



Effect of Heating on Tensile Strength and Elongation at Break of Bioplastic from Taro Starch Filled Chitosan (*Colocasia esculenta*) with Glycerol Plasticizer

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Taro starch is a potential raw material for bioplastics by adding glycerol and chitosan. The casting method is used for making bioplastics with 30%w/v starch solution. Moreover, the variation added volume of glycerol (1%, 2% and 3% v/v), addition of chitosan (1% w/v, 2%w/v and 3%w/v) and pasting temperature was 75 °C. Taro starch with particle size 100 mesh has 93.55% starch, 17.9% amylose, 75.66% amylopectin. Analysis of plastic properties showed that taro starch has a gelatinization temperature of 74.52 °C with high peak viscosity 5953.5 cP. FTIR analysis show bands due to presence of O-H, C-H, C=H, C-O-H and C-O. The best of bioplastic in this study was 30% w/v on starch, 1% v/v glycerol and 2% w/v chitosan at 75 °C produces 42.86% water absorption, 8.297 MPa tensile strength and elongation at break was 45.846%.

Keywords: Starch, Taro, Bioplastic, Glycerol, Chitosan.

INTRODUCTION

Environmentally degradable polymers (EDPs) or bioplastics are a type of plastic, which constitutes renewable materials, such as vegetable oil and starch [1]. All green plants store their energy in the form of starch. Studies on starch-based bioplastics are performed using sago, potato starch, corn, durian seeds, and avocado [2-6]. Taro (*Colocasia esculenta*), a tropical tuber crop, is cultivated in various countries. This plant easily grows in Indonesia. Taro tuber requires 6-8 months for production with the characteristic yellow leaves. Taro contains 70-80% starch [7] and is used in bioplastics because it comprises high amounts of starch [8].

Starch, amylopectin and amylose are biopolymers and potential to be used as barriers in bioplastics [8]. Amylose is a water soluble, linear polymer with glucose bonding amylose a-D-(1→4), whereas amylopectin is a water insoluble, branched polymer with glucose bonding amylopectin a-D-(1→6) [9]. The unique properties of starch make its use as a raw material in bioplastic preparation complex because its thermal properties cause chemical interactions and many reactions. Water diffusion, gelatinization, granule enlargement, crystallization,

and decomposition occur during bioplastic manufacturing. Crystallization and gelatinization are important in bioplastic manufacturing because these processes convert starch into thermoplastics and bioplastics [10]. This study employed taro starch as the raw material to manufacture bioplastics from plasticizer glycerol and starch chitosan filler.

Chitosan (C₆H₉NO₃) was used as the filler to manufacture bioplastics using starch to enhance their mechanical properties because chitosan can cause hydrogen bonding between amylopectin and amylose in a starch solution [11]. The chemical structures of chitosan and starch are almost similar [7]. Chitosan also contains primary and secondary hydroxyl and amine groups; thus, it is ideal as a filler material to manufacture bioplastics using starch.

Glycerol, a plasticizer, is commonly used due to its efficacy. Bioplastics formed from pure polymers are fragile thus, plasticizers are used to enhance flexibility. Bioplastics containing plasticizers show more flexible properties than those without plasticizers [12]. This study determined the effects of heating temperature of starch solutions and chitosan on bioplastic properties, such as elongation at break, tensile strength, morphological properties and functional groups.

EXPERIMENTAL

Taro starch acquired from Tanjung Anom, Medan, Indonesia, was used as obtained. Chitosan flake of commercial grade (approximately 75% degree of deacetylation) having the average molecular weight of approximately 75 K Da and glycerol glacial were obtained E. Merck.

Starch isolation: Taro starch was isolated with 100 g of taro washed with cold water and dried. Taro was excoriated with a knife and was cut into a cuboid of approximately 1 cm × 1 cm × 1 cm. Then, it was placed in a blender along with 100 mL of water. This mixture was blended until smooth and then solution was filtered to separate the starch solution and pulp. For 24 h, the starch solution was sedimented to settle starch. Then, starch was separated by filtration, washed with distilled water until to acquire pure taro starch. Taro starch was dried, mashed and screened using a sieve with the mesh size of 100 [13-15].

Manufacture process of bioplastic: Bioplastics were manufactured from starch by using the casting method [4]. Chitosan (2 g) was weighed and diluted using 2% glacial acetic acid to 100 mL. Then, in a glass beaker containing 100 mL of distilled water, 30% w/v starch was diluted and the chitosan solution was mixed with the starch solution. Glacial acetic acid (2 mL) was added to the solution with constant stirring and then 1% glycerol was added until the solution became homogeneous. On a hot plate, the solution then was heated for gelatinization at 75 °C. The solution was curdled due to gelatinization. The solution was poured into a 25 cm × 25 cm × 1 mm mould and then dried for 24 h at room temperature [16].

Analysis of taro starch: For taro starch, starch content was characterized using the Luff-Schoorl method (SNI 01-2892-1992). Amylose and amylopectin content was also analyzed with the Luff-Schoorl method. Analyses of protein content, fat content, pasting properties, water content, ash content and functional groups were performed using the Kjeldahl method (SNI 01-2891-1992), the Soxhlet method (SNI 01-2891-1992), the rapid visco analyser (RVA), the oven method, the furnace method and the Fourier transform infrared (FTIR) technique, respectively.

Characteristic of bioplastic: The thickness of taro starch-based bioplastics was measured with an accuracy of 0.0001 mm.

Pasting properties analysis: Rapid visco analyzer (RVA) provides the systematic results for the properties of starch-containing materials. In RVA analysis, the determination of material viscosity involves parameters, such as hold, pasting peak viscosity, final viscosity, breakdown, peak time and set back [17].

Fourier transform infrared (FTIR): FTIR analyses were conducted at Research Laboratory, Faculty of Pharmacy, University of Sumatera Utara, on the instrument IR Prestige-21 Fourier Transform Infrared Spectrophotometer with Serial Number A21004602022 LP, Power 220-240 V 50/60 Hz, Shimadzu Corporation.

Mechanical properties: Elongation at break and tensile strength tests were conducted according to ASTM D 882 on a universal testing machine. The test was performed under the

conditions: temperature of 23 ± 2 °C and relative humidity of 50%. For this test, thickness was not >1 mm, with a length of 50 mm.

$$\text{Tensile strength value (MPa), } \sigma = \frac{\text{Tensile force (F)}}{\text{Surface area (A)}} \quad (1)$$

$$\text{Elongation at break (\%)} = \frac{\text{Stretch at break (mm)}}{\text{Initial long (mm)}} \times 100 \quad (2)$$

Scanning electron microscopy (SEM): SEM analysis was conducted using JEOL JSM-6390 LV at the Integrated Laboratory State University of Medan, Indonesia.

Water of absorption (WAB): The difference between the initial weight of and the weight after the bioplastic sample was soaked in water for approximately 10 s were estimated.

$$\text{WAB (\%)} = \frac{\text{Weight after being soaked} - \text{Initial weight}}{\text{Initial weight}} \times 100 \quad (3)$$

RESULTS AND DISCUSSION

Characterization of taro starch: White taro starch was prepared through isolation and Table-1 presents the taro starch characteristics.

TABLE-1
ANALYSIS OF TARO STARCH CHARACTERISTICS

Analysis	Indonesia industry standard (%)	Results by analysis methods (%)
Starch content	*min 75	93.55
Water content	*maks 14	6.5
Ash content	*maks 15	0.76
Amylose content	–	17.89
Amylopectin content	–	75.66
Protein content	–	1.02
Fat content	–	1.44

The obtained taro starch showed a considerably high starch, amylose and amylopectin of 93.55%, 17.89% and 75.66%, respectively. Compared with the results of Rahmawati *et al.* [18] with the same method present results were superior; they obtained the taro starch, amylose and amylopectin of 80%, 5.55% and 75.66%, respectively. The starch content relies on the starch isolation. Pudjiono [19] reported that raw material milling causes cell wall breaking to break starch granules. Cell wall can be broken by chopping or resprings. The condition is known as the reaping effect. From the comparison of amylopectin and amylose contents, taro starch can be used as the potential raw material to manufacture bioplastics. Due to the high content of amylopectin, taro starch shows high water absorption and thermal stability. It also had the following properties of amylopectin.

The obtained taro starch had water and ash contents of 6.5% and 0.76%, respectively. These amounts are lower than the results of Rahmawati *et al.* [18] by using the same method, contains water and ash contents of 9.4% and 2.25%, respectively. The water content was analyzed to determine the water amount available in taro starch. The water content influences the gelatinization temperature and shelf life of starch. The higher is the

water content of starch, the easier for starch to undergo weathering. The ash content determines the quality of bioplastics and starch.

The protein content was analyzed to determine proteins bound to water, which affect gelatinization viscosity. The protein content in taro starch and taro starch were 1.02% and 1.44%, respectively. The fat content affects rancidity and weathering during storage of bioplastics and starch. These results comply with the starch quality of Indonesian Industrial Standard, *i.e.* the starch comprises a maximum water content, maximum ash content, and minimum starch content of 14%, 15% and 75%, respectively.

Gelatinization profiles of taro starch: Starch pasting characteristics were analyzed using RVA, which was used to investigate taro starch gelatinization. Fig. 1 shows the starch pasting characteristics. Taro starch began to gelatinize at 74.52 °C (Table-2). The gelatinization temperature of taro starch was within temperature of 67.75-81.40 °C [20]. The peak viscosity of 5953.5 cP was attained at 90.43 °C. The time required to attain taro starch gelatinization temperature was 182 min. At heating temperature of > 95.05 °C, taro starch viscosity decreased to a breakdown viscosity of 3258 cP (Fig. 2). During cooling, the viscosity gradually decreased to the setback of 1184 cP.

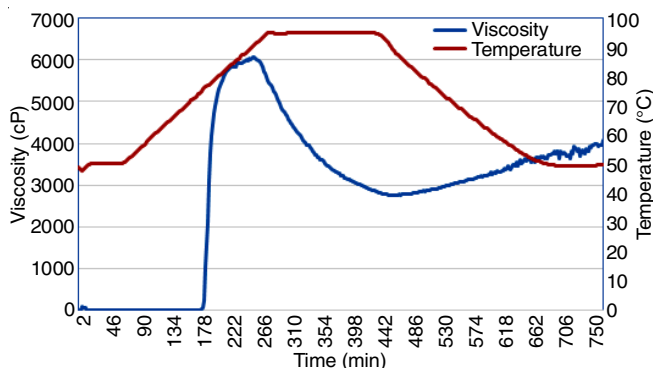


Fig. 1. Gelatinization profile of taro starch

TABLE-2 GELATINIZATION PROFILE OF TARO STARCH	
Gelatinization profile	Results
Pasting temperature (°C)	74.52
Peak viscosity (cP)	5953.5
Hold viscosity (cP)	2695.5
Final viscosity (cP)	3879.5
Breakdown (cP)	3258
Setback (cP)	1184

According to Chen [21], taro starch gelatinization exhibits a profile viscosity of type B (Table-2). Type B profile of starch gelatinization is attributed to the low peaks for paste viscosity. Moreover, its breakdown viscosity less sharp than that of type A. Starch type A can substantially expand; thus, it has considerably high viscosity. However, type A leads to a sharp decrease in viscosity when the starch is heated at the temperature higher than gelatinization temperature. Type B shows a lower expansion capacity than type A (Fig. 2). This result was supported by the

amylopectin and amylose contents obtained through spectrophotometry. The amylopectin was larger (75.66%) than the amylose (17.89%). Water soluble amylose showed a high expansion capacity after heating because amylose can form more hydrogen bonds than amylopectin. Amylopectin could not expand even after heating. Heating only led to destabilization of amylopectin. However, the amylopectin structure remained bound and did not form any hydrogen bond [22].

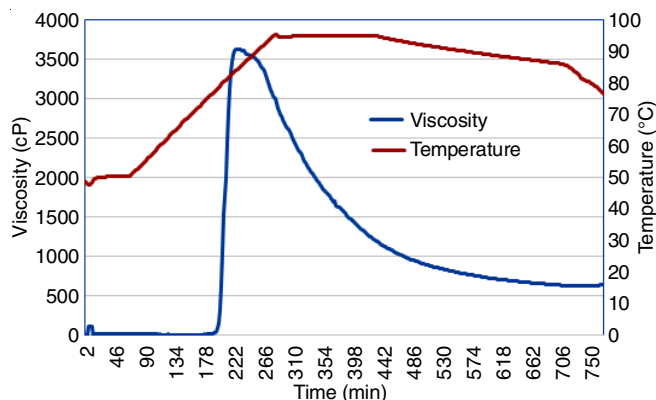


Fig. 2. Gelatinization profile bioplastic from taro starch with plasticizer glycerol and chitosan addition

Gelatinization profile of bioplastics from taro starch with addition of chitosan and plasticizer glycerol: The pasting characteristics of taro starch based bioplastics were acquired using 2 wt.% chitosan fillers and 1 v/v% glycerol plasticizer, which provided the profile of the gelatinization of bioplastics with the chitosan filler and glycerol plasticizer (Fig. 2).

Taro starch began to gelatinize at 76.67 °C (Table-3). The peak viscosity of 3630 cP was attained at 84.74 °C. The time required to attain the gelatinization temperature of taro starch was 192 min. At heating temperature of > 95.3 °C, for taro starch, viscosity decreased to the breakdown viscosity of 2998 cP (Fig. 2). During cooling, taro starch viscosity gradually decreased to the setback viscosity of 9 cP. From the pasting characteristics of taro starch based bioplastics, 2 wt.% chitosan fillers and with 1 v/v% glycerol plasticizer, chitosan solution addition influences the time needed to attain the gelatinization temperature. The time required to achieve the gelatinization temperature of taro starch was 182 min, whereas gelatinization temperature taro starch based bioplastics with 2 wt.% chitosan fillers and 1v/v% glycerol plasticizer was 192 min.

Glycerol as plasticizer and water as solvent led to an increase and a decrease in gelatinization temperature and peak

TABLE-3 GELATINIZATION PROFILE BIOPLASTIC FROM TARO STARCH WITH PLASTICIZER GLYCEROL AND CHITOSAN ADDITION	
Gelatinization profile	Results
Pasting temperature (°C)	76.67
Peak viscosity (cP)	3630
Hold viscosity (cP)	632
Final viscosity (cP)	641
Breakdown (cP)	2998
Setback (cP)	9

viscosity, respectively. This is because glycerol and water have polar properties and a high boiling point and low viscosity. Taro starch exhibited a viscosity peak and gelatinization temperature of 5953.5 cP and 74.52 °C, respectively, whereas taro starch based bioplastics with chitosan fillers and glycerol plasticizer showed those of 3630 cP and 76.67 °C, respectively (Table-2). Moreover, glacial acetic acid was added as a starch catalyst while the chitosan affected the bioplastic viscosity. Moreover, addition of glacial acetic acid led to a decrease in the pH solution and thus in starch viscosity.

Effects of heating temperature and chitosan addition on the tensile strength of taro starch based bioplastics with glycerol plasticizers: Fig. 3a shows the impact of the heating temperature of starch solution and chitosan on the tensile strength of taro starch based bioplastics with glycerol plasticizer of 1, 2 and 3 wt.%. The tensile strength of 8.297 MPa was obtained with 1 v/v% glycerol plasticizers, different addition amounts of 2 wt.% chitosan and heating temperature of 75 °C.

Fig. 3b shows the effects of heating temperature and chitosan on the tensile strength of taro starch based bioplastic with glycerol plasticizer 2 v/v%. With an increase in chitosan addition amount, the tensile strength of bioplastics increased. The highest tensile strength of 4.769 MPa was obtained with 2 wt.% chitosan, 2 v/v% glycerol plasticizers and heating temperature of 75 °C.

Fig. 3c shows the effects of heating temperature and chitosan on the tensile strength of taro starch based bioplastics 3 v/v% with glycerol plasticizers. The tensile strength of 3.924 MPa was obtained with 2 wt.% chitosan, 3 v/v% glycerol plasticizer, and heating temperature of 75 °C.

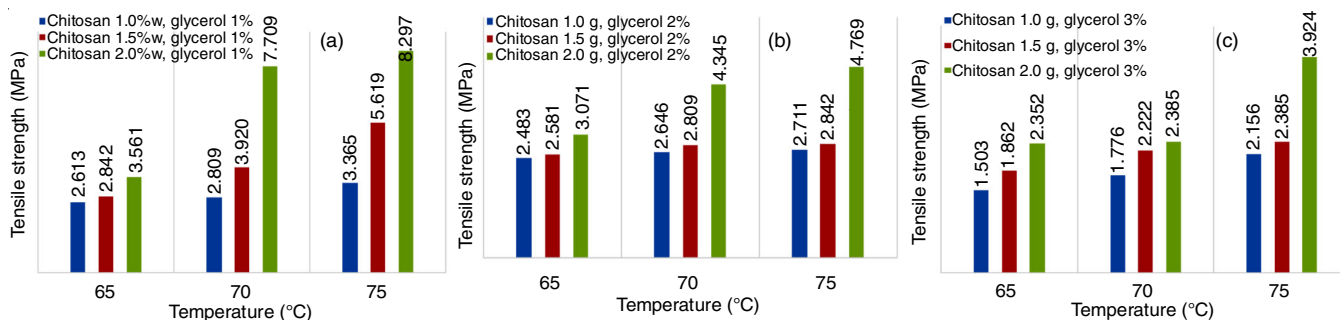


Fig. 3. Effect of chitosan addition and temperature heating tensile strength of bioplastics from taro starch with glycerol plasticizer (a) 1%v, 2%v and (c) 3%v

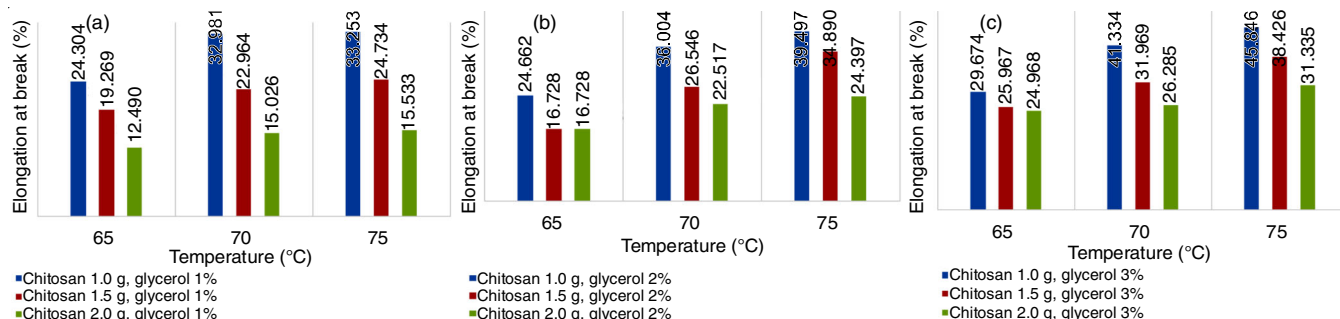


Fig. 4. Effect of chitosan addition and temperature of heating for elongation at break of bioplastics taro starch with glycerol plasticizer (a) 1%v, (b) 2%v and (c) 3%v

Fig. 3a-c show the tensile strength of 8.297 MPa of taro starch based bioplastics with glycerol plasticizer and chitosan is optimum at 75 °C with 1 v/v% glycerol and 2 wt.% chitosan. With an increase in the chitosan amount, the tensile strength of bioplastics increased. The pasting temperature for taro starch was 76.67 °C, which is close to bioplastic gelatinization temperature for starch solution heating; thus, tensile strength is high (Table-3). The higher chitosan concentration provides more hydrogen bonds in bioplastics; hence, the chemical bonds become stronger and are more difficult to break [23]. The glycerol plasticizer exhibited low tensile strength because plasticizer molecules disrupt starch cohesiveness, thus reducing the hydrogen bonding and increasing polymer mobility [24].

Effect of heating temperature and chitosan addition on elongation at break of taro starch based bioplastics with glycerol plasticizers: The effects of heating temperature and chitosan on elongation at break properties of taro starch based bioplastics with 1 wt.% glycerol plasticizers were also investigated. Fig. 4a shows the effects of heating temperature and chitosan on elongation at break properties of taro starch based bioplastic with 1 v/v% glycerol plasticizers. The highest elongation at break of 33.253% was obtained with 1 wt.% chitosan, 1 v/v% glycerol plasticizers and heating temperature of 75 °C.

Fig. 4b shows the effects of heating temperature and chitosan on the elongation at break properties of taro starch based bioplastic with 2 v/v% glycerol plasticizers. The highest elongation at break of 39.497% was obtained with 1 wt.% chitosan, 2 v/v% glycerol plasticizers and heating temperature of 75 °C.

Fig. 4c shows the effects of heating temperature and chitosan on the elongation at break properties of starch-based

bioplastic with 3 v/v% glycerol plasticizers. The highest elongation at break of 45.846% was obtained with 1 wt.% chitosan, 3 v/v% glycerol plasticizers at 75 °C.

Fig. 4a-c show that the optimum elongation at break of 45.846% of taro starch based bioplastics with glycerol plasticizers and chitosan was obtained at 75 °C with 1 wt.% chitosan and 3 v/v% glycerol. With an increase in the chitosan amount, the elongation at break of bioplastics decreased. The pasting temperature of taro starch based bioplastics was 76.67 °C (Table-3). When the gelatinization temperature of bioplastics is closer during starch solution heating, then the elongation at break is higher. The higher chitosan concentration presents more hydrogen bonds in bioplastics; thus, the chemical bonds become stronger and more difficult to break. The glycerol plasticizer showed a high elongation at break because plasticizer molecules disrupt starch cohesiveness, thus reducing hydrogen bonding and increasing polymer mobility [23].

Water absorption of taro starch based bioplastic: Fig. 5 shows the relation of chitosan addition with water absorption of taro starch based bioplastics with 1v/v% glycerol plasticizers.

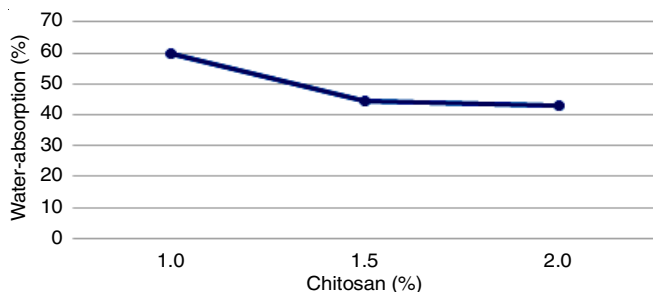


Fig. 5. Water absorption of taro starch based bioplastics

Fig. 5 shows the effect of chitosan addition on water absorption of taro starch based bioplastics having 1v/v% glycerol plasticizers. With an increase in the chitosan amount in taro starch based bioplastics with 1 v/v% glycerol plasticizers, the water resistance of bioplastics increased. The optimum water resistance of 42.86% of taro starch based bioplastics was obtained with 1 v/v% glycerol plasticizers and 2 wt.% chitosan. This result agrees with the findings of Kumar [24]. It is reported that the biopolymer chitosan provides high water resistance for bioplastic materials because chitosan is readily biodegradable and has hydrophobic properties. Conventional plastic (polypropylene) presents a water resistance of 0.01% [25]. Taro starch based bioplastics with 1 v/v% glycerol plasticizers and 2 wt.% chitosan filler provides a higher water resistance than polypropylene. Thus, taro starch based bioplastics with 1 v/v% glycerol plasticizers and 2 wt.% chitosan filler can absorb considerable water.

Morphology analysis of bioplastics from taro starch with chitosan and plasticizer glycerol on fracture: Fig. 6 illustrates the morphology of taro starch based bioplastics with 2 v/v% glycerol plasticizers and 2 wt.% chitosan for fracture regions obtained at 75 °C at 1000x magnification.

Fig. 6 shows the morphology of fracture areas of taro starch based bioplastics obtained at 75 °C, which indicate few empty fractions (voids) exist. In taro starch bioplastics with

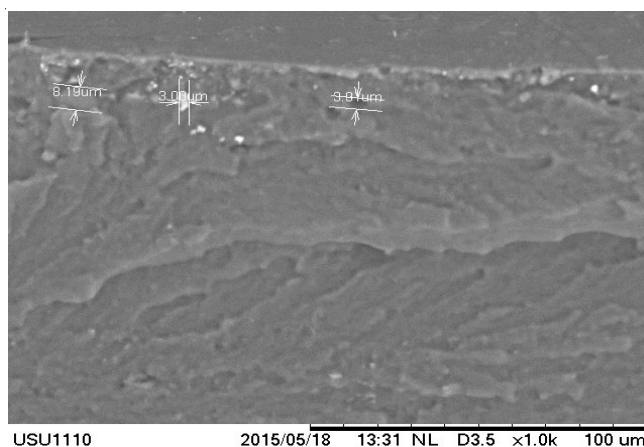


Fig. 6. Morphology image of bioplastics from taro starch with chitosan and plasticizer glycerol

glycerol plasticizers and chitosan, void existed because bioplastics weakened. When bioplastics were provided loads, their strength moved to void areas; thus, the tensile strength of taro starch based bioplastics decreased.

FTIR studies: Fig. 7 shows the FTIR of chitosan, taro starch and taro starch-based bioplastic with glycerol plasticizers and chitosan filler. The chitosan constitutes amino and hydroxyl groups [26]. Its chemical structure is similar to that of lignin and cellulose [27], which was confirmed by the FTIR spectra. From the FTIR results, the chitosan group comprised C-H, N-H, O-H, C-O-H, C=O, C-O-C and C-O.

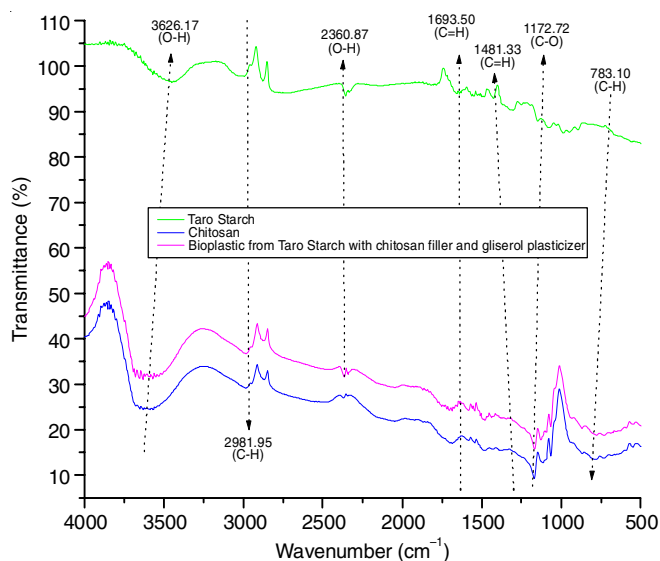


Fig. 7. FTIR spectra of taro starch

The peaks appearing at 3626.17 cm⁻¹ represents the strain O-H group of alcohols and phenols, while at 2981.95 cm⁻¹ denotes the C-H of strain alkanes. A peak at 2360.87 cm⁻¹ indicates the strain O-H group of carboxylic acids, while at 1701.22 cm⁻¹ revealed the C-H of strain CH₃. A peak at 1481.33 cm⁻¹ represents the C-O-H group of strain CH₃, whereas 1172.72 cm⁻¹ is attributed to the C-O group of strain esters. Finally, at 783.10 cm⁻¹ corresponds to the C-H of aromatic benzene rings.

Taro starch constitutes amylose, amylopectin and reducing glucose (C₆H₁₀O₅)_n [28] along with the glycerol (C₃H₈O₃) plasticizers and chitosan comprising hydroxyl and amino groups [26], which has the chemical structure similar to lignin and cellulose [28]. The taro starch based bioplastics with glycerol plasticizers and chitosan comprised C-H, O-H, C-O-H, C=H, and C-O groups. The functional groups for taro starch based bioplastics without chitosan fillers and glycerol plasticizer and for those with glycerol plasticizers and without chitosan fillers are different. The O-H group of the aforementioned two taro starch based bioplastics interacts with glycerol plasticizers and water. However, this group reappears in the taro starch based bioplastics with glycerol plasticizers and chitosan. Chitosan filler addition causes the tensile strength of bioplastics to be maintained by converting the O-H groups of starch, which interact with plasticizers and solvents, into the O-H groups of chitosan.

Conclusion

The gelatinization temperature and peak viscosity of taro starch were 74.52 °C and 5953.5 cP, respectively, whereas those of taro starch-based bioplastics with glycerol plasticizers and chitosan fillers were 76.67 °C and 3630 cP, respectively. Chitosan filler addition affects the tensile strength of bioplastics. The increase in chitosan filler amounts leads to an increase in tensile strength and a decrease in elongation at the break. The optimum tensile strength of 8.297 MPa of taro starch based bioplastics with filler chitosan and glycerol plasticizers was obtained at 75 °C with 1 v/v% glycerol plasticizer and 2 wt.% chitosan fillers. Glycerol plasticizer addition influences the elongation at break. With the increase in glycerol plasticizer amounts, the elongation at break increases and tensile strength decreases. The optimum elongation at break of 45.846% of taro starch based bioplastics with chitosan fillers and glycerol plasticizers was obtained at 75 °C, with 3 v/v% glycerol plasticizers and 1 wt.% chitosan fillers. During starch solution heating, at temperature closer to the gelatinization temperature of bioplastics, the tensile strength and elongation at break values are high. The FTIR spectra of taro starch, chitosan and fabricated bioplastics were different due to a few functional groups interact, thus forming novel bonds. From the SEM results, amylopectin remains completely insoluble in bioplastics with acetic acid 2 wt.%. The voids influence elongation at break, tensile strength and water resistance of bioplastics.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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