Preparation and Characterization of Biodegradable Starch Based Bioplastic Film from Cassava

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Abstract

This paper focused on the green-production of bioplastic film and the characterization of starch based bioplastics. The starch powders were extracted from fresh cassava by the cold extraction method. The yield percent of the extracted cassava starch is 14.32 %. The physicochemical properties of prepared cassava starch such as pH, moisture content, ash content and bulk density were detected. The bioplastic film was prepared by the casting method with varying amounts of starch powder, glycerol and vinegar. The physicochemical properties of prepared bioplastic films were evaluated by pH, moisture content, viscosity and specific gravity. In addition, the physicomechanical properties such as thickness, tensile strength, elongation at break percent, and tear strength of prepared bioplastic films were also evaluated. The ratios of starch, glycerol, and vinegar (3g : 0.5mL : 1mL) are the most suitable for the physicomechanical properties for the preparation of bioplastic films. The transparency, water absorption capacity, shelf life, and biodegradable ability of prepared bioplastic films were also investigated. The transparency of prepared bioplastic films was measured by using a UV-Visible spectrophotometer at the wavelength of 550 nm. The prepared bioplastic films were characterised by Fourier transform infrared (FT IR) spectroscopy. The FT IR results showed the presence of O-H, C-H, C-O-C stretching and C-O-C ring vibration in carbohydrate. The decomposition nature of prepared biodegradable film was investigated by TG DTA. The water absorption capacity of prepared bioplastic films was determined by soaking them in water for 24 hours. The biodegradable ability of selected bioplastic films was examined by soil burial test. The bioplastic film was found to be almost degraded after 12 days. The prepared bioplastic films were synthesised using natural and environmentally safe raw materials and showed biodegradation, thus proving to be a good alternative to conventional plastics.

Keywords: cassava starch, casting method, bioplastic, biodegradation, shelf life

Introduction

Environmental pollution due to plastic waste taking too long to decompose has become a global problem. There have been numerous solutions proposed, one of which is the use of bioplastics. The use cassava starch as the main ingredient in the manufacture of bioplastics shows great potential has a diverse range of starch-producing plants. Plastic waste poses a risk to the human health and the environment. Waste plastic has become an environmental problem around the world because it takes a long time to be degraded and take 50 years to be degraded it in nature. Bioplastics can help to reduce the problem of plastic waste because of easy to make it, abundant and affordable raw materials. It is categorized as environmentally friendly and able to be degraded by the activity of microorganisms by 10 to 20 times faster than conventional plastic resulting in carbon dioxide, methane, water, biomass, humus and other natural substances from the sources of compounds in plants such as starch, cellulose and lignin. Bioplastics are fabricated from biopolymers obtained from biomass, such as starch, cellulose and proteins. Biodegradable plastic will decompose in natural aerobic and anaerobic environments. It is a biodegradable plastic in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi and algae (Park *et al*., 1994). Starch is a naturally occurring polymer which is inexpensive and abundantly available. It is main storage of carbohydrate in most plants and is also widely consumed polysaccharide in the human diet. Starch is a hydrophilic polymer and white, granules, organic chemical that is produced by all green plants as a means of storing energy. It is a soft, white tasteless powder that is soluble in water, alcohol or other solvents. The molecular structure of starch is

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$(C_6H_{10}O_5)_n$. The starch granule is a heterogeneous material, chemically, is contains both linear amylose and branched amylopectin structure, physically, it has both amorphous and crystalline regions. The ratio of amylose to amylopectin in starches varies as a function of the source, age and etc. There are many types of starches and the most common types are corn, cassava, potato, wheat and rice starch (Michele Avella *et al*., 2005).

Cassava is the third major source of carbohydrates in the word and has a wide range of uses and serves as food security for millions of living in the developing world. The starch plays important role in bioplastic forming. Cassava is extensively cultivated as an annual crop in tropical and subtropical regions for its edible starchy tuberous root which is a major source of carbohydrates. Cassava starch for example is one of the most commonly use biopolymers as a food packaging material because it is non-toxic, biodegradable, biocompatible, low cost, renewable and abundantly available in nature (Ezeoha *et al*., 2013). Plasticizers as polymer additives serve to decrease the intermolecular forces between the polymer chains, resulting in a softened and flexible polymeric matrix. Glycerol is plasticizers that function to reduce internal hydrogen bonds so that they will increase intermolecular distances (Mulyono *et al*., 2015).

This study is centered on the use of cassava for the production of bioplastic films. The prepared bioplastic films were characterized by physicochemical and physicomechanical properties. Moreover, the behaviors of prepared bioplastic films were also investigated by modern techniques such as FT IR and TG DTA. The biodegradable ability of prepared bioplastic film was investigated by soil burial method.

Materials and Methods

Materials

In this research, cassava was used as a starch source for preparation of bioplastic film. They were collected from Thitseik market in Dagon Seikkan Township, Yangon Region and Myanmar.

Extraction of starch from cassava

Firstly, 1000g of cassava root was cut into pieces and blended with distilled water at high speed for 2 min using a blender. The cassava slurry was collected through a clean cloth. The filtrate was left to settle in the beaker for 3 hours. After that the resulting supernatant liquid was decanted, leaving behind white starch which had settled at the bottom for 3 hours. The residue was washed with distilled water repeated two times and dried in a vacuum oven at 105 ºC. After drying, they were ground to a uniform powder using a motor and pestle in the laboratory. Then cassava starch powder was weighed and calculated the yield percent. The physicochemical properties of cassava starch such as pH, moisture content, ash content and bulk density were determined.

Preparation of bioplastic film from cassava starch

Bioplastic film was prepared with starch, glycerol and vinegar and made up of 100 mL of distilled water by casting method. The mixed solution was heated by constantly stirring using magnetic stirrer until the mixture gelatinized at approximately 70 ºC. The resultant starch paste was poured and cast onto a melamine plate and then dried in the oven at 60 ºC to obtain film. The effect of amount of starch powder on the physicomechanical properties of prepared plastic films were investigated as the same procedure mentioned above except variable of starch in the range of (1, 2 and 3 g) and denoted as CSS 1 to CSS 3. The effect amount of glycerol of prepared bioplastic films (0.5, 1 and 1.5 mL) were carried out while the other variables were fixed using the same method mentioned above. The obtained bioplastic films were noted as CSS 3, CSSG 1 and CSSG 2. The effect amount of vinegar (0.5, 1 and 1.5 mL) for the bioplastic films were used while the other variables were fixed using the same method mentioned above and denoted as CSSV 1, CSS 3 and CSSV 2.

Characterization Physicomechanical properties of bioplastic film

The film thickness of bioplastic film was determined by NSK micrometer, tensile strength, elongation at break % and tear strength were determined with Tensile Testing Machine.

Average Transparency of the bioplastic films

Transparency is the ability of a material to transmit light. The transparency of bioplastics was using a spectrometer (λ = 550 nm). Transparency is measured using the Al-Hassan and Norziah method (Hassan *et al*., 2012) and calculated using the formula:

$$T = A_{550} / x, \; A_{550} = \text{Absorbance } (\lambda) \text{ 550 nm}, \; x = \text{Bioplastic thickness (mm)}$$

Determination of water absorption capacity (%)

The water absorption capacity (%) test identified the ability of bioplastics to absorb water as determined by standard ASTM D 570. Bioplastics were cut into 1″ × 1″ size which had been previously dried for 24 hours in an oven at 50 ºC, cooled in a desiccator and weight. The water absorption capacity (%) data of bioplastics was obtained by soaking them in water for 24 hours. After that, the bioplastics were dried with a cloth and immediately weighed (Nanang *et al*., 2017).

$$\text{Water Absorption Capacity (\%)} = \frac{(\text{Post} - \text{Brake weigh}) - (\text{Initial weigh})}{\text{Initial weigh}} \times 100$$

FT IR analysis of biodegradable plastic film

FT IR analysis supports evidence for the presence of functional groups in bioplastic film. The functional groups of prepared selected bioplastic films were characterized by Fourier Transform Infrared Spectroscopy (FT IR). Spectra were collected in the wavenumber range of 500−4000 cm^{-1}.

Thermo-gravimetric differential thermal analysis (TG DTA)

The thermal stability of the tested bioplastic film was evaluated by a simultaneous TG DTA (DTG-60) operation under nitrogen atmosphere. The measurement was carried out at a heating range of 20.0 kJ min^{-1} and scanning from 40 ºC to 600 ºC.

Determination of shelf life

The bioplastic films were cut into 2″ × 2″ size. And then these films were placed in a plastic bag at room temperature and watched day by day. Finally, the selected bioplastic film was analyzed through visual observation with a camera (Susilawati *et al*., 2019).

Determination of Biodegradability

Biodegradable behavior of bioplastic was determined using a soil burial degradation test, i.e. bioplastic was buried in the soil, so that it would be degraded. Bioplastic was cut into 1″ × 1″ size. Then, they were buried into the ground at 5 cm depth; the burial duration interval of two days (Park *et al*., 1994).

Results and Discussion

Physicochemical Properties of Cassava Starch

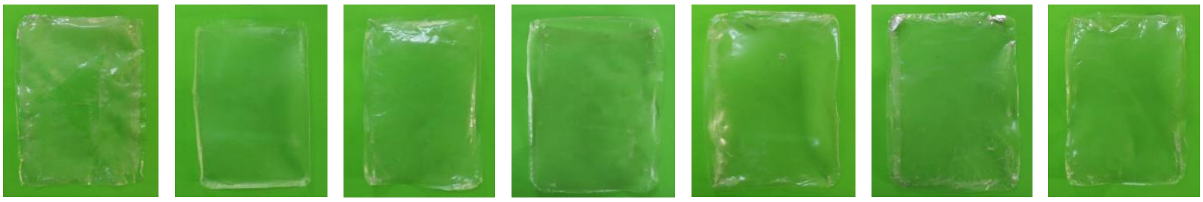
The physiochemical characteristics of cassava starch are shown in Table (1). According to the results, the value of pH is 6.8, ash content (%) is 1.65 %, moisture content (%) is 7.80, bulk density is 0.75 cm^{-3} and yield percent is 14.32 %.

Table 1. Physicochemical Properties of Cassava Starch

Sample	pH	Ash content (%)	Moisture content (%)	Bulk Density (gcm^{-3})	Yield (%)
Cassava starch	6.8	1.65	7.80	0.75	14.32

Starch Based Cassava Bioplastic Film

The purpose of bioplastic production is an alternative process for synthetic plastic. The starch is a natural biopolymer, and cassava is employed to provide the bioplastic film by using a glycerol plasticizer. The prepared starch-based cassava bioplastic films were presented in Figure 1. The prepared bioplastics films in the present work were odorless, transparent, and smooth. The glycerol may increase the flexibility of bioplastic film.



CSS 1 CSS 2 CSS 3 CSSG 1 CSSG 2 CSSV 1 CSSV 2

Figure 1. Photographs of prepared cassava starch-based biodegradable plastics

Physicochemical Properties of Bioplastic film

Preparation of bioplastic films was carried out by various ratios of starch, glycerol and vinegar. The effect amount of starch, amount of glycerol and amount of vinegar on the physicochemical properties of prepared bioplastic films are shown in Table 2. The prepared bioplastic films were shown in Figure 1.

Table 2. Physicochemical Properties of Prepared Bioplastics from Cassava Starch

Sample	pH	Moisture content (%)	Viscosity (cP)	Specific gravity
CSS 1	4.0	19.05	53.5	1.0134
CSS 2	4.1	16.67	80.3	1.0162
CSS 3	4.2	14.28	114.8	1.0194
CSSG 1	4.0	18.18	112.8	1.0176
CSSG 2	4.4	19.09	124.6	1.0199
CSSV 1	4.4	15.38	125.6	1.0174
CSSV 2	3.8	18.05	110.8	1.0194

CSS 1 = Starch (g), Glycerol (mL) and Vinegar (mL) (1: 0.5: 1)
CSS 2 = Starch (g), Glycerol (mL) and Vinegar (mL) (2: 0.5: 1)
CSS 3 = Starch (g), Glycerol (mL) and Vinegar (mL) (3: 0.5: 1)
CSSG 1 = Starch (g), Glycerol (mL) and Vinegar (mL) (3: 1: 1)
CSSG 2 = Starch (g), Glycerol (mL) and Vinegar (mL) (3: 1.5: 1)
CSSV 1 = Starch (g), Glycerol (mL) and Vinegar (mL) (3: 0.5: 0.5)
CSSV 2 = Starch (g), Glycerol (mL) and Vinegar (mL) (3: 0.5: 1.5)

According to physicochemical properties, pH and viscosity increased with increasing of starch and glycerol and decreased with an increasing amount of vinegar, respectively. The moisture content increased with increasing the amount of glycerol due to its hydrophilic nature. It can be suggested that glycerol comprises of hydroxyl group which has an affinity for water molecules that allow them to make hydrogen bonds and contain water in the structure.

Physicomechanical properties of Bioplastic Films

Observing the mechanical properties of bioplastics is essential to find their usability. Tensile strength and Young's Modulus of prepared bioplastics produced were tested in the study using a Universal testing machine. The resulting data were described in Table 3 and Figure 2-4. It was found that 3 g of starch, 0.5 mL of glycerol and 1 mL of vinegar were suitable conditions because they gave high tensile strength and tear strength than others. According to with physicomechanical data, increasing amount of 1 to 3 g of starch, the value of tensile strength increased because the strong hydrogen bonds formed by the starch-starch intermolecular interaction were more dominant than starch-glycerol interaction. Adding more of glycerol decreased the value of tensile strength because glycerol has the ability to reduce internal hydrogen bonds in intermolecular bonds, so that the addition of glycerol ratio increases plastic elasticity while the value of tensile strength is getting lower (Akbar *et al*., 2019). And then, 1 mL of vinegar was the most suitable condition because it gave more tensile strength and tear strength than other amounts. Based on the physicomechanical properties, 3g of starch, 0.5 mL of glycerol and 1 mL of vinegar were the most suitable conditions for preparation of bioplastic film. The prepared cassava starch based films using glycerol as plasticizer showed interesting mechanical properties such as being transparent, clear, homogeneous, flexible, and easily handled.

Table 3. Physicomechanical Properties of Cassava Starch Based Bioplastics Films

Sample (ratios) starch: glycerol: vinegar	Thickness (mm)	Tensile strength (MPa)	Elongation at break (%)	Tear strength (kN/m)
CSS 1 (1:0.5:1)	0.29	7.2	169	37.0
CSS 2 (2:0.5:1)	0.29	8.7	175	37.6
CSS 3 (3:0.5:1)	0.30	11.9	105	66.6
CSS 3 (3:0.5:1)	0.30	11.9	105	66.6
CSSG 1 (3:1.0:1)	0.30	7.4	178	37.8
CSSG 2 (3:1.5:1)	0.30	3.2	132	35.2
CSSV 1 (3:0.5:0.5)	0.29	4.3	105	60.0
CSS 3 (3:0.5:1)	0.30	11.9	105	66.6
CSSV 2 (3:0.5:1.5)	0.29	6.0	145	44.5

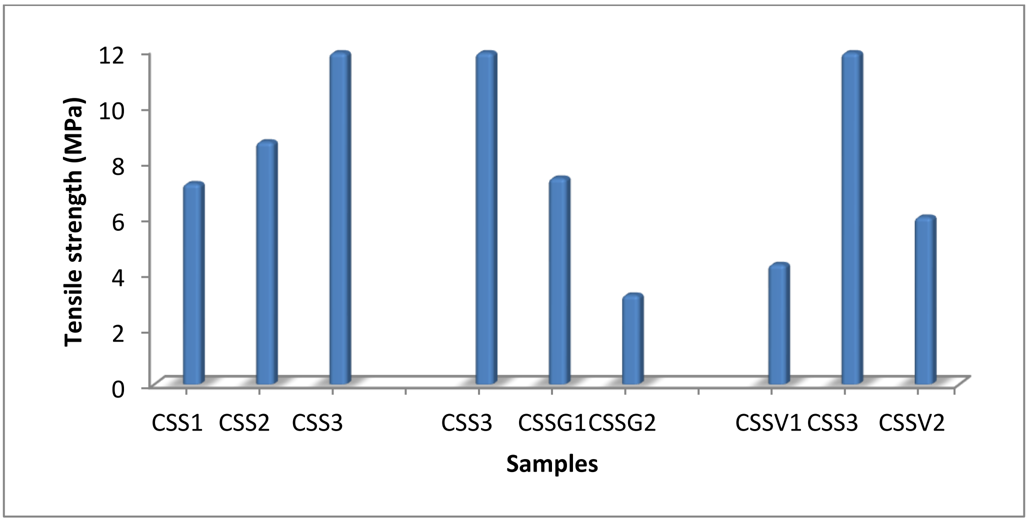


Figure 2. The bar graph of tensile strength vs prepared bioplastics films

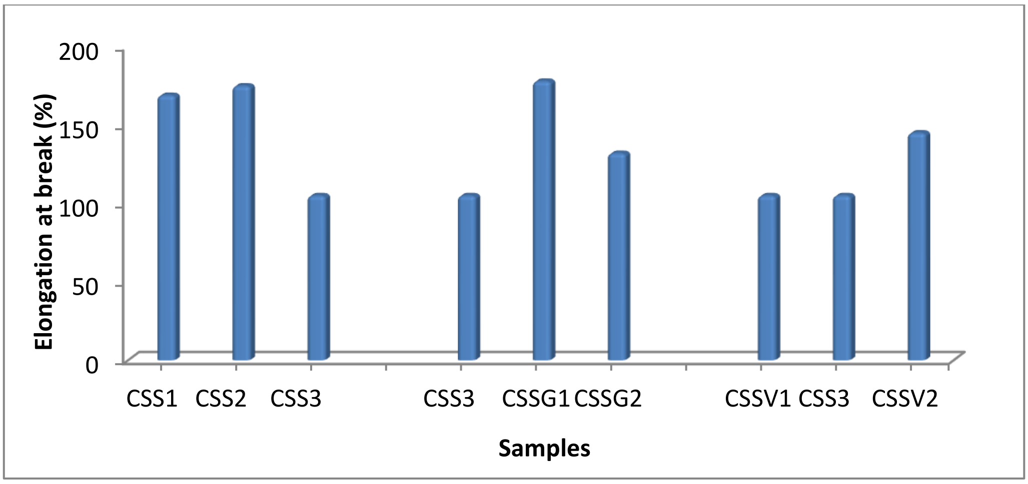


Figure 3. The bar graph of elongation at break (%) vs prepared bioplastics films

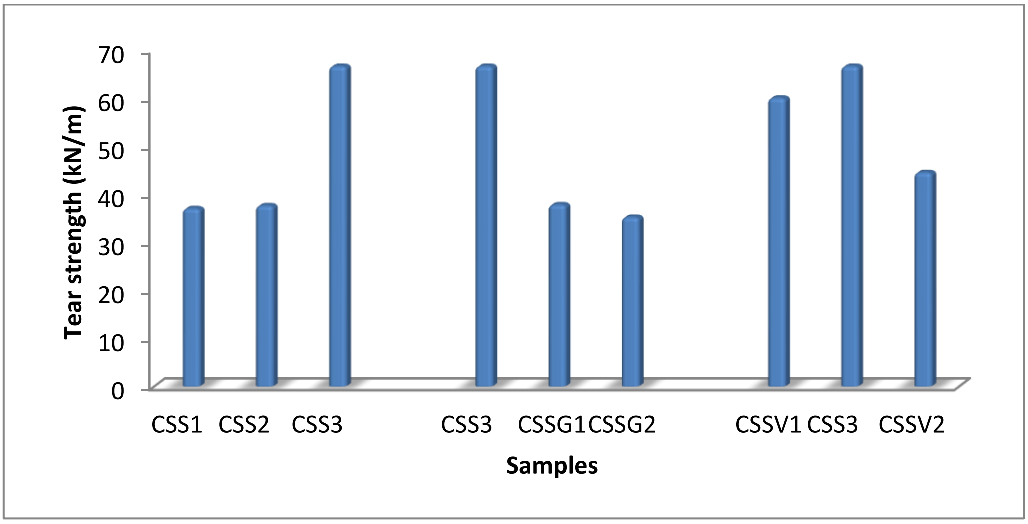


Figure 4. The bar graph of tear strength vs prepared bioplastics films

Transparency Test Results of Prepared Cassava Starch Based Bioplastics Films

The transparency of prepared starch based Cassava bioplastic films was investigated by using UV-vis spectrophotometer. The results were presented in Tables 4, 5, 6 and Figure 5.

Table 4. Transparency Test Results for Prepared Bioplastic Films with Various Ratios of Starch

Samples	Absorbance	Thickness (mm)	Average Transparency
CSS 1	0.452	0.29	1.55
CSS 2	0.612	0.30	2.04
CSS 3	0.706	0.29	2.43

Table 5. Transparency Test Results for Prepared Bioplastics Films with Various Ratios of Glycerol

Samples	Absorbance	Thickness (mm)	Average Transparency
CSS 3	0.706	0.29	2.43
CSSG 1	0.501	0.30	1.67
CSSG 2	0.485	0.30	1.62

Table 6. Transparency Test Results for Prepared Bioplastics Films with Various Ratios of Vinegar

Samples	Absorbance	Thickness (mm)	Average Transparency
CSSV 1	0.411	0.29	1.41
CSS 3	0.706	0.29	2.43
CSSV 2	0.836	0.29	2.88

CSS 1 CSS 2 CSS 3 CSSG 1 CSSG 2 CSSV 1 CSSV 2

Figure 5. Transparency test of prepared bioplastics

According to experimental results and transparency images, the highest value of average transparency was found in CSS 3 bioplastic film. It can be assumed that the more amount of starch was added, the higher the value of transparency. The more glycerol used, the value of transparency will tend to decrease because the concentration of the solution of bioplastic was increasing so that its use did not work optimally. The vinegar helps the starch to dissolve easily because it adds ions to the mixture (Susilawati *et al*., 2019).

Water Absorption Capacity (%) of Prepared Cassava Starch-Based Bioplastic Films

The water absorption capacity of cassava starch-based bioplastic films depends on the concentration of starch and glycerol ratios. The results were summarized in Table 7 and Figure 6.

Table 7. Water Absorption Capacity (%) of Prepared Bioplastic Films

Samples	Initial Weight	Post-Brake Weight	Water Absorption Capacity (%)
CSS 1	0.12	0.34	183.3
CSS 2	0.15	0.47	213.3
CSS 3	0.19	0.77	305.2
CSS 3	0.19	0.77	305.2
CSSG 1	0.21	0.88	319.0
CSSG 2	0.22	0.93	322.7
CSSV 1	0.16	0.63	293.0
CSS 3	0.19	0.77	305.2
CSSV 2	0.20	0.75	275.0

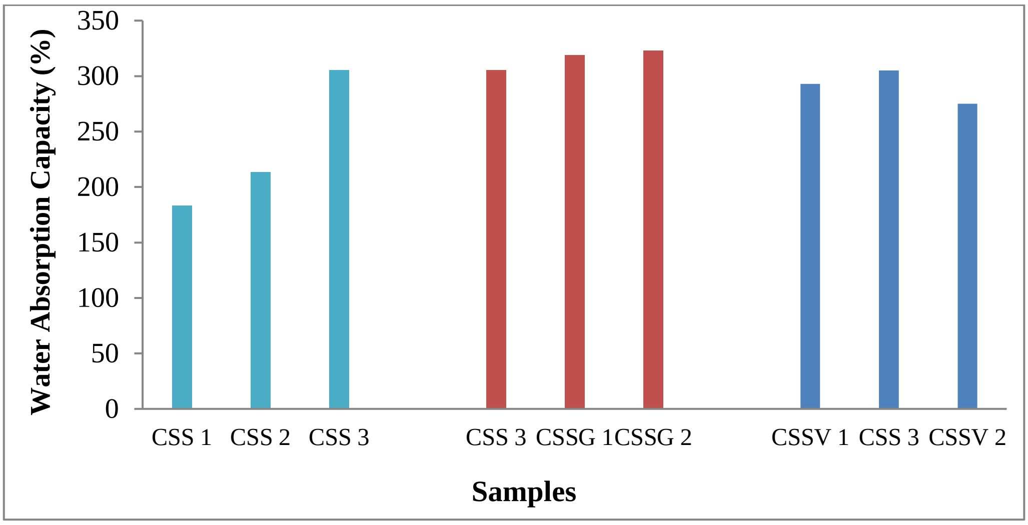


Figure 6. The bar graph of water absorption capacity vs prepared bioplastic films

According to this data, the water absorption capacity of bioplastic increased, with an increasing amount of starch because starch indicates the concentration of hydrophilic properties in the bioplastics. The reason for this is that the hydroxyl group in starch has an affinity for water molecules and the gelatinization also breaks up starch a granule which lets water diffuse in it (Nanang *et al*., 2017).

FT IR analysis of selected bioplastic film

Functional groups and any potential chemical alterations upon the addition of plasticizers and fillers were observed using the FT IR analysis. The FT IR spectrum and resulted data were evaluated in Figure 7 and Table 8. The broad band at 3000−3600 cm^{-1} is attributed to the O−H stretching vibrations of absorbed water present in starch. Moreover, the peak at 2800−3000 cm^{-1} is assigned to the band of flexion of the CH_2 deformation. Additionally, the peak at 1630−1650 cm^{-1} is assigned to the bending vibration of O−H bonds of absorbed water molecules in the amorphous region of cassava starch. Moreover, the peak in the region of 1400−1430 cm^{-1} corresponds to the $-CH_2$ symmetric scissoring. Apart from the above, the peak at 1323−1380 cm^{-1} corresponds to the C−O−H bending and C−O−C bending vibrations.

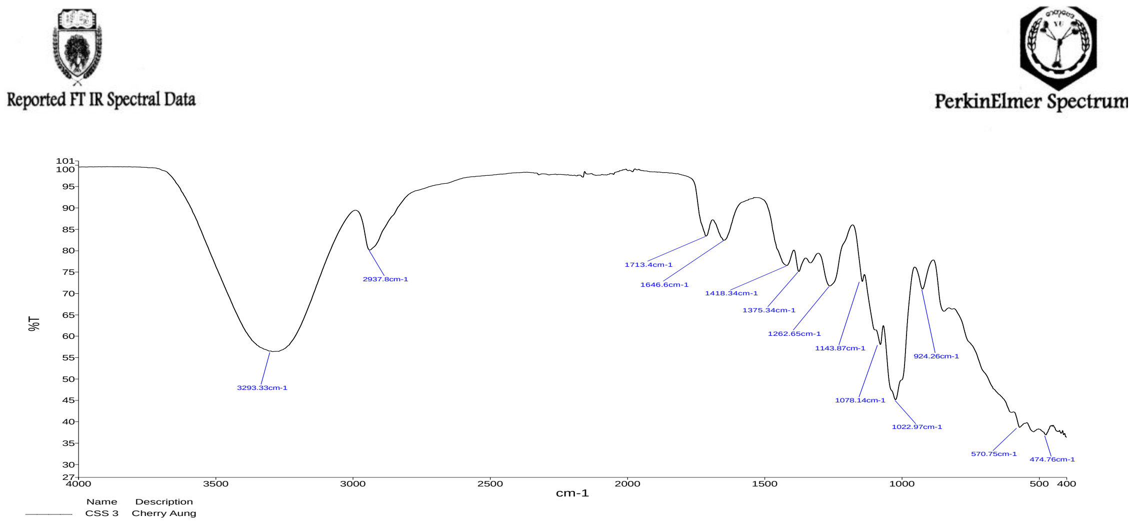


Figure 7. FT IR spectrum of cassava starch based bioplastic film

Table 8. FT IR Spectral Data of Selected Bioplastics

Observed wave number (cm^{-1})	***Literature wave number (cm^{-1})**	**Band assignment**
3293	3307	OH stretching
2937	2928	CH stretching
1646	1645	C-O bending associated with OH group
1022	1018	C-O-C stretching
929	920	C-O-C ring vibration of carbohydrate

***** Akbar *et al*., 2019

Thermogravimetry analysis of selected bioplastics films CSS 3

Thermo gravimetric analysis is a method used to study the reaction of thermal decomposition between weight change and temperature which are lost due to the effect of temperature on the materials. The TG DTA thermogram of prepared bioplastic film was shown in Figure 8 and the results were described in Table 9. The TG profiles of starch-based prepared bioplastic film showed three distinct weight loss regions at the peak temperatures of 80 °C, 331 °C and 537 °C.

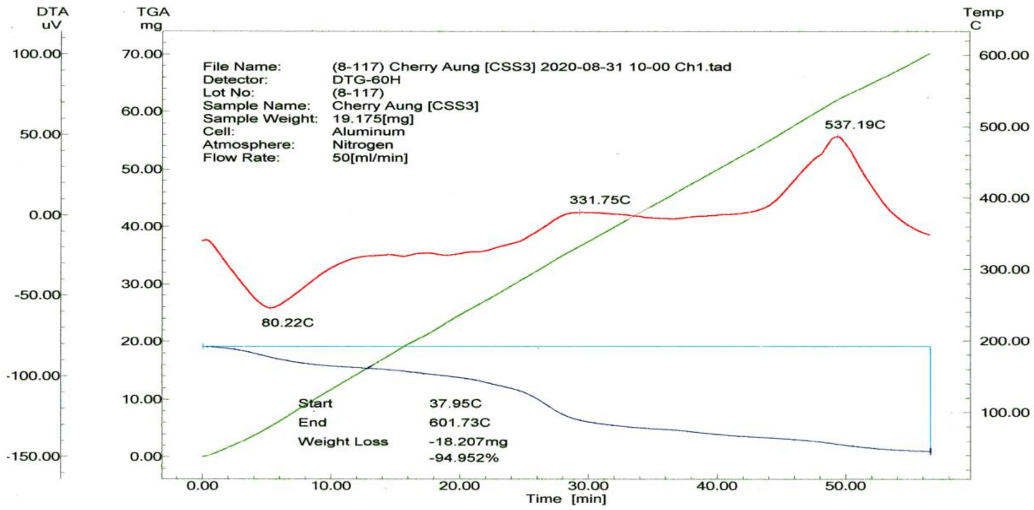


Figure 8. TG DTA thermogram of selected bioplastics films CSS 3

Table 9. TG DTA Analysis Data of selected Bioplastic CSS 3

Temperature Range (°C)	Weight Loss (%)	Peak's Temperature (°C)	Nature of Peak	TG Remark
37-100	11.343	80	endothermic	volatilization of absorbed water
100-340	59.974	331	exothermic	decomposition of hemicellulose and cellulose
340-560	20.860	537	exothermic	decomposition of lignin and ash formation

According to TG DTA analysis data, the endothermic was found at 80 °C with weight loss within the temperature from 37 – 100 ºC which was attributed to the volatilization of absorbed water and the release of excess water in the prepared bioplastic film. The DTA profiles of prepared bioplastic film exhibited exothermic peak corresponding to the degradation temperature of hemicelluloses and celluloses in cassava starch at 331 °C, suggesting the thermal stability of starch. The later event at a peak temperature 537 °C was attributed to the thermal decomposition of lignin and ash formation. The TG DTA data were reliable with reported literature because cassava starch contained around 26% of cellulose, 32% starch, 17% hemicelluloses and 16% lignin. Similar results in starch based cassava bioplastics samples have been reported before as well in the literature (Nanang *et al*., 2017).

Shelf Life testing of selected bioplastic film

The shelf life of prepared bioplastic films was evaluated at the storage of room temperature for 105 days. As shown in Figure 9, a little fungus was introduced at 45 days and it gradually increased day by day. The increase of fungus was fully covered at 75 days to 105 days. It can be suggested that the polymer chain in prepared bioplastic film split as a result of enzymes of depolymerize the polymers it can be excreted outside the cell. The shelf life of bioplastic is quite low and it will damage food stuff being packed due to the migration of spoilage bacteria to the food (Susilawati *et al*., 2019).

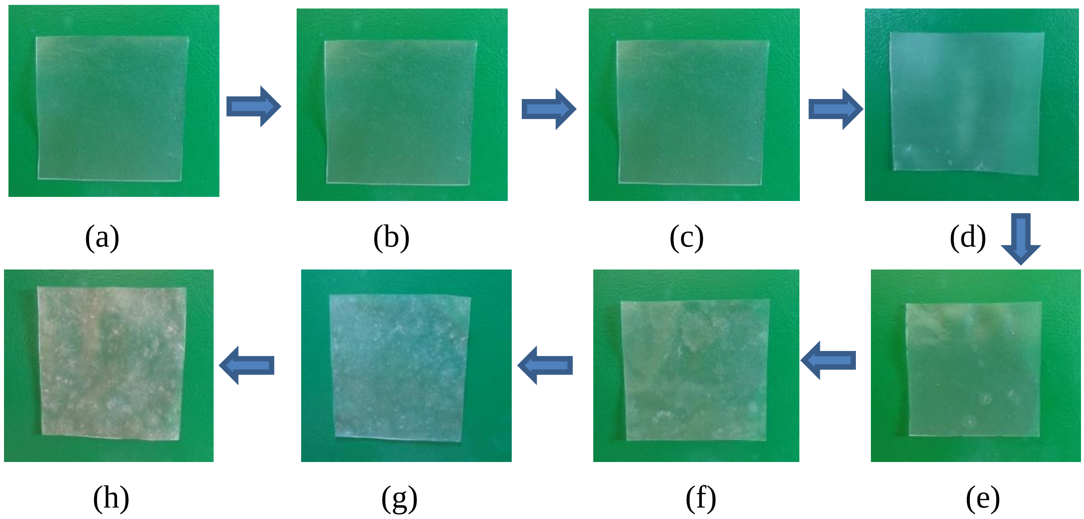


Figure 9. Photographs of the physical appearances of selected bioplastic films (CSS 3) (a) before and after (b) 15 days (c) 30 days (d) 45 days (e) 60 days (f) 75 days (g) 90 days and (h) 105 days at room temperature by shelf life testing

Biodegradable ability of the selected bioplastic film

The biodegradable ability of prepared bioplastic films was investigated by the soil burial method. Biodegradation is the process of organic matter broken down by microorganisms such as bacteria and fungi that live in the soil (Park *et al.*, 1994). The biodegradation of the prepared bioplastic film was tested in a compost environment over 12 days, and the image of degradable behavior was presented in Figure 10.

In the compost environment, the water molecules diffuse into the cassava starch chain causing swelling and enhancing biodegradation via microbial activity. According to the physical appearances, the bioplastic film was almost degraded after 12 days. The addition of glycerol in prepared bioplastic film improving the biodegradation of samples can be attributed to the better water absorption capacity of the samples which is because of the affinity of the plasticizer (glycerol) towards water.

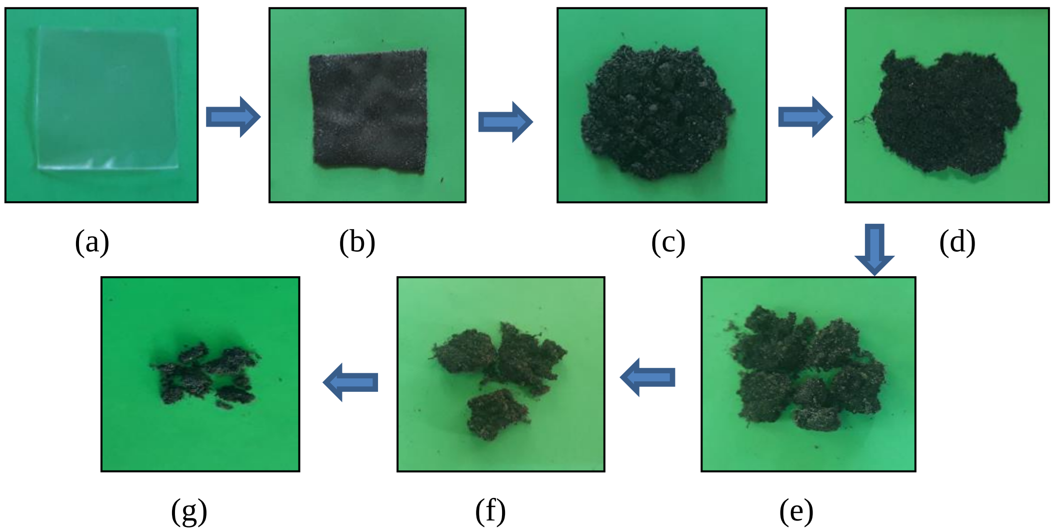


Figure 10. The physical appearances of selected bioplastic films (CSS 3) (a) before and after (b) 2 days (c) 4 days (d) 6 days (e) 8 days (f) 10 days (g) 12 days in soil by soil burial method

Plastic waste is Global crisis; degradable plastic is introduced to overcome this limitation. The properties evaluation results of the prepared starch-based cassava bioplastic films thus demonstrated their aptness for packaging applications. The present work has demonstrated the ability of starch in the production of bioplastics that could serve as packaging materials to replace synthetic plastic, thus preventing environmental pollution.

Conclusion

This study showed that starches from natural sources can be used with the addition of plasticizers to produce bioplastic films boasting different physical and chemical characteristics. According to the physicomechanical properties, the optimum formulation compositions of starch, glycerol and vinegar (3g : 0.5mL : 1mL) were investigated. The transparency and water absorption capacity of the bioplastic films depends on the starch and glycerol ratio. The differences in these properties will allow the bioplastics to be suitable for varying applications. All the bioplastics produced were biodegradable and environmentally friendly, thus being a good substitute for petroleum-based plastics, and an efficacious way to alleviate the problem of plastic pollution. Moreover, using inexpensive raw materials for the synthesis of bioplastics can also make them economically feasible.

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