Emirates Journal of Food and Agriculture. 2023. 35(12): 1-9
doi: 10.9755/ejfa.2023.3203
http://www.ejfa.me/

RESEARCH ARTICLE

Physicochemical and sensory characteristics of bioplastics from phosphate butyrylated arenga starches

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ABSTRACT

The aim of this study was to obtain the optimum concentration of phosphate butyrylated arenga starches (PBAS) of bioplastic based on the physicochemical and sensory properties. The PBAS was obtained by dual modification of native arenga starch through butyrylation using 5% butyric anhydride and crosslinking using 6% a mixture of sodium trimetaphosphate (STMP) and sodium tripolyphosphate (STPP) at 99:1 (*w/w*). The concentration of the PBAS tested varied at 4.7; 5.0; 5.3; 5.7; 6.0; 6.3; 6.7; 7.0; and 7.3% (w/v) with three replications so that there were 27 experimental units used. The thickness, water holding capacity (WHC), oil holding capacity (OHC), swelling power, solubility, water vapor transmission rate (WVTR), water content, pH, biodegradation and properties of sensory were investigated. Results showed that the thickness, solubility, WVTR and water content of bioplastics increased with the increase in the concentrations of PBAS. On the other hand, WHC and OHC, swelling power, pH, and biodegradation decreased along with the increase in PBAS concentrations. Sensory color of bioplastics increased, while texture, aroma and overall acceptability decreased with the increase in PBAS concentrations. The concentration of PBAS at 7.0% was optimum for the physicochemical and sensory properties of bioplastics as indicated by low WVTR and water content as well as panelist preference on bioplastic texture.

Keywords: Phosphate butyrylated arenga starches; Physicochemical of bioplastic; Sensory of bioplastic.

INTRODUCTION

The increase in population along with economic growth has resulted in an increase in national waste production. One of the most significant and extreme environmental problems worldwide is the increase in plastic waste generated every day. Plastics, with their easily available and durable properties (Shafqat et al., 2021), have become a major commodity on a global scale, and are now present in almost all types of commercial products (Liang et al., 2021). The results of the plastic recycling survey revealed that only 12% of the total plastic consumption was recycled (Yang et al., 2021). The 2019 Coronavirus Diseases pandemic has had a major impact on the management of plastic waste in many countries due to the sudden surge in particular of medical waste which has caused a global waste management crisis. Improper management of plastic waste can cause various negative impacts on the environment, animals, and human health. Around 40% of plastic waste ends up in final disposal sites, 25% is burned, 16% is recycled and the remaining 19% is dumped into the environment (Khoo et al., 2021). The accumulation of plastic waste in the environment occurs because plastic is made of synthetic polymers which are difficult to decompose naturally (Pellis et al., 2021; Wu et al., 2021). To tackle the problem of plastics in environment, a number of strategies such as prohibiting single-use carrying bags and encouraging the use of bioplastics has recently gained significant interest (Ali et al., 2023; Moshood et al., 2022).

Bioplastics as an alternative to plastic substitutes have several special properties, namely environmental friendliness (Mehta et al., 2021), derived from renewable resources (Escobar and Britz, 2021) and non-toxic (Surya et al., 2021). Changes in the biodegradation process of bioplastics are caused by changes in the chemical structure of biopolymers during the modification process (Polman et al., 2021). Biopolymers produced from various natural

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Received: 30 June 2023; **Accepted:** 22 November 2023

resources such as starch, cellulose, chitosan, and various proteins from plants and animals are attractive alternative materials because they can improve the quality of packaging materials and extend their shelf life (Oyeoka et al., 2021). Starch, is one type of polysaccharide that can be synthesized by plants and is found mainly in fruits, root tubers, legumes, and cereals usually found in the range of 25-90%. Starch polymers are semi-crystalline which have about 1,000-2,000,000 glucose monomers linked by -1,4 glycosidic bonds (Ojogbo et al., 2020).

The main benefit of starch modification is that starch is as considered a natural resource and a very safe material. Thus, starches used in various industries, such as food, bioplastics, paper, and fabrics, can be expanded through the application of different modification techniques. Starch is often modified enzymatically which include hydrolysis and oxidation; chemically such as etherification, esterification, cross-linking, and starch grafting; and physically involves heat, extrusion, or hot moisture. Physical modification of starch is growing, especially for its use in food, whereas chemical modification has not been widely published (Obadi et al., 2021; Basu et al., 2021). One of starch sources which is widespread in Indonesia but has not been used optimally is arenga starch (Rahim et al., 2020). In this report, it is described an effort to obtain the optimum concentration of PBAS of bioplastic based on the physicochemical and sensory properties.

MATERIAL AND METHODS

Materials

The materials used in this study consisted of native arenga starch from the pith of sugar palm trees (*Arenga pinnata*). The 98% butyric anhydride was obtained from Sigma-Aldrich (Steinheim, Germany). Sodium trimetaphosphate (STMP), sodium tripolyphosphate (STPP), sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol, glycerol and acetic acid were purchased from Merck (Darmstadt, Germany). Distilled water and olive oil were obtained from the agro-industrial laboratory, Faculty of Agriculture, Tadulako University, Central Sulawesi, Indonesia. All the chemicals used for the bioplastic analysis were of analytical grade.

Research design

This experiment used a completely randomized design applied to the observation of physicochemical properties and a randomized block design which was applied to the sensory test. The concentrations of PBAS tested consisted of nine levels, i.e 4.7; 5.0; 5.3; 5.7; 6.0; 6.3; 6.7; 7.0; and 7.3% (w/v) which were repeated three times so that there were 27 experimental units used.

Preparation of PBAS

Butyrylation of native arenga starch was conducted according to the method of Phillips et al. (1999) while crosslinking followed the Koo et al. (2010) approach with a slight modification. Starch (100 g) was dispersed in 225 ml of distilled water and stirred for 1 hour at 25°C. Butyric anhydride of 5 % (starch basis, sb) was added drop-wise to the stirred suspension and the pH of the suspension was maintained at 8.0-8.5 by adding 3% NaOH. After 1 hour, the pH was increased to 10 with the addition of 3.0% NaOH to the solution and the reactions were allowed to proceed for 1 hour. The suspension was then adjusted to pH 4.5 with 0.5 N HCl. After sedimentation, the suspension was washed twice with distilled water and once with 95% ethanol for releasing acid, and then oven-dried at 40°C for 16 h, mashed, and sieved with 100 mesh. The modified arenga starches were called PBAS and used as bioplastic polymer materials.

Preparation of bioplastics

Bioplastic was prepared according to the method developed by Chung et al. (2010) with a slight modification. The PBAS (4.7; 5.0; 5.3; 5.7; 6.0; 6.3; 6.7; 7.0 and 7.3%) were added 150 mL of distilled water was added. The solutions were heated and stirred on a stirring-hot plate at temperature of 100°C after which 1 mL of acetic acid and 4 mL of glycerol were added. The heating lasted until the gel started forming. The solution was further stirred for 10 min and was poured into stainless steel strips and dried at room temperature for 4 to 6 days. The bioplastics produced were analyzed for physicochemical and sensory properties.

Parameters measured

Thickness determination

Bioplastic thickness was measured according to the method proposed by Turhan and Sabhaz (2004) through the use of calipers. The samples were placed between the jaws of the caliper and the thickness was measured at five different places and the average was calculated

Water and oil holding capacity determination

The water and oil holding capacity (WHC and OHC, respectively) were determined by using a method developed by Larrauri et al. (1996) with some modifications. Briefly, 25 mL of distilled water or olive oil were added to 250 mg of bioplastic samples, stirred, and the mixture was left at room temperature for 1 h. After centrifugation at 3,500×g for 30 min (GS 150 Centrifuge, Clements, Sydney, Australia), the residue was weighed and the holding capacity of water and oil was calculated as g of water or oil, respectively per g of dry bioplastic sample.

Swelling power and solubility

The method of Adebowale et al. (2009) was used to determine the swelling power and solubility of the starch.

Five hundred mg of starch sample was weighed into a centrifuge tube and it was reweighed (W1). The starch was then dispersed in 20 ml of water, heated in a water bath at 80°C for 30 min, cooled at room temperature and centrifuged at 3000 rpm for 15 min. The supernatant was decanted carefully into a new tube and the residue was weighed for swelling power determination. The weight of the centrifuge tube with the residue and the water retained was referred as W2. Aliquots (5 ml) of the supernatant were dried to a constant weight at 110°C and the residue obtained after drying represented the amount of starch solubilized in water. Solubility was calculated as g per 100 g of starch on dry weight basis.

Water vapor transmission rate determination

Water vapor transmission rate (WVTR) was determined by using the method proposed by Xu et al. (2004). This involved the production of a saturated salt solution in a chamber using a jar with a diameter of 12 cm and a height of 15 cm. The adjustment of the relative humidity (RH) in the chamber to 75% by the addition of 40% NaCl solution (w/v) at room temperature (Rahim et al., 2020). Furthermore, an acrylic cup with diameter of 5 cm and height of 1.8 cm was filled with 10 g silica gel, covered with bioplastic, and placed in the 75% RH chamber. The water vapor diffusing through the bioplastics and silica gel was added to the weight and the whole setup was weighed every hour for 8 h to determine the weight gain. The data was used to plot a graph of the time vs. weight, and the slope was recorded to calculate WVTR using the following equation: WVTR ($g/h/m^2$) = Change of bioplastics sample weight (g/h): Surface area of the bioplastic samples (m^2).

Water content

Water content was measured following the method applied by AOAC (2005). The portions of bioplastics (0.5 g) were dried in an oven at 105°C for 2 h.

pH

pH was determined by using pH meter (AOAC, 2005) where 1 g of bioplastic was diluted into distilled water until the volume up to 10 mL. pH of the samples was determined when the pH monitor showing a constant value.

Biodegradability analysis

The biodegradability of the bioplastic samples was observed according to the method developed by Ashok et al. (2018) with a slight modification. The samples were cut into 1 × 4 cm, weighed (W_1), and buried in the soil at a depth of 8 cm for a curing duration of 9 days after which they were reweighed (W_2). The weight loss due to the biodegradation process was determined with the following formula:

$$\text{Weight loss (\%)} = [(W_1 - W_2)/W_1] \times 100$$

Sensory evaluation

The sensory analysis was performed by the involvement of 15 students from the Faculty of Agriculture, Tadulako University, Central Sulawesi Indonesia as panelists. The bioplastic samples were cut into small pieces and the color, texture, aroma, and overall acceptability were tested using a 7-point hedonic scale, where 7 - highly very like, 6 – very like, 5 – like, 4 – neither like nor dislike, 3 – somewhat like, 2 – dislike, and 1 – very dislike. The panelists determined their degrees of likeness based on the hedonic scale.

Data analysis

All data were analysed by using one-way analysis of variance (ANOVA) with SPSS version 22, followed by Duncan's multiple range tests. The statistical significance was defined at $p \leq 0.05$.

RESULTS AND DISCUSSION

Thickness

Analysis of variance showed that various concentrations of PBAS significantly affected the thickness of the bioplastic. The photographs and average thickness of bioplastic is presented in Fig. 1a-1b respectively. The Fig. 1 showed that the thickness of the bioplastic increases with the increase in the concentration of PBAS. The average thickness of bioplastic of PBAS is in line with the results of Marichelvam et al. (2019) who stated that the thickness of bioplastic from the corn and rice flour mixture was 0.25 mm, and this means that it can be used as a biodegradable plastic bag. Different results were reported by Aversa et al. (2021) who found that the thickness of bioplastic bottles ranged from 0.65-0.90 mm. The thickness of the bioplastic is affected by the amounts of dissolved solids and the surface area of the container.

In a previous study by Susilawati et al. (2019), it was reported the range of bioplastic thickness of tapioca flour added with chitosan and fish bone gelatin ranged from 0.07 to 0.33 mm. Results of the study indicated that the higher the concentration of chitosan and fish bone gelatin added to tapioca flour, the thicker the bioplastic produced. According to Anggraini et al. (2017), the thickness of the bioplastic was influenced by the amounts of dissolved solids and the volume of the solution in the mold. Furthermore Santana et al. (2018) stated that the increase in the concentration of jackfruit seed starch and glycerol was closely related to higher concentrations of amylose and

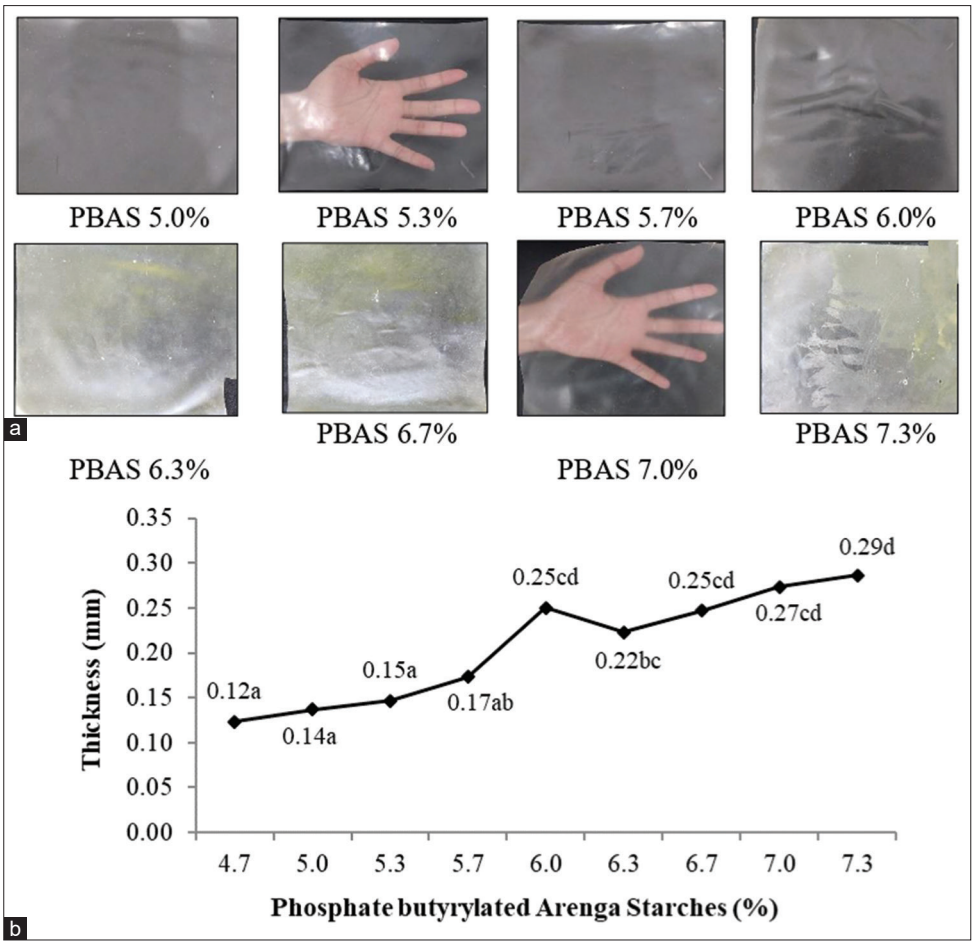

Fig 1. The photographs (a) and the thickness (b) of bioplastics on various concentration of PBAS.

amylopectin which led to higher soluble solids. As a result, the formation of a thick paste resulted in thicker bioplastics.

Water Holding Capacity and Oil Holding Capacity

Analysis of variance showed that the use of PBAS was insignificantly different in affecting the ability of bioplastics to hold water and oil. The water holding capacity (WHC) and oil holding capacity (OHC) decreased with the increase in PBAS concentrations. The average values of WHC and OHC are presented in Fig. 2. Fig. 2 showed that phosphorylation and butyrylation tend to improve the hydrophilic properties of PBAS. The decrease in WHC and OHC occurred due to the phosphate ester functional group in arenga starch was able to retain water and oil. WHC values ranged from 56.58-70.71%.

Azmin et al. (2020) reported that the water absorption of bioplastic from cellulose pod husk of cocoa and sugarcane fibers (with a ratio of 50:50) was 46.76%. Starch is hydrophilic and makes it more sensitive to water than cellulose. Hong et al. (2021) stated that bioplastics have several limitations, including low resistance to water and chemicals, low heat resistance, and brittleness. The strength

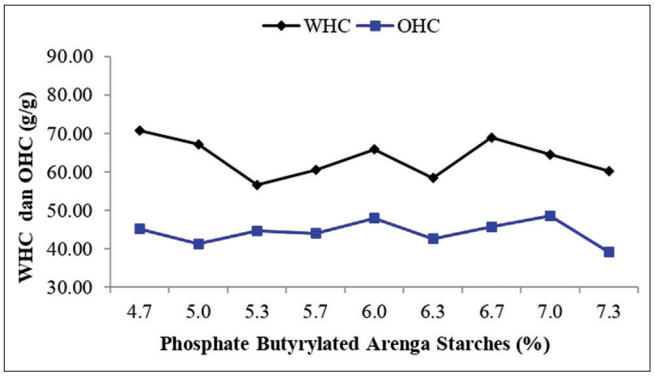

Fig 2. WHC and OHC of bioplastics on various concentration ofPBAS.

of bioplastics can be increased through several methods including physical and chemical linkages. Several studies have been carried out with physical methods including heat treatment, dehydrothermal, and ultrasonic, protein-based and methods of strengthening bioplastics through chemical crosslinking of bioplastics (Jiménez et al., 2020). Zhang et al. (2023) reported that water and chemical resistance were also observed for DAS-Bio-PI. These properties made the DAS-Bio-PI to be attractive for practical applications such as in petroleum-based plastics substitutes.

Swelling and solubility

Analysis of variance showed that the use of PBAS at various concentrations significantly affected the swelling power and solubility. The average values of swelling power and solubility of bioplastics are presented in Fig. 3. Fig. 3 showed that the swelling power of PBAS decreased with the increase in the concentration of PBAS while the solubility increased. These results in line with the report of Golachowski et al. (2020) who observed that esterification/crosslinking of starch with citric acid affected the swelling power and solubility of starch nanoparticles. The swelling power of starch particles crosslinked with citrate is lower than that of native starch. According to Hedayati and Niakousari (2018), esterification using 40% citrate in corn starch resulted in low swelling power and solubility compared to native starch. This parameter is strongly influenced by amylose content, starch chain length, granule structure and interactions between starch molecules.

On the other hand, the results of the physical modification by Zhong et al. (2021) showed that long-term microwave treatment of starch with high amylose content could reduce its swelling power and solubility in water. Saiful et al. (2019) stated that the ability of bioplastics to regulate oxygen exchange is largely determined by the basic structure and chemical properties of the polymers which make up the bioplastics. The ability to release gas particles is highly dependent on the porosity and swelling power. The permeability of bioplastics to oxygen is useful for estimating the shelf life of packaged products. According to Yang et al. (2022), the heterogeneous structure of bioplastics could reduce the cohesiveness of the starch matrix and lead to water permeation.

Water vapor transmission rate

Analysis of variance showed that the use of PBAS at various concentrations had a significant effect on the WVTR of bioplastics. The average value of the WVTR of bioplastics is presented in Fig. 4. The concentration of 7% PBAS had a low water vapor transmission rate of bioplastics (Fig. 4) and this was suitable for use in packaging. Results of this study were in line with the report of Rahim et al. (2020) who noted that the lowest WVTR of phosphate acetylated arenga starches (PAAS) bioplastic was recorded at 10 g PAAS. Xu et al. (2021) reported that the permeability of the biofilm to water vapor (WVP) decreased with the increase in the concentration of cellulose nanofiber (CNF). Pure amylose biofilms showed that WVP was 7-fold higher than CNF films.

The results of Campa et al. (2020) on agar-based biodegradable films with glycerol concentration of 0, 15 or 30% (by weight) as a plasticizer developed by combining hydroalcoholic garlic extract concentration 0; 0.5; 1; or 1.5 g/mL on the surface of the film showed a decrease in the rate of water vapor transmission with the increase in the concentrations of glycerol and garlic extract. This is in line with the results of Deshmukh et al. (2021) who noted that the increase in the concentration of chitosan composite and fat-free Chlorella biomass from 5 to 35% resulted in the decrease in more than 60% of water vapor permeability compared to pure chitosan.

Water content

Results of the analysis of variance showed that the PBAS concentrations had insignificant effect on bioplastic water content. The average value of bioplastic water content is presented in Fig. 5. Fig. 5 showed that the average water

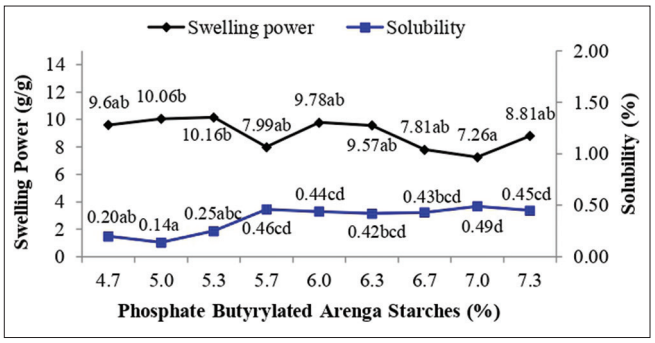

Fig 3. Swelling power and solubility of bioplastics on various concentration of PBAS.

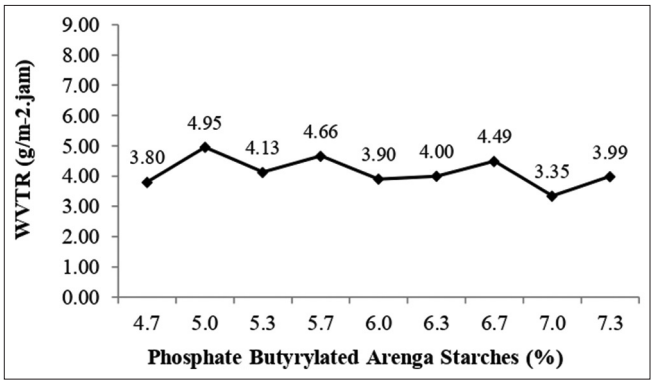

Fig 4. WVTR of bioplastics on various concentration of PBAS.

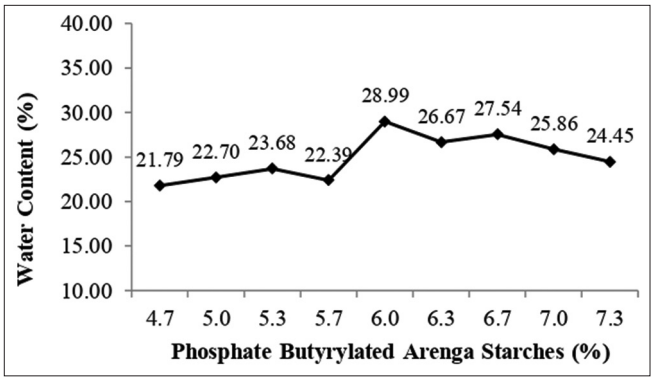

Fig 5. Water content of bioplastics on various concentration of PBAS.

content of bioplastics ranged from 21.79 to 28.99%. Results of this experiment were in line with prior study who reported that water content of native arenga starch bioplastics and phosphate acetylated arenga starches bioplastics were between 21.17 and 24.11% (Rahim et al., 2020).

The increase in the concentration of PBAS was also accompanied by the increase in the bioplastic water content. This is due to the hydrophilic native of the arenga starch, so the higher the concentration of PBAS, the higher the water content of the bioplastic. Lopes et al. (2020) reported that there was no difference in the water content of the biopolymers from the mixture of babassu coconut mesocarp, alginate and glycerol. Water content from various mixture of babassu coconut mesocarp, alginate and glycerol ranged from 36.4% to 46.5%.

pH

The analysis of variance showed that the use of PBAS at different concentrations had a significant effect on the pH of bioplastics. Results of the pH test are presented in Fig. 6. It was found that there was a decrease in the pH of bioplastics at concentrations of PBAS from 6.7-7.3%, namely 6.76-6.79 if compared to concentrations of 5.3-6.0% with the pH 7.13-7.31. According to Alotaibi and Bukhari (2021), one of the factors influencing the physicochemical properties of biofilms is pH. pH adjustment was able to affect the texture of the biofilm.

In their work on bilayer polylactic acid and bio-flex biofilms, Scaffaro et al. (2020) showed that bilayer and monolayer bioplastics had a high stability against degradation under acidic and neutral pH environments, but rapidly degraded under alkaline conditions. Al-Khalili et al. (2021) performed characterization of residues and precipitated from fat-free date palm fiber at different pH and found that cellulose dominated the precipitated matrix at alkaline pH whereas the matrix of fiber residue precipitated at pH 5 was predominantly hemicellulose and highly crystalline with interconnected porous particles.

Biodegradation

Analysis of variance showed that the use of crosslinked PBAS at different concentrations had a significant effect on the weight loss of bioplastics during degradation. The average value of biodegradation was presented in Fig. 7. The concentration of PBAS caused a decrease in the weight loss of bioplastics. Biodegradation is one of the parameters indicating for the environmentally friendly characteristics of bioplastic. According to Pudełko et al. (2021), biodegradation was influenced by various environmental conditions such as humidity, temperature, soil properties, and plant types. Amulya et al. (2021) stated that green

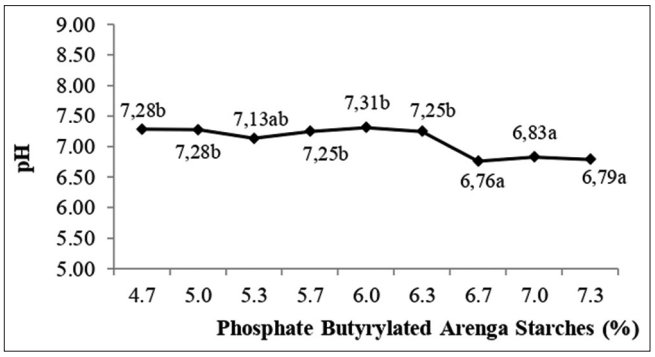

Fig 6. PH of bioplastics on various concentration of PBAS.

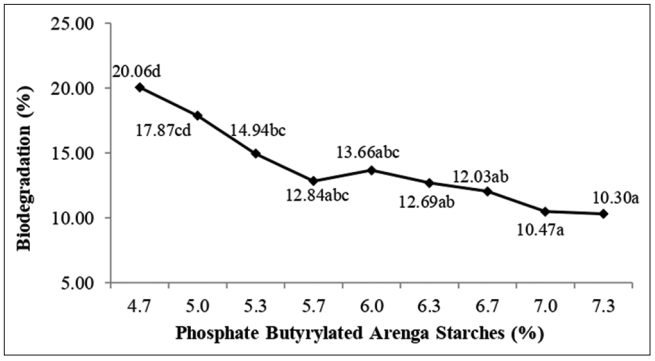

Fig 7. Weight loss of bioplastics on various concentration of PBAS.

economy principles were able to be implemented in the polymer industry by replacing conventional plastics with biodegradable ones. Polymers produced from renewable resources used light chemical processes and therefore suitable for the environment.

This is in conjunction with Jafarzadeh et al. (2020) who stated that the time required for bioplastics to expand completely depends on the source of the material and environmental conditions such as temperature, location, decomposition, and humidity. Compostable plastic decomposes quickly and turns into humus. The end result of decomposition has less harm to the environment than petroleum-based bioplastics. In addition, the decomposition of some biopolymers takes only a few weeks, while the degradation of others needs several months. De et al. (2021) reported that the average biodegradability of lignocellulosic biorefinery, coconut fiber, and nanocrystalline cellulose was 52.6% after 15 days of soil burial; and this value is proportional to the weight of bioplastics. According to Bracciale et al. (2024), biodegradation and disintegration of material were mainly influenced by the chemical composition of the material.

Sensory evaluation

The sensory evaluation, namely the panelists preference for color, texture, aroma and overall acceptability of bioplastic produced are presented in Table 1. Results from the sensory evaluation showed that the range of sensory scores given

Table 1: Sensories of bioplastics on various concentration of PBAS

PBAS (%)	Sensory score			
	Color	Texture	Aroma	Overall Acceptability
4.7	4.87^{ab}	5.40	4.33^{a}	5.40
5.0	4.80^{ab}	5.40	3.40^{a}	5.40
5.3	5.00^{ab}	5.00	3.40^{a}	5.00
5.7	5.07^{ab}	5.13	3.87^{a}	5.07
6.0	4.73^{a}	4.80	4.33^{ab}	4.80
6.3	5.07^{ab}	5.27	4.93^{b}	5.27
6.7	5.00^{ab}	5.13	5.33^{c}	5.13
7.0	5.07^{ab}	4.93	4.87^{b}	4.87
7.3	5.13^{b}	5.33	4.07^{ab}	5.13

Values in the same column with different superscript indicate a significant difference ($p \leq 0.05$).

by the panelists was 3.40 – 5.33 where the concentration of 6.7% PBAS was the most preferred concentration by the panelists. According to Azmin et al. (2020), color and texture of bioplastics were affected by cellulose fibers. At low cellulose concentration, bioplastics become dry and hard. Conversely, at a high cellulose concentration (50% and 75%) the bioplastics have a sweet aroma. Oluwasina et al. (2019) investigated the effect of oxidized starch on the physicochemical properties of tapioca starch bioplastics and showed that large particle sizes of the dispersed starch component fill the pores of the starch matrix and result in a reduction in light paths which causes a high opacity of the bioplastics.

It was also observed that the addition of oxidized starch led to an increase in the biofilm opacity. The increase in biofilm opacity was also able to be attributed to the formation of cross-linked starch molecules which result in strong bonds due to intermolecular binding i.e. covalent bonds and hydrogen bonds, leading to biofilm cohesiveness. Compactness causes a reduction in light transmission pathways through the biofilm leading to an increase in opacity. The storage of foodstuffs such as fruits, fishes, and vegetables with opaque biofilms are expected to have a longer shelf life than those stored with transparent biofilms. This is because the opaque biofilms are able to reduce the transmission of oxygen, water, light, and air from the stored foodstuffs and therefore the spoilage of the foodstuffs is minimized.

CONCLUSION

Concentration of PBAS affected the physicochemical and sensory characteristics of bioplastics. The optimum concentration of PBAS for producing bioplastics was 7.0% as indicated by low WVTR and water content as well as panelist preference on bioplastic texture. These bioplastics have the potency to be used as a plastic substitute packaging.

Acknowledgements

The authors are grateful to the Directorate of Research and Community Services, the Ministry of Education, Culture, Research and Technology of the Republic of Indonesia, which provided core-funding support through the Implementation Research Scheme with Contract Number: 061/E5/PG.02.00.PT/2022 dated March 16, 2022.

Conflict of interest

The authors have no conflict of interest

Contribution of authors

Credit authorship contribution statement: Syahraeni Kadir: Writing original draft, review and editing, methodology, investigation, data curation, conceptualization and supervision. Abdul Rahim: Writing original draft, review and editing, methodology, conceptualization and corresponding. Rostiati Rahmatu: Conceptualization, methodology, investigation, writing – review and editing. Gatot Siswo Hutomo: Writing – review and editing, methodology and conceptualization. Bahrudin: Writing – review and editing, data curation and conceptualization. Chitra Anggriani Salingkat: Writing, methodology and conceptualization. Iyan Andriani: Writing, methodology, investigation aand data curation. Siti Marwiah: Methodology and writing.

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