Preparation and Characterization of Biodegradable Starch Based Bioplastic Films

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Abstract

This paper focused on green-production of bioplastic films and characterization of starch based bioplastics. The starch powders were extracted from fresh corn and cassava by cold extraction method. The yield percent of the extracted corn and cassava starch is 5.71 and 14.32 %. The physicochemical properties of prepared starch such as pH, moisture content, ash content and bulk density were detected. The bioplastic films: CS 1,2 & 3, CSSG 1,2 & 3 and CSSV 1,2 &3 were prepared by casting method with varying amount 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. Based on the physicomechanical properties, the ratio of starch, glycerol and vinegar (3: 0.5: 1 g/mL/mL) is the most suitable ratio for preparation of bioplastic films. The water absorption, shelf life and biodegradable ability of prepared bioplastic films were also investigated. The prepared bioplastic films were characterized by using Fourier transform infrared (FT IR) analysis. The FT IR results showed the presence of O-H, C-H, C-O-C stretching and C-O-C ring vibration of carbohydrate. The decomposition nature of prepared biodegradable films was investigated by TG DTA. The water absorption of prepared bioplastic films was determined by soaking in water for 24 hours. The biodegradable ability of bioplastic films was examined by soil burial test. The soil burial test results, CS 3 and CSS 3 bioplastic films were almost degraded after 14 days and 12 days respectively. According to the overall results, among the prepared bioplastic films; CS 3 and CSS 3 were selected and applied for production of food container: cups, boxes, spoons and forks, etc. In this study, the raw materials used for preparation of bioplastic films were natural, renewable resources and environmental friendly and showed biodegradation, and thus proving to be a good alternative to the conventional plastics.

Keywords: corn, cassava, starch, bioplastic, biodegradation, shelf life

1. 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 of cassava starch as the main ingredient in the manufacture of bioplastics shows great potential as a diverse range of starch-producing plants. Plastic waste poses a risk to 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 it is easy to make it, and there are abundant and affordable raw materials. It is categorized environmental friendly and able to be degraded by the activity of microorganisms by 10 to 20 times faster than the conventional plastic resulting in carbon dioxide, methane, water, biomass, humus and other

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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 soft, white tasteless powder that is soluble in water, alcohol or other solvents. The molecular structure of starch is $(C_6H_{10}O_5)_n$. The starch granule is a heterogeneous material and chemically, it 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, etc. There are many types of starches and the most common types are corn, cassava, potato, wheat and rice starch (Avella *et al*., 2005).

A few plastics use corn starch and cassava starch as an added substance. It helps in the normal decay of the plastic materials. Corn (*Zea mays L.)* is one of the major food crops and it is considered to be a very distinctive plant, since it is able to produce a large amount of the natural polymer of starch through its capacity to utilize large amount of sunlight. Corn is the cheapest available source of commercially available sugar and also the most abundantly available. Corn starch is used in a wide range of products and applications. Corn starch plastic has become very popular as a replacement for traditional plastics as it is more environmentally friendly.

Cassava is the third major source of carbohydrate 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 an 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 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 corn and cassava for the production of bioplastic films. Physicochemical and physico-mechanical properties of corn and cassava starch based bioplastic films were studied. Based on the data, the best optimum ratios of ingredients for preparing bioplastic were identified. Moreover, the behaviors of prepared bioplastic films were also investigated. The biodegradable ability of prepared bioplastic film was investigated by soil burial method. The best selected bioplastic films are intended for making various food containers as plastic substitute bioplastic films.

2. Materials and Methods

Materials

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

Extraction of starch from corn and cassava

Firstly, 1,000g of corn was blended with distilled water at high speed for 2 min using a blender. The slurry was collected through a piece of clean cloth. The filtrates were left to settle in the beaker for 3 hours. After that the resulting supernatant liquids were decanted, leaving behind white starches which had settled at the bottom for 3 hours. The residue was washed with distilled water two times and dried in a vacuum oven at 105 ºC. After drying, it was grinded to a uniform powder using motor and pestle in the laboratory. Then corn starch powder was weighed and the yield percent was calculated.

Secondly, cassava starch powder was prepared in the same way. The physicochemical properties such as pH, moisture content, ash content and bulk density of corn and cassava starch were determined.

Preparation of bioplastic film from corn and cassava starch

Bioplastic films were prepared by casting method using various ratios of starch, glycerol and vinegar and made up with 100 mL of distilled water. The mixed solution was heated, constantly stirred using magnetic stirrer until the mixture to gelatinize at approximately 70 ºC. The resultant starch paste was poured and casted onto a melamine plate and then dried in oven at 60 ºC to obtain bioplastic films. Obtained bioplastic films using variable amount of corn starch (1, 2& 3g) were noted as CS 1 to CS 3 and CSS 1 to CSS 3 for cassava starch. Similarly, prepared bioplastic films using variable amount of glycerol (0.5,1&1.5mL) were noted as CSG 1 to 3 for corn starch and CSSG 1 to 3 for cassava and variable amount of vinegar (0.5,1&1.5mL) were noted as CSV1 to 3 for corn starch and CSSV 1 to 3 for cassava. Totally 18 bioplastic films were prepared: 9 films of corn starch based and 9 films of cassava starch based. The aim was to evaluate the effect of amount of starch, glycerol and vinegar on the physicomechanical properties of prepared films, one of three in the ratio was varied while the other variables fixed when preparing bioplastic films.

Characterization of physicomechanical properties of bioplastic film

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

Determination of water absorption (%)

The water absorption showed the ability of bioplastics to absorb water. Bioplastics were cut into 1″ × 1″ size and weighed. Then they were dried for 24 hours in an oven at 50 ºC, cooled in a desiccator and weighed. After that they were soaked in water for 24 hours. Then, they were dropped out and wrapped with tissue paper and immediately weighed (Nanang *et al.*, 2017). The water absorption (%) of bioplastic films were calculated by using the equation:-

$$\text{Water absorption (\%)} = \frac{W_f - W_i}{W_i} \times 100$$

FT IR analysis of bioplastic film

FT IR analysis indicates the presence of functional groups in bioplastic film. The functional groups of prepared selected bioplastic films were analyzed by Fourier Transform Infrared Spectroscopy (FT IR) in the wavenumber range of 400−4000 cm^{-1}.

Thermo-gravimetric differential thermal analysis (TG DTA)

Thermal stability of 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. These films were placed in a plastic bag at room temperature and watched on day by day. These films were analyzed through visual observation with a camera (Susilawati *et al.*, 2019).

Determination of biodegradability

Biodegradable behavior of bioplastic was determined using soil burial test. In this test, bioplastic films were cut into 1″ × 1″ size, and buried in the ground at 5 cm depth with the burial duration interval of two days (Park *et al.*, 1994).

3. Results and discussion

Physicochemical properties of corn and cassava starch

The physiochemical characteristics of corn and cassava starch were shown in Table (1). It was found that the values of pH, bulk density and yield percent of cassava starch were higher but ash content (%) and moisture content (%) were lower than that of corn starch. It may depend on cultivar, region and climatic condition.

Starch based bioplastic films

Prepared corn and cassava starch-based bioplastic films were shown in Figures 1 and 2. All the prepared bioplastics films were odorless, transparent, and smooth. It was observed that cassava starch-based bioplastic films were more transparent and smoother than corn starch based bioplastic films.

Table 1. Physicochemical properties of corn and cassava starch

Starch	pH	Ash content (%)	Moisture content (%)	Bulk Density (gcm^{-3})	Yield (%)
Corn	6.0	1.97	11.50	0.52	5.71
Cassava	6.8	1.65	7.80	0.75	14.32

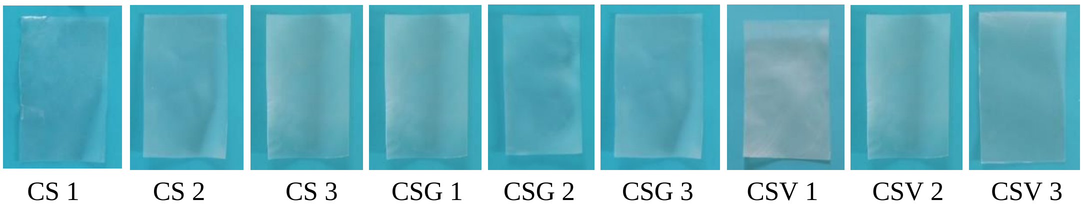


Figure 1. Photographs of prepared corn starch based bioplastic films

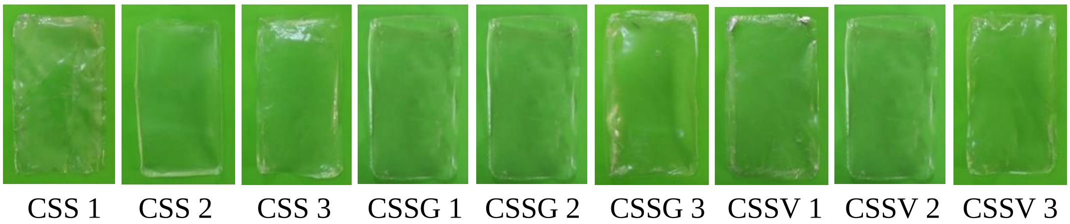


Figure 2. Photographs of prepared cassava starch based bioplastic films

Physicochemical properties of bioplastic films

Physicochemical properties of corn and cassava starch based bioplastic films were determined. The effect of amount of starch, glycerol and vinegar on the physicochemical properties of prepared films were investigated and shown in Tables 2 and 3.

Table 2. Physicochemical properties of corn starch based bioplastics films

Bioplastics Films (corn:glycerol:vinegar)	pH	Moisture content (%)	Viscosity (cP)	Specific gravity
CS 1 (1: 0.5: 1)	3.8	19.85	18.8	1.0198
CS 2 (2: 0.5: 1)	3.9	19.04	23.3	1.0189
CS 3 (3:0.5:1)	4.0	17.18	102.2	1.0149
CSG 1 (3:0.5:1)	4.0	17.18	102.2	1.0149
CSG 2 (3:1.0:1)	3.8	16.66	87.2	1.0234
CSG 3 (3:1.5:1)	4.1	18.52	123.4	1.0128
CSV 1 (3:0.5:0.5)	4.2	19.05	143.2	1.0254
CSV 2 (3:0.5:1)	4.0	17.18	102.2	1.0149
CSV 3 (3:0.5:1.5)	3.7	16.67	89.1	1.0125

According to physicochemical properties, pH and viscosity increased with the increase of starch and glycerol and decreased with the increasing amount of vinegar, respectively. The moisture content increased with the increasing amount of glycerol due to the 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. The values of pH and viscosity (cP) of cassava starch based bioplastic were higher but the moisture content (%) was lower than that of corn starch based bioplastic.

Table 3. Physicochemical properties of cassava starch based bioplastics films

Bioplastic Films (cassava:glycerol:vinegar)	pH	Moisture content (%)	Viscosity (cP)	Specific gravity
CSS 1 (1:0.5:1)	4.0	19.05	53.5	1.0134
CSS 2 (2:0.5:1)	4.1	16.67	80.3	1.0162
CSS 3 (3:0.5:1)	4.2	14.28	114.8	1.0194
CSSG 1 (3:0.5:1)	4.2	14.28	114.8	1.0194
CSSG 2 (3:1.0:1)	4.0	18.18	112.8	1.0176
CSSG 3 (3:1.5:1)	4.4	19.09	124.6	1.0199
CSSV 1 (3:0.5:0.5)	4.4	15.38	125.6	1.0174
CSSV 2 (3:0.5:1)	4.2	14.28	114.8	1.0194
CSSV 3 (3:0.5:1.5)	3.8	18.05	110.8	1.0194

Physicomechanical Properties of Bioplastic Films

Observing the mechanical properties of bioplastics is essential for their usability. Tensile strength and Young's Modulus of prepared bioplastics were tested by using Universal testing machine. The resulted data were described in Tables 4 and 5 and Figures 3, 4 and 5.

Table 4. Physicomechanical Properties of Corn Starch Based Bioplastics Films

Bioplastic films (corn: glycerol: vinegar)	Thickness (mm)	Tensile strength (MPa)	Elongation at break (%)	Tear strength (kN/m)
CS 1 (1:0.5:1)	0.39	6.2	236	52.0
CS 2 (2:0.5:1)	0.39	6.4	245	58.3
CS 3 (3:0.5:1)	0.40	9.8	142	79.5
CSG 1 (3:0.5:1)	0.40	9.8	142	79.5
CSG 2 (3:1.0:1)	0.40	6.6	256	76.7
CSG 3 (3:1.5:1)	0.40	4.6	210	37.6
CSV 1 (3:0.5:0.5)	0.39	7.9	190	53.5
CSV 2 (3:0.5:1)	0.40	9.8	142	79.5
CSV 3 (3:0.5:1.5)	0.39	7.0	168	70.0

The data show that with the 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. By adding more of glycerol decreased the value of tensile strength because glycerol has the ability to reduce internal hydrogen bonds in intermolecular bonds, so the addition of glycerol ratio to increase plastic elasticity while the value of tensile strength is getting lower (Akbar *et al*., 2019). It was observed that 1 mL of vinegar was the most suitable amount of bioplastic films because it gave more tensile strength and tear strength than the other amount. In this process, glycerol was used as a plasticizer and it improved the properties of prepared films: transparent, clear, homogeneous, flexible, and easily handled. Based on the physicomechnical properties of prepared bioplastic films, the optimum ratios of starch, glycerol and vinegar was found to be 3 g of starch, 0.5 mL of glycerol and 1 mL of vinegar for both corn and cassava starch. Therefore, CS 3 and CSS 3 were selected bioplastic films due to their high tensile strength and tear strength than the other films.

Table 5. Physicomechanical Properties of Cassava Starch Based Bioplastics Films

Bioplastic films (cassava: 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
CSSG 1 (3:0.5:1)	0.30	11.9	105	66.6
CSSG 2 (3:1.0:1)	0.30	7.4	178	37.8
CSSG 3 (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
CSSV 2 (3:0.5:1)	0.30	11.9	105	66.6
CSSV 3 (3:0.5:1.5)	0.29	6.0	145	44.5

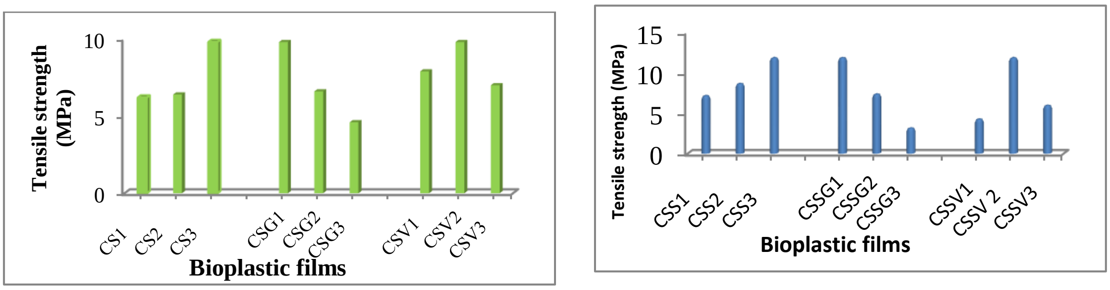


Figure 3. The bar graph of tensile strength vs prepared bioplastics films

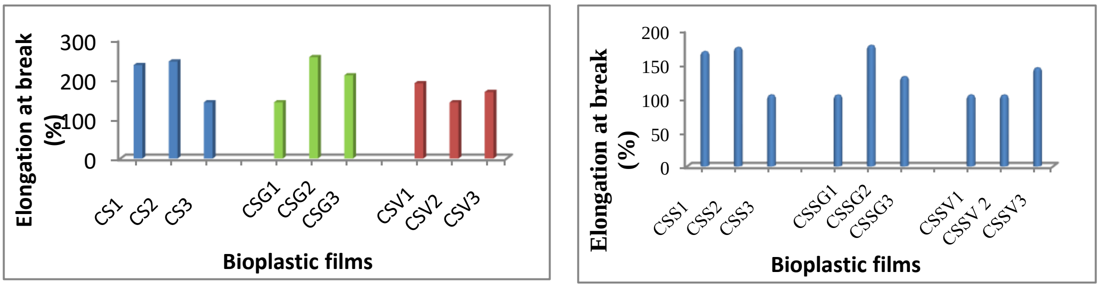


Figure 4. The bar graph of elongation at break (%) vs prepared bioplastics films

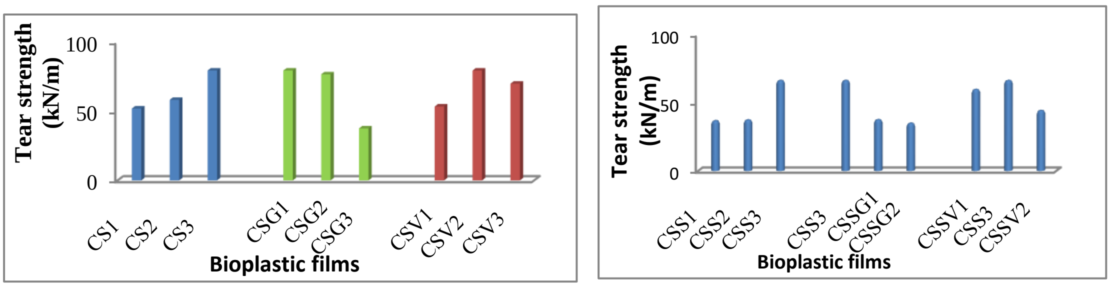


Figure 5. The bar graph of tear strength vs prepared bioplastics films

Water Absorption (%) of Starch Based Bioplastic Films

The water absorption of starch based bioplastic films depends on the concentration of starch and glycerol ratios. The results were shown in Tables 6 and 7 and Figure 6.

Table 6. Water absorption (%) of corn starch based bioplastic films

Samples	Initial Weight (g)	Final Weight (g)	Water Absorption (%)
CS 1	0.12	0.29	141.6
CS 2	0.19	0.50	163.2
CS 3	0.21	0.62	195.2
CSG 1	0.21	0.62	195.2
CSG 2	0.23	0.70	204.3
CSG 3	0.24	0.75	212.5
CSV 1	0.21	0.55	161.9
CSV 2	0.21	0.62	195.2
CSV 3	0.21	0.60	185.7

Table 7. Water absorption (%) of cassava starch based bioplastic films

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

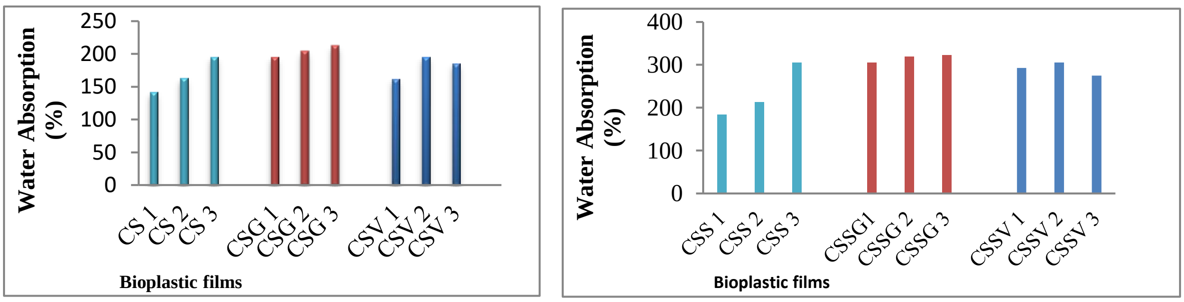


Figure 6. The bar graph of water absorption vs prepared bioplastic films

According to the data, the water absorption of bioplastic increased, with the 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). Water absorption (%) of cassava starch based bioplastic film is higher than corn starch based bioplastic films.

FT IR analysis of selected bioplastic films

Functional groups and any potential chemical alterations due to addition of plasticizers and fillers were evaluated by 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 starch. Moreover, the peak in the region of 1400−1430 cm^{-1} corresponds to the $-CH_2$ symmetric scissoring. Apart from that, the peak at 1323−1380 cm^{-1} corresponds to the C−O−H bending and C−O−C bending vibrations. Both starches possess an O-H, C-H, C-O-C and C-O functional group respectively. Comparison of FT IR spectra showed that starch from corn and cassava possess similar chemical structure.

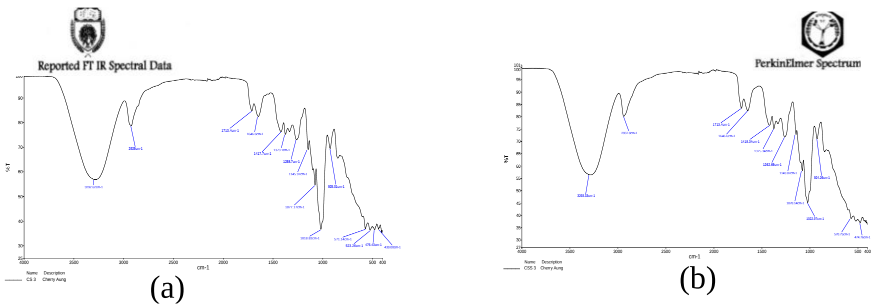


Figure 7. FT IR spectra of (a) CS 3 and (b) CSS 3

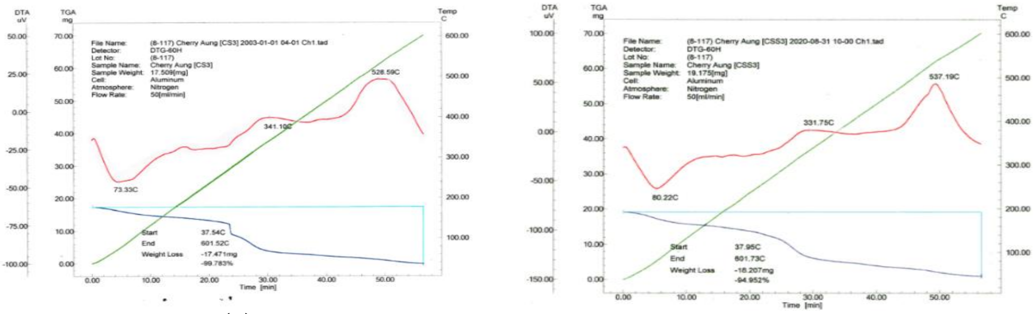
Table 8. FT IR spectral data of selected bioplastics film (CS 3 and CSS 3)

Observed wave number (cm^{-1})		*Literature wave number (cm^{-1})	Band assignment
CS 3	CSS 3		
3292	3293	3307	OH stretching
2925	2937	2928	CH stretching
1646	1646	1645	C-O bending associated with OH group
1018	1022	1018	C-O-C stretching
925	929	920	C-O-C ring vibration of carbohydrate

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

TG DTA analysis of selected bioplastic films

According to TG DTA data of selected bioplastic film (CS 3), the endothermic was found to be at 70-80 °C with weight loss within the temperature from 37 – 100 ºC which 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 the degradation temperature of hemicelluloses and celluloses at 331-341 °C, suggesting the thermal stability of starch. The later event at peak temperature range 528-537 °C was attributed to the thermal decomposition of lignin and ash formation. The TG DTA data were reliable with reported literature because starch contained around 26% of cellulose, 32% starch, 17% hemicelluloses and 16% lignin. The similar results in cassava starch based bioplastics film (CSS 3) have been reported before as well as in literature (Nanang *et al.*, 2017).



(a) (b)

Figure 8. TG DTA thermograms of selected bioplastics films (a) CS 3 and (b) CSS 3

Table 9. TG DTA analysis data of selected bioplastic films (CS 3 and CSS 3)

Temperature Range (°C)		Weight Loss (%)		Peak's Temperature (°C)		Nature of Peak	TG Remark
CS 3	CSS 3	CS 3	CSS3	CS 3	CSS 3		
37-100	37-100	10.046	11.343	73	80	endothermic	volatilization of absorbed water
100-360	100-340	77.103	59.974	341	331	exothermic	decomposition of hemicellulose and cellulose
360-560	340-560	9.995	20.860	528	537	exothermic	decomposition of lignin and ash formation

Shelf life testing of selected bioplastic films (CS 3 and CSS 3)

The shelf life of prepared bioplastic films was evaluated at the storage of room temperature during 105 days. As shown in Figures 9 and 10, a little fungus was introduced at 60 days in CS 3 and 45 days in CSS 3, and it gradually increased day by day. The increasing 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. If the shelf life of bioplastic is quite low, it will damage food stuff being packed due to migration of spoilage bacteria to the food (Susilawati *et al.*, 2019).

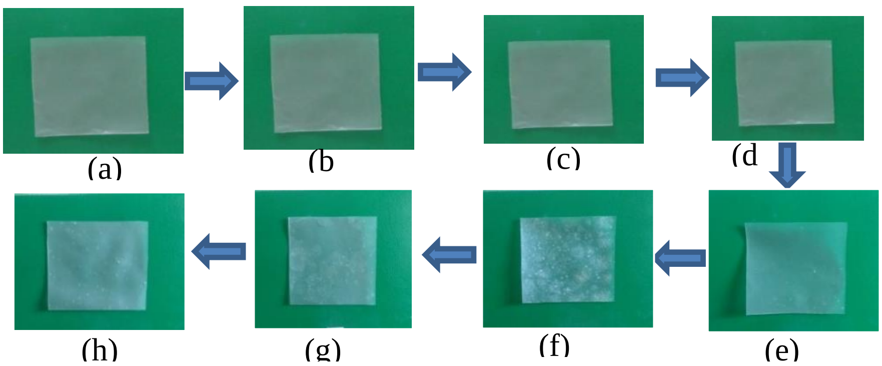


Figure 9. Photographs of the physical appearances of selected bioplastic films (CS 3)
(a) before and after (b) 15 (c) 30 (d) 45 (e) 60 (f) 75 (g) 90 and (h) 105 days at room temperature by shelf life testing

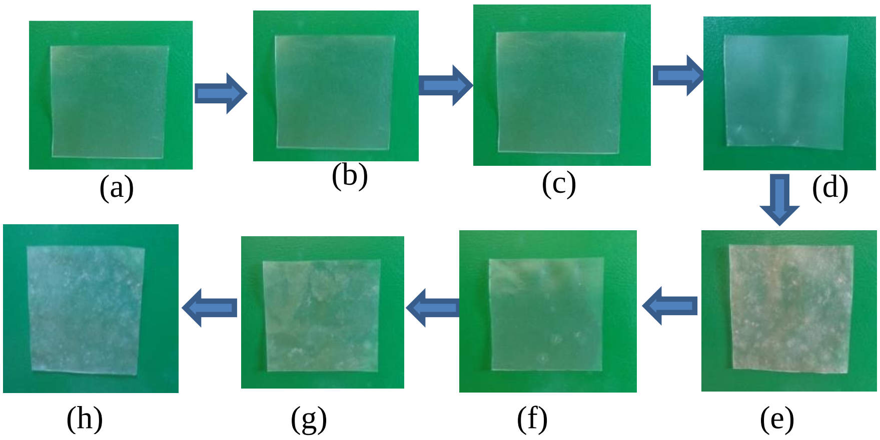


Figure 10. Photographs of the physical appearances of selected bioplastic films (CSS 3) (a) before and after (b) 15 (c) 30 (d) 45 (e) 60 (f) 75 (g) 90 and (h) 105 days at room temperature by shelf life testing

Biodegradable ability of the selected bioplastic films

The biodegradable ability of prepared bioplastic films was investigated by soil burial method. Biodegradation is the process of organic matter broken down by microorganisms such as bacterial and fungi that are live in the soil (Park *et al*., 1994). The biodegradation of the prepared bioplastic film was tested in a compost environment over 14 days in CS 3 and 12 days in CSS 3 and the image of degradable behavior was presented in Figures 11 and 12.

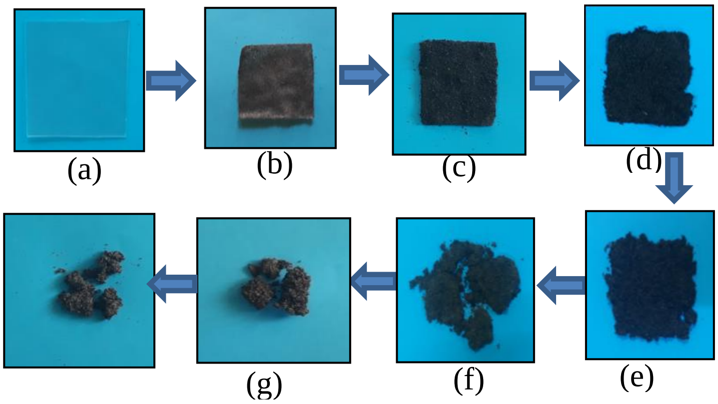


Figure 11. The physical appearances of selected bioplastic fims (CS 3) (a) before and after (b) 2 (c) 4 (d) 6 (e) 8 (f) 10 (g) 12 and (h) 14 days in soil by soil burial method

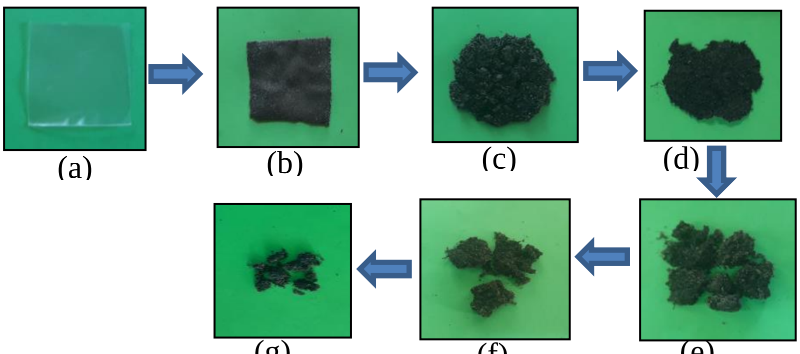


Figure 12. The physical appearances of selected bioplastic films (CSS 3) (a) before and after (b) 2 (c) 4 (d) 6 (e) 8 (f) 10 and (g) 12 days in soil by soil burial method

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 and 14 days. Addition of glycerol in prepared bioplastic film improving the biodegradation of samples can be attributed to better water absorption capacity of the samples which is because of the affinity of the plasticizer (glycerol) towards water.

4. Conclusion

In this study, starch based bioplastic films were successfully prepared by casting method and investigated their physical and chemical characteristics. According to the physicomechanical properties, the optimum formulation compositions of starch, glycerol and vinegar (3: 0.5: 1) was chosen to prepare as selected bioplastic films: CS 3 and CSS 3 for applications. The water absorption, shelf life and biodegradable ability of selected bioplastic films were also investigated. Based on the overall results, CS 3 and CSS 3 bioplastic films were found to be used for the production of food containers. This research pointed that prepared bioplastic films were biodegradable and environmental-friendly, thus being a good substitute of 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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