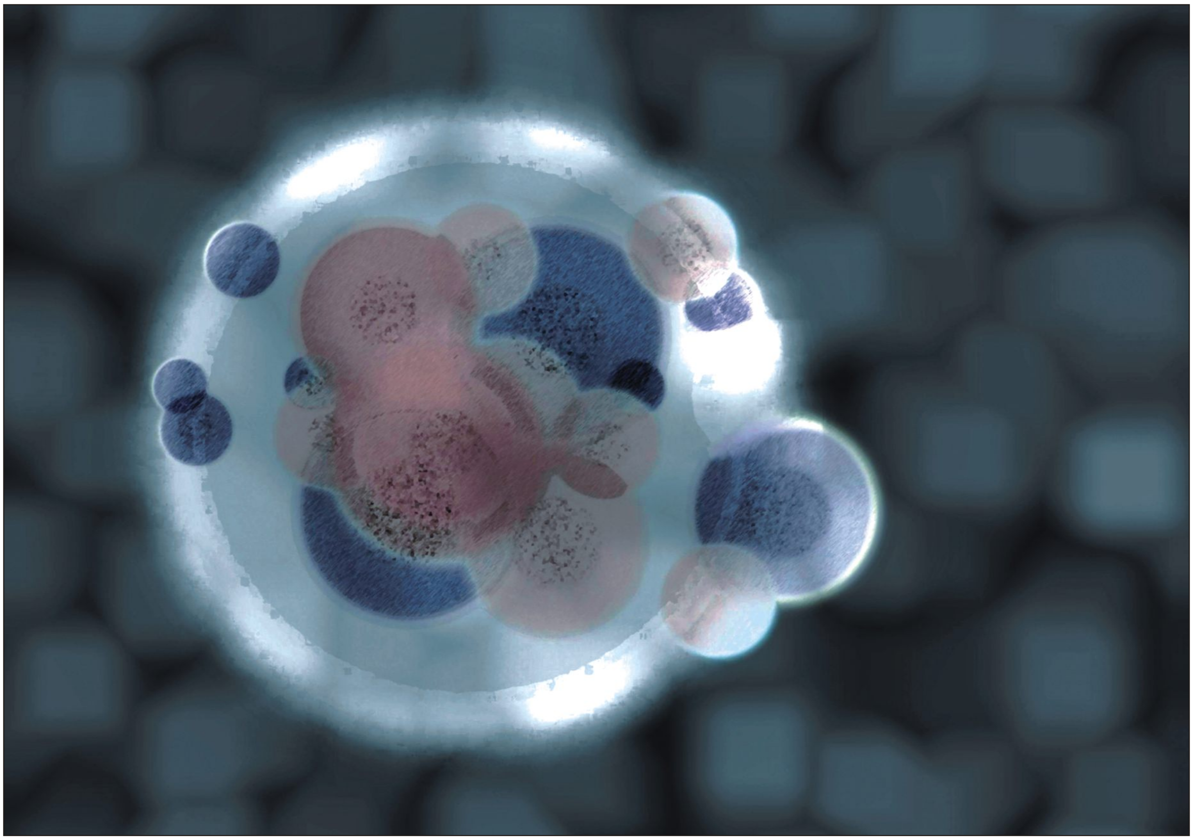

Journal of Biological Sciences

ISSN 1727-3048

ANSInet
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OPEN ACCESS **Journal of Biological Sciences**

ISSN 1727-3048
DOI: 10.3923/jbs.2019.57.64

Research Article

Fabrication and Characterization of Sweet Potato Starch-based Bioplastics Plasticized with Glycerol

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Abstract

Background and Objective: Poor biodegradability and the contamination risk of petrochemical-based plastics encouraged the utilization of renewable resources to replace them due to their inexpensive, renewable, biodegradable and compostable properties. This study aimed to investigate the utilization of sweet potato sourced from Indonesia as a base material of bioplastic and its characteristics for food packaging application. **Materials and Methods:** Starch was extracted from sweet potato and bioplastics was prepared by mixing starch with glycerol using different starch:glycerol w/w ratio (2.5:1, 2.75:1, 3:1 and 3.5:1). Intermolecular interactions and microstructure of bioplastics were assessed by using FTIR and SEM, respectively. The physical and mechanical properties were evaluated by measuring the density, tensile strength and elongation at break. The relative hydrophobicity was examined by measuring the water contact angle. The biodegradability was also investigated with the aid of enzymatic degradation by microbes. **Results:** Microstructure of bioplastics showed the incomplete gelatinization with the increase of starch:glycerol ratio indicated by the visible inhomogeneous granules. FTIR spectra exhibited that the intermolecular interaction in bioplastics occurred through C-O-H, O-H, C-H aliphatic and C=O groups. Mechanical properties evaluation showed that bioplastic with 3.5:1 (starch:glycerol) ratio exhibited the highest tensile strength of 2.57 MPa with the lowest elongation of 6.27%. Bioplastic with 3.5:1 (starch:glycerol) ratio also showed the highest density and contact angle of 1.66 g cm^{-3} and 50.1°, respectively. The fastest enzymatic degradation showed by the highest microbial growth was presented by bioplastic with 3.5:1 (starch:glycerol) ratio as well. **Conclusion:** Bioplastic with the highest sweet potato starch:glycerol ratio showed the most excellent physical, mechanical and biodegradability properties.

Key words: Sweet potato starch, bioplastics, glycerol, tensile strength, food packaging application and biodegradability

Received: October 02, 2018 Accepted: November 07, 2018 Published: December 15, 2018

Citation: Akbar Hanif Dawam Abdullah, Sri Pudjiraharti, Myrtha Karina, Oceu Dwi Putri and Rani Hasna Fauziyyah, 2019. Fabrication and characterization of sweet potato starch-based bioplastics plasticized with glycerol. J. Biol. Sci., 19: 57-64.

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Petrochemical-based plastics such as polyethylene terephthalate (PET), polyvinylchloride (PVC), polyethylene (PE), polypropylene (PP), polystyrene (PS) and polyamide (PA) have been widely used as packaging materials due to their large availability and affordability. By virtue of its flexibility, petrochemical-based plastics were easily formed into sheets, shapes and structures. Moreover, the appearance of excellent mechanical performance such as tensile and tear strength, good barrier to oxygen and carbon dioxide and heat stability of these petrochemical-based plastics, increased their use for packaging materials. However, their use has been reduced currently because of their poor biodegradability which causes severe environmental problems[1,2]. Furthermore, any chemicals from these packaging materials migrated into food and contaminate the foodstuffs. The contamination at the certain level would be poisoning and risking for human health[3]. To overcome these drawbacks, plastics based on renewable resources which are biodegradable and non-toxic would be needed as a replacement for synthetic plastics.

Biodegradable plastics or bioplastics are fabricated from biopolymers obtained from biomass, such as starch, cellulose and proteins. Among those biopolymers, starch would be desirable for the manufacturing of bioplastics because it is inexpensive, renewable, biodegradable and compostable[4]. Nevertheless, starch-based plastics also showed several limitations, such as brittleness, high sensitivity to moisture and poor mechanical strength, compared to synthetic stretchable plastics made from high-density polyethylene (HDPE) or low-density polyethylene (LDPE). Accordingly, the modification of starch-based plastics has been performed to improve their performance. The use of a plasticizer is the simplest and most effective approach to enhance the flexibility and extensibility of starch-based plastics. The most common plasticizer used in the manufacturing of bioplastics is glycerol, a small molecule which is non-toxic that reduced intermolecular polymer interactions and increased intermolecular spacing by gaining access to the polymer chains through hydrogen bonds and consequently increases the stretchability and improves the melt flow ability of bioplastics[5,6].

Sweet potato, one of the sources of starch, can be easily found in the tropical zone especially in Indonesia. The relative easiness for growing and low-cost cultivation, made people have a preference to use sweet potato in providing their food consumption and raw material for industrial manufacturing[7]. Sweet potato contains a 50-80% starch on a dry basis and sweet potato starch comprises 70-80% of highly branched amylopectin and 20-30% of linear and slightly branched amylose[8]. In addition, sweet potato starch has comparable properties with ordinary potato starch which is rich in dietary fiber, minerals, vitamins and antioxidants, such as phenolic acids, anthocyanins, tocopherol, β-carotene and ascorbic acid which could be migrated into food in case that sweet potato starch-based plastics are used for food packaging and would enhance the nutritional value of the food package[9].

To the best of authors' knowledge, little information is presented concerning the fabrication and characterization of sweet potato starch-based bioplastic for food packaging application in the literature. Therefore, this study aimed to investigate the effects of different starch: glycerol ratio on the physical, mechanical and biodegradability properties of bioplastics.

MATERIALS AND METHODS

This study was performed from March-July, 2018 in Bioplastic Laboratory, Indonesian Institute of Sciences (Bandung, Indonesia).

Materials: Native starch was obtained from one variety of sweet potatoes cultivated in west Java, Indonesia. Glycerol (99% purity) and *Aspergillus Niger* sp. were purchased from Merck (Darmstadt, Germany) and from InaCC-LIPI, respectively. Potato dextrose agar (Oxoid, UK) and all other chemicals were analytical grade and were purchased from Merck (Darmstadt, Germany).

Methods

Starch extraction and preparation of bioplastics: Sweet potato was ground in a blender (Phillips HR2118/01) in distilled water (1:2, w/v) until the powder was very fine. The crushed mass was filtered through cotton cloth. The starch suspension obtained was decanted for 5 h at room temperature. After decantation, the liquid was removed and the precipitate was collected as wet starch. The wet starch was dried in an oven at 50°C for 24 h.

Bioplastics were prepared by mixing starch with glycerol using different starch: glycerol w/w ratio (2.5:1, 2.75:1, 3:1 and 3.5:1). The mixtures were heated in an oven at 50°C for 1.5 h and were poured into iron molds of 20×20 cm. The samples were pressed by using compression molding at

135°C for 10 min under a load of 50 kg cm^{-2} to form bioplastic sheets. The samples then were stored at 25°C and 55±5% RH for further characterization.

Morphology of bioplastics: The cross-section morphology of bioplastics was observed bya Scanning Electron Microscope (Jeol JSM IT-300, Japan). Prior to SEM analysis, the samples were coated with a thin layer of gold (~10 nm). All samples were analyzed using an accelerating voltage of 20 Kv.

Chemical interaction of bioplastics: Intermolecular bonding of bioplastics was evaluated using a Fourier Transform Infrared (Thermo Fisher Scientific, Nicolet iS5) in a 4000-500 cm^{-1} wave range with an average of 16 scans for spectrum integration and a scanning resolution of 4 cm^{-1} with the aid of attenuated total reflectance (ATR) accessory to collect IR spectra.

Density and hydrophobicity of bioplastics: The density of the bioplastics was determined using a 25 mL pycnometer and a water bath with a constant temperature of 25°C according to ASTM[10] D792-08. Surface hydrophobicity was assessed by means of contact angle measurement[11]. Water droplets (±5 μL) were dropped onto the bioplastics surface (1×1 cm) in ambient condition. The measurement of three different spots on the sample surface was carried out to determine the average static contact angle.

Mechanical properties of bioplastics: The tensile strength and elongation at break of bioplastics were measured on a universal testing machine (UCT-5T, Orientec Co. Ltd, Japan) as Mean±SD (n = 3) according to ASTM[12] D882-12 with initial gauge separation and the crosshead speed were 100 mm and 50 mm min^{-1}, respectively. Bioplastic samples were conditioned at 25°C and 55% relative humidity for 24 h prior to testing.

Biodegradability of bioplastics: Degradation of bioplastics was determined in accordance with Song *et al.*[13]. This test was performed in Petri dishes containing a 3.9 g of potato dextrose agar (PDA) dissolved in 100 mL distilled water. The samples (3×3 cm) were placed on the PDA medium and ±5 μL of *Aspergillus niger* were dropped and spread on each sample surface using a disposable sterile spreader. The Petri dishes were incubated at 37°C and 90% RH for 7 days. Microbial biodegradability was determined by observing the growth of microbes on the samples at an interval time of 1 day.

Statistical analysis: All the specimens used at least three replicates to determine each property. ANOVA and regression analysis were performed in all results by using SPSS 11.0 for Windows (SPSS Inc, Chicago, IL) at a confidence interval of 95%.

RESULTS

Bioplastics appearance: The Fig. 1 showed the appearances of sweet potato starch bioplastics. Irrespective of starch: glycerol ratio, the bioplastics exhibited a yellow color. The increase of starch:glycerol ratio changed the homogeneity of the bioplastics. Bioplastic with the lowest starch:glycerol ratio (Fig. 1a) appeared homogeneous. Conversely, inhomogeneous appearances were obvious when starch:glycerol ratio in the sample was increased (Fig. 1b-d).

Morphology and intermolecular interaction of bioplastics: As Fig. 2 showed the cross-section morphology of sweet potato starch bioplastics. The visible inhomogeneous starch granules indicated the incomplete gelatinization process. The incomplete gelatinization occurred differently for each starch:glycerol ratio at the same heat treatment as shown by the different roughness of bioplastic cross-section. The effective interaction between plasticizer molecules and starch to reduce intermolecular hydrogen bonds within the starch ensued at a relatively low concentration of starch in which the relatively low concentration of starch exhibited smoother surfaces (Fig. 2a, b). The number of inhomogeneous granules increased in accordance with the starch:glycerol ratio in the sample as shown in Fig. 2c, d, which generated bioplastics with a rough surface.

Figure 3 compared the FTIR spectra of sweet potato starch bioplastics with the raw sweet potato starch. The obvious differences between the bioplastics and the raw starch were associated to the semi-crystalline nature of starch in which the peaks at 997 and 1011 cm^{-1} assigned for the C-O-H bending of starch. In addition, the broad peaks at 3000-3600 cm^{-1} were attributed to the hydrogen bonds generated by O-H group interactions of starch and plasticizer. Moreover, the peaks around 2900 and 1600 cm^{-1} were ascribed to C-H aliphatic and C = O, respectively.

Mechanical and physical properties of bioplastics: Tensile strength and elongation of sweet potato starch bioplastics were shown in Fig. 4. From the Fig. 4a, tensile strength of bioplastics increased with the increase of starch:glycerol ratio.

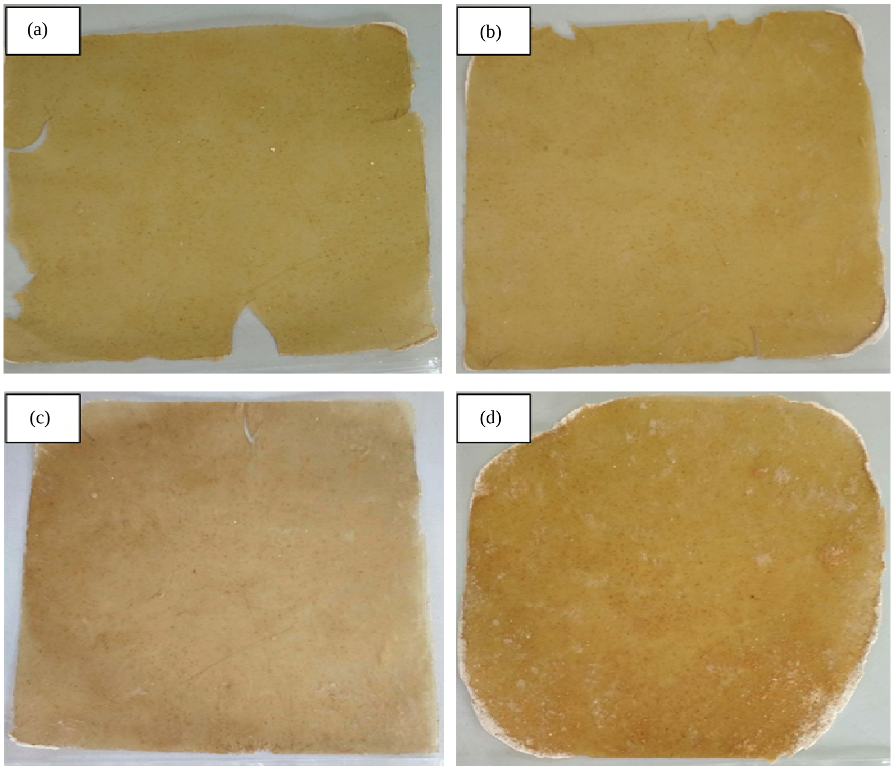

Fig. 1(a-d): Bioplastics prepared using different starch:glycerol ratio of (a) 2.5:1, (b) 2.75:1, (c) 3:1 and (d) 3.5:1

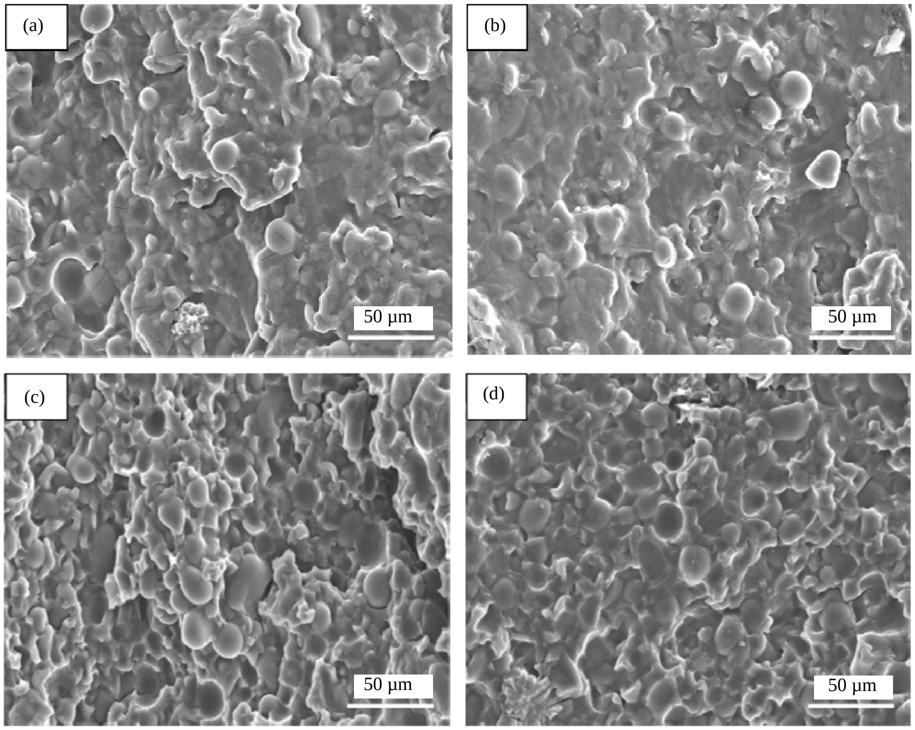

Fig. 2(a-d): SEM images of (a) 2.5:1, (b) 2.75:1, (c) 3:1 and (d) 3.5:1 samples

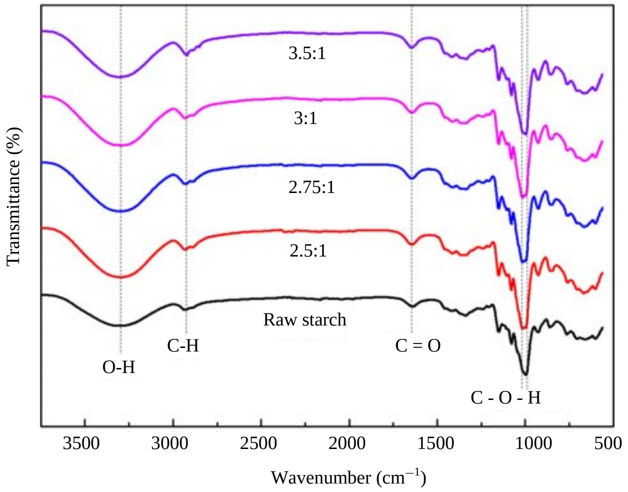

Fig. 3: FTIR spectra of bioplastics with different starch:glycerol ratio and raw sweet potato starch

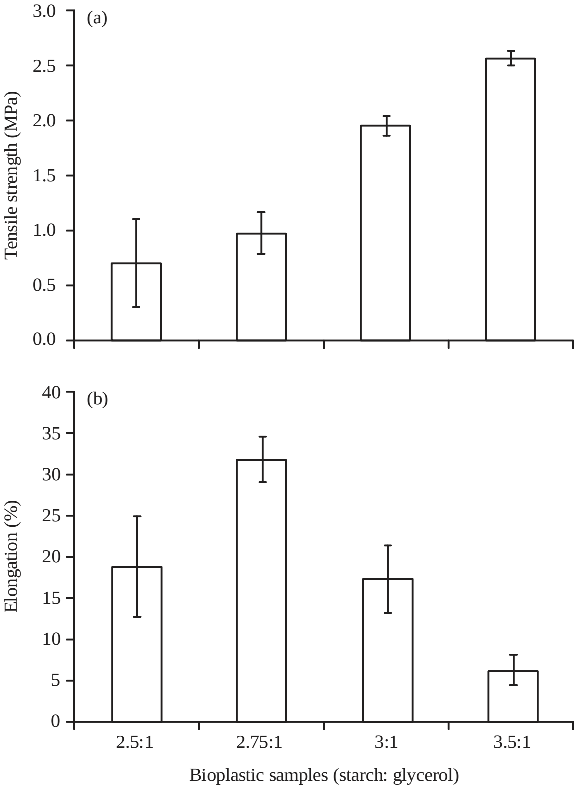

Fig. 4(a-b): (a) Tensile strength and (b) Elongation of bioplastics with different starch:glycerol ratio

Bioplastic with the highest starch:glycerol ratio exhibited the highest tensile strength (2.57 MPa) whereas the lowest starch:glycerol ratio exhibited the lowest tensile strength (0.71 MPa) which indicated that high concentration of starch increased the strength and stiffness. Conversely, the elongation of bioplastics tended to be decreased with the increase of starch:glycerol ratio from 2.75:1-3.5:1 while the ratio of 2.5:1 exhibited lower elongation than that of 2.75:1 (Fig. 4b).

Table 1: Physical properties of bioplastics at different starch:glycerol ratio

Samples	Density (g cm^{-3})	Contact angle (°)
2.5:1	1.39	42.4
2.75:1	1.59	43.9
3:1	1.66	48.1
3.5:1	1.66	50.1

The density of bioplastics was presented in Table 1. The density of bioplastics increased with the increase in starch:glycerol ratio. This result might be attributed to the increase of thickness of bioplastics which corresponded to the higher content of starch. Furthermore, at high starch:glycerol ratio, the contact angle of bioplastics was larger than a low concentration of starch (Table 1) which indicated that the addition of starch in the samples increased the contact angle and increased the hydrophobicity of bioplastics.

Biodegradability of bioplastics: Degradation of bioplastics was shown in Fig. 5. After 7 days of degradability test, the signs of degradation appeared. Bioplastics samples with 2.5:1 and 2.75:1 ratio emerged nearly one-quarter degradation (Fig. 5a, b). On the other hand, the 3:1 sample showed half degradation (Fig. 5c). Degradation was more discernible at the high starch:glycerol ratio (Fig. 5d). This result indicated that the enzymatic degradation occurred effectively in the samples with a high concentration of starch.

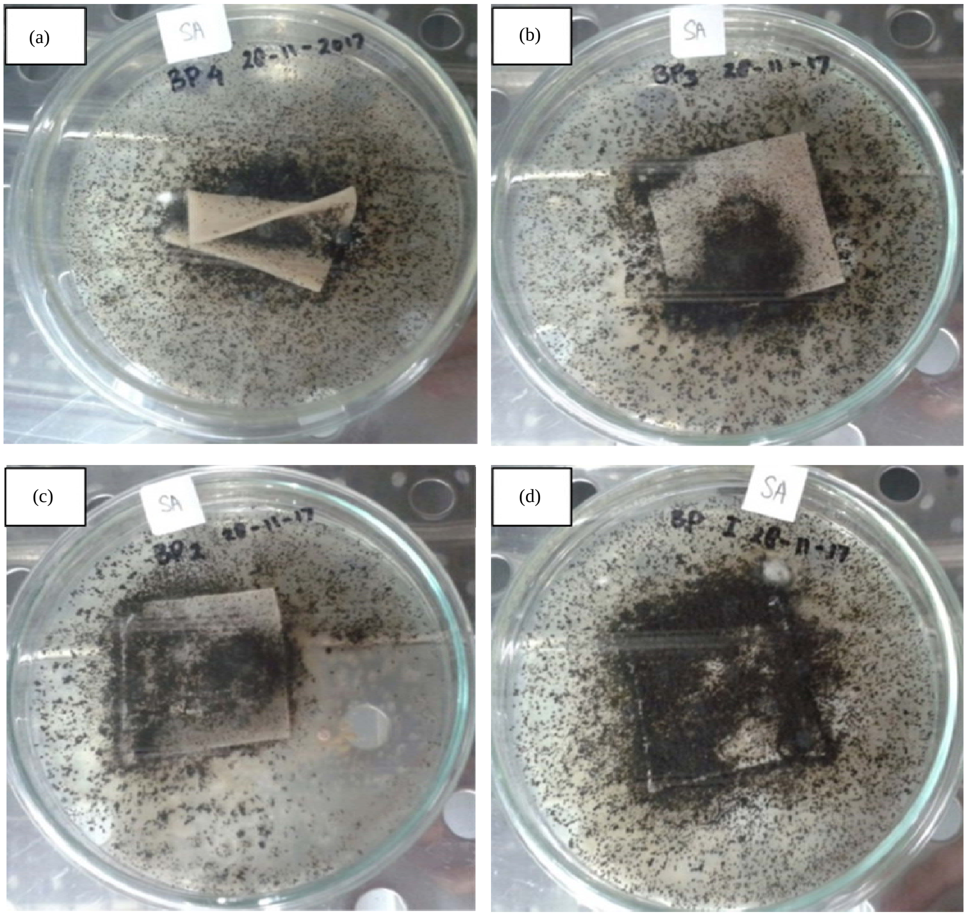

Fig. 5(a-d): Degradation of bioplastics (a) 2.5:1, (b) 2.75:1, (c) 3:1 and (d) 3.5:1 exposed for 7 days

DISCUSSION

The effects of different starch:glycerol ratio on characteristics of bioplastics were investigated in this study. In terms of bioplastics appearance and morphology, bioplastic with the lowest starch:glycerol ratio showed a homogeneous structure while the inhomogeneous appearances were obvious when starch:glycerol ratio increased. The incomplete gelatinization of starch molecules that was responsible for this appearance of inhomogeneous starch granules was influenced by several factors. The native structure of starch was difficult to be destroyed when the high concentration of starch was applied, therefore, the re-organization of starch through gelatinization in which starch granules swelled and disrupted by plasticizer molecules were impeded[14]. In addition, the heat treatment prior to gelatinization process also assisted in the formation of new bonds between starch molecules. Accordingly, incomplete gelatinization at high concentration of starch would be accompanied by molecular re-arrangements[15-18]. On the other hand, the structural differences of bioplastics with different starch:glycerol ratio caused the change of amorphous, semi-crystalline and crystalline arrangement of starch in the bioplastics[19]. The intensity of peaks at 997 cm^{-1} assigned for the C-O-H bending of the crystalline of starch[20,21] increased when the starch:glycerol ratio increased which indicated the increase of the crystalline part of starch after gelatinization. The change in intensity of specific peaks was attributed to the change in specific starch conformations such as crystallinity and long-range ordering, leading to polymer disordering and increasing number of conformations[20]. Furthermore, bioplastic with the highest starch:glycerol ratio (3.5:1) exhibited the highest tensile strength (2.57 MPa) indicated the highest strength and stiffness. These results revealed that the reinforcing effect occurred when the concentration of plasticizer decreased due to the decrease of the free volume of starch macromolecules and it would facilitate the macromolecular entanglement of starch which enhanced the bonding between polymer chains, thus increased the strength and stiffness[22]. The decrease of the free volume of starch macromolecules caused the reduction of extensibility and flexibility of bioplasticsas well and resulted in a decrease of elongation[23].

In line with mechanical properties, bioplastic with the highest starch:glycerol ratio (3.5:1) exhibited the highest density (1.66 g cm^{-3}), indicating the increase of bioplastics thickness. The insignificant role of glycerol in disrupting the intermolecular bonding between polymer chains would rearrange the polymer structure to a more compressed arrangement with decreased volume and thus increased the density of bioplastics[24]. Furthermore, the large contact angle in high starch:glycerol ratio could be attributed to the low concentration of plasticizer which has hygroscopicity (water-binding capacity) properties, hence, the wetting ability of water could be detained and hydrophobicity of bioplastics increased[23-26]. The most important characteristic of bioplastics based on the natural polymer in replacing the utilization of conventional synthetic plastics was its biodegradability. Bioplastics based on sweet potato starch prepared in this study exhibited the signs of degradation after 7 days of degradability test. Bioplastics with 3.5:1 ratio showed the highest growth of microbes. The complex polymer chains experienced cleavage enzymatically and crumbled subsequently into short chains of oligomers, dimers and monomers which could pass through the bacterial membranes and acted as carbon sources. The high number of carbon sources would increase the biodegradation rate due to the enhancement of hydrolysis of random chain scission which caused rapid molecular weight reduction. The smaller resulting molecules would be more susceptible to enzymatic attack, resulting in faster degradation[27,28]. Therefore, bioplastics with high starch:glycerol ratio exhibited faster degradation. In summary, the change in starch:glycerol ratio would alter the characteristics and properties of bioplastics which allows sweet potato starch-based bioplastics to be used for food packaging application.

CONCLUSION

The different starch:glycerol ratio affected the physical, mechanical and biodegradability properties of bioplastics. At the highest starch:glycerol ratio, the incomplete gelatinization of starch was observed, where the intermolecular interaction in bioplastics occurred through C-O-H, O-H, C-H aliphatic and C = O groups. The highest starch:glycerol ratio also exhibited the highest tensile strength, density and hydrophobicity with the lowest elongation at break. In addition, the highest sweet potato starch:glycerol ratio showed the fastest enzymatic degradation. These results indicated that sweet potato would be a good base material for bioplastic production and its application as food packaging.

SIGNIFICANCE STATEMENT

This study discovers the utilization of sweet potato sourced from Indonesia as a base material in bioplastic fabrication and its characteristics that can be beneficial for food packaging application. This study will help the researcher to disclose the critical areas of physical, mechanical and biodegradability properties of bioplastic based on sweet potato starch related to food packaging application which described in comprehensive explanation that many researchers were not able to present. Accordingly, a new erudition on the sweet potato potential as a base material for bioplastic production may be arrived at.

ACKNOWLEDGMENT

This study was supported by Ministry of Research, Technology and Higher Education of the Republic of Indonesia, Insinas Project fiscal year 2018 with grant number 38/P/RPL-LIPI/INSINAS-1/III/2018.

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