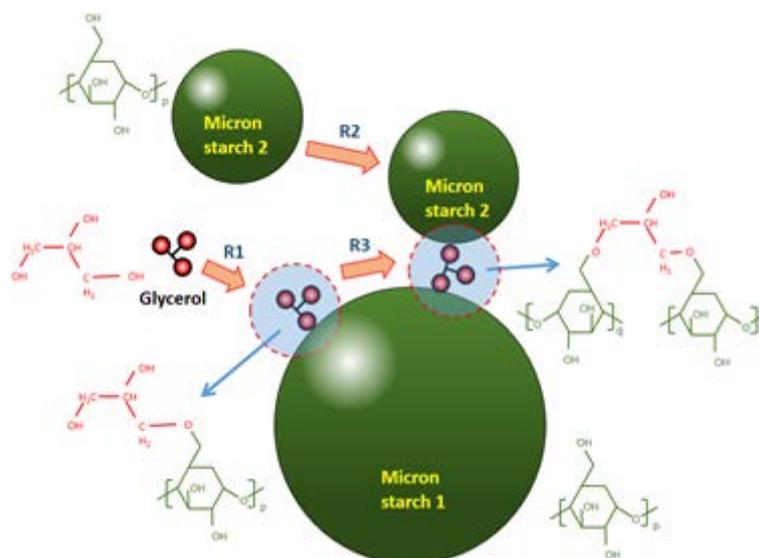


Fig. 3. The proposal formation mechanism of biplastic. R1 is the route for the diffusion of glycerol to the surface of micron starch. R2 is the movement of other micron starch. R3 is the polymerization involving the connection of micron starches

Figure 4 depicts the mechanical properties of cornstarch bioplastics. The tensile properties' values are added in the inserted table in Fig. 4. We also presented the hardness test results in Table 3, which indicate different mechanical characteristics as those from the tensile tests. The different performance is likely due to the inhomogeneity effect of bioplastics in the material [26]. Different types of molding processes play important roles, making different types of polymerizations [16,17].

In the case of the tensile strength (the maximum load that a material can withstand before it breaks [28]) bioplastics with smaller dimensions have greater tensile strength than that with a larger dimension. Smaller tensile strength indicates that the material can easily deform in plastic behavior [9]. The sample with a smaller dimension has a higher degree of homogeneity (compared to larger dimensions), causing the distribution of bioplastics constituent molecules to be consistently distributed. On the contrary, the larger dimension has a smaller degree of homogeneity, making an uneven distribution of bioplastic constituent molecules [17].

The inserted table in Fig. 4 presents the elongation at break of the samples. Higher elongation value means that the bioplastic sample is more deformable. The sample with larger dimension has higher elongation value than that with a smaller dimension, indicating the larger sample can hold its shape better than the smaller size under the same elongation. This unique deformation behavior is likely due to the uneven distribution of bioplastics' constituent molecules inside the sample body. Indeed, this reduces the strong intermolecular interactions between starch molecules (due to the longer hydrogen bonding) [15].

The stiffness of a material is confirmed by evaluating the Young's modulus (see inserted table in Fig. 4). The value of tensile strength is directly proportional to the value of Young's modulus. Young's modulus evaluates the stiffness of the produced material. The greater Young's modulus value has correlations to more stiffness of the material. Sample with smaller dimensions has better stiffness than that with a larger dimension, which is in good agreement with [17].

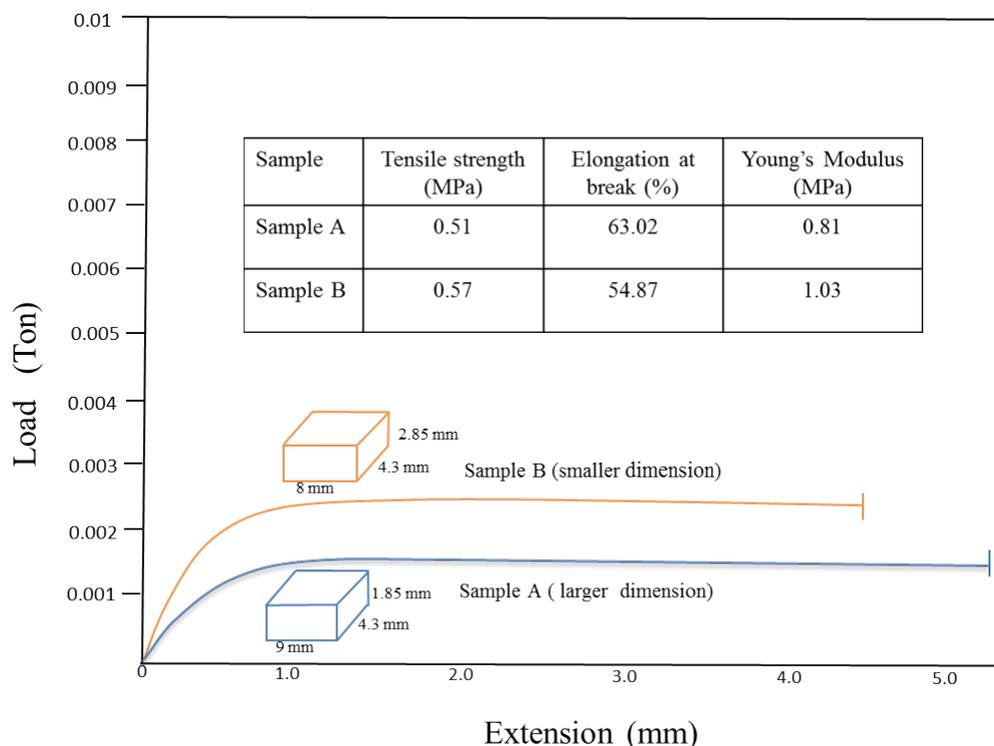


Fig. 4. Tensile tests result of cornstarch bioplastic

Table 3 shows the results of hardness tests on the produced bioplastics to evaluate its response when it is used under compressive load. Based on the obtained results, it was found in average that the sample possessed a hardness value of 25.67 D with thicknesses ranging from 1.70 to 3.00 mm. If the hardness value is expressed per one mm thickness, the sample with 1.7 mm showed significantly larger value compared with others. This indicates that the thickness or size affected the hardness of material. However, if the 1.7 mm sample is excluded, counting only the other 5 samples with thickness of more than 2.45 mm, the hardness value per one mm thickness showed small deviation, which is 8.64 ± 1.27 D/mm. This shows that a more reliable hardness value could be obtained from a specimen with larger thickness. In order to obtain a more holistic understanding, a hardness test using nano-indentation test technique can be considered for the future work to omit the size effect [29,30].

In overall, fabricated bioplastic samples are still far from the standard for moderate quality of bioplastic. This is because the present bioplastic is formed from pure cornstarch (without additional reinforcing component). The standard values of tensile strength, elongation, and Young's modulus for moderate quality bioplastics are 1-10 MPa, 10-20%, and 2.72 MPa respectively [17], when applying this method for producing bioplastics with a moderate grade, we need to put other reinforcing chemicals. Otherwise, we cannot get the obtainment of higher-grade quality of bioplastic.

Table 3. Hardness test results of cornstarch bioplastic

No	Hardness, Shore D	Thickness, mm	Hardness value per 1 mm thickness, shore D/mm
1	36	1.70	21.17
2	21	2.45	8.57
3	19	2.55	7.45
4	27	2.70	10.00
5	29	2.95	9.83
6	22	3.00	7.33
Average	25.67		

5. Conclusion

The mechanical and biodegradation properties of the cornstarch-based bioplastic without additional reinforcing components have been evaluated. The analysis was completed with the proposed mechanism for the formation of bioplastic based on characterization analysis. The results showed that the prepared cornstarch-based bioplastic had good biodegradability properties although the mechanical properties have not yet reached standard value for the moderate grade of bioplastic. To improve the mechanical properties, additional reinforcement material must be added. The present bioplastic is stable in the open air, and it started to degrade after 5-day soaking in water, shown by the appearance of fungi on its surface. This informs that this bioplastic is good to be used as a packaging material as long as in the dry condition. Thus, directly disposing of this material after using will not create problems for the environment.

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