SYNTHESIS AND CHARACTERIZATION OF BIODEGRADABLE PLASTIC MATERIAL COMPOSED OF LOW-DENSITY POLYETHYLENE AND CASSAVA STARCH

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ABSTRACT

Bioplastics make up less than 1% of overall plastics manufacturing, but the industry is growing. In 2019, about half of all bioplastics on the market (44.5 percent) are bio-based but not biodegradable. Biopolymers are the most hopeful material for this application because of their biodegradability and prolonged storage life properties which includes resistance to chemical and enzymatic activity. Starch is biodegradable, low-cost, and easily obtainable material. Starch cannot be considered a thermoplastic material due to van der Waals forces interactions and hydrogen bonding. Several plasticizers are used to make thermoplastic starches, which are thermoplastic materials that deform. Glycerol and palm oil were used individually, together with cassava starch and Low-Density Polyethylene. The compression, micro hardness, and biodegradability of the bioplastic were investigated. SEM and EDX were used to investigate the starch particles. According to biodegradation experiments applying glycerol as a plasticizer, 95% of the LDPE has degraded by 2.26%, 5.44%, and 9.5% after 7, 14, and 28 days respectively. Utilizing palm oil as a plasticizer depicts that 95% of the LDPE degraded by 1.22%, 1.81%, and 3.92% after 7, 14, and 28 days. This indicates that glycerol and starch biodegrade faster than palm oil and starch. This study reveals that the combination of glycerol and cassava starch has better physicochemical properties than palm oil and starch.

Keywords: biodegradation, bioplastics, biopolymers, LDPE, thermoplastic starch.

1. INTRODUCTION

The bulk population of plastics is synthetic, and they pose danger to human health and the environment when disposed of, because of land and water pollution. Plastics are commonly used because of their properties which make them look indestructible and irreplaceable [1]. Plastic products are widely accepted across the globe, and this makes the production of Bioplastics very vital. There are two types of bioplastics and are bio-based and biodegradable plastic [2]. Bio-based plastics can be sourced totally or partially renewably. Biodegradable plastics are those that decompose through microbial activity into water, carbon dioxide, methane, and biomass over a specific period and in several environments. However, some bioplastics and degradable plastics are not biodegradable [3]. Although bioplastics account for a lower portion of whole plastic manufacturing, this industry is growing. According to Jeremic et al., (2020) [4] approximately half of all bioplastics on the market (44.5 percent in 2019) are bio-based but not biodegradable. A significant proportion of these so-called drop-in materials are bio-PE, bio-based PET, and bio-based PTT. Lackner, (2015) [5] postulated that bioplastics are built on two sustainability concepts which are biodegradability and renewability. Biodegradability is the ability of bioplastics to biodegrade into CO₂ and H₂O within the environment without causing harm to marine organisms. Also,

renewability in this context refers to the use of renewable feedstocks such as corn, cassava, potato, etc., rather than petroleum reduces the reliance on crude oil while also lowering overall climate impact.

Bioplastics are approximately comparable to their fossil-based counterparts, making them suitable for industry. Aside from Polylactic Acid (PLA), which accounts for 14 percent of global production capacity for bioplastics (biodegradable and non-biodegradable), primarily starch blends (21 percent), other biodegradable polyesters such as Polybutylene succinate PBS (4 percent) and Polyhydroxyalkanoates, PHAs (1 percent) are produced on an industrial scale [4, 17]. In the biodegradable plastic industry, PLA presently has the most market share and production. PLA has excellent optical transparency due to its amorphous nature, which is very useful in the field of food packing. However, its barrier qualities are poor, and its high oxygen/water vapor permeability limits its use.

The poor barrier characteristics of amorphous PLA can be improved by converting it to mainly crystalline form [6]. Over the last two decades, well over 150 different types of poly (hydroxyl alkanoic acid) (PHAs) produced by various bacteria have been discovered and reported. Most of them have only been researched and produced on a laboratory scale [7]. PBS, a biodegradable polymer derived from succinic acid and



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1,4-butanediol, has an oxygen/water vapor barrier range comparable to PLA. According to Yusoff *et al.*, (2021) [8] Polylactic acid (PLA) was mixed with tapioca starch (TS) as a composite material to study the impact of tapioca starch on the mechanical properties of bioplastics. As a result of this research, material for food packaging with temperature restrictions and applications below 160°C was established. The tensile strength improved significantly when tapioca starch was added. At 30 wt. % loading, the highest reading was obtained, and it gradually decreased with further addition of TS.

In recent years, biodegradable alternatives to petroleum-based polymers in food packaging have been the subject of research. Biopolymers are the most hopeful for this application because of their material biodegradability and prolonged storage life properties which includes resistance to chemical and enzymatic activity. Starch is an arenewable biopolymer, an inexpensive, and easily accessible material. [18-19]. Starch cannot be considered a thermoplastic material due to van der Waals forces interactions and hydrogen bonding. Several plasticizers are used to make thermoplastic starches, which are thermoplastic materials that deform. Below the breakdown temperature, a plasticizer improves starch's elasticity and process stability. The glass transition temperature is lowered by using plasticizers [9]. Since native starch is brittle and hydrophilic, it is inappropriate for a collection of uses, including plastic bags and food packaging. It has poor heat resilience due to its high fusion point and low thermal temperature malfunction. However, with the help of a plasticizer and the combined effects of temperature and shear, starch can be transformed into thermoplastic [10].

2. MATERIALS AND METHODOLOGY

These composite materials consist of proportions of cassava starch along with other materials namely lowdensity polyethylene (polymer), plasticizer (glycerol, palm oil), and distilled water. Starch is a renewable material available from various crops. Most renewable materials are degradable, and therefore, to retain their mechanical plastic properties, Low-density polyethylene will remain in primary proportion in the composite. In nature, starch is hydrophilic (it has a great attraction to water), whereas LDPE is hydrophobic (it has no affinity for water).To create a blend/mix, a substance called a plasticizer is needed. Distilled water is used to dilute the plasticizer quantity.

2.1 Preparation of Cassava (Manihot Esculentum) Starch

The cassava tubers utilized in this experimental research were obtained from the Sango Ota market in Ogun State and brought to Covenant University, Department of Mechanical Engineering in Ota, Nigeria. To remove soil and dirt from the cassava tubers they were rinsed in distilled water and peeled. The cassava peels are mixed to make a smooth mush. The starch was extracted from the mash using a starch extractor (see Figure-1a); with the use of a sieve for the separation of the thick slurry paste from the supernatant fluid. This process is called sedimentation. The supernatant fluid was drained out. The cassava paste was baked in an oven at40°C to create the dry starch powder [11, 20].

2.2 Cassava Sheet Plastic Blend Production

All compositions were measured with the OHAUS weighing scale. Each sample net weight for all the compositions was a hundred grams (100 g). 10 millimetres of glycerol was diluted in 1000 millimetres of purified water producing glycerol of one percent concentration. The final sample weight was made up of 30% glycerol, low-density polyethylene, and cassava starch (6g of glycerol). Other studies used 6g of palm oil combine the LDPE/starch combinations. to The LDPE/starch combination was poured into the 250 ml glass beakers and melted at a temperature of $105^{\circ} \pm 2^{\circ}C$ for six minutes in the reported study [12, 21]. Under the intense heat created by the electric cooking stove, it was properly blended using the 5-speed sabichi electric hand mixer to produce a homogenous mixture of liquid viscous. The molten (liquid) composite blend was instantly poured out onto an aluminum sheet and coated with aluminum foil before quickly being rolled into thin plates before the molten composite hardened [13]. The aluminium plates/sheets were coated with a thin layer of glycerol which acted as a lubricant. After solidification, the produced composite materials were sections into different dimensions for further tests. The composite materials were subject to the same procedure, as depicted in Table 1 for the various starch sources. The LDPE starch mix is shown in Figure-1b.



Figure-1a. Cassava starch sample.





Figure-1b. LDPE/CaS blend sample.

Table-1. LDPE/Cassava Starch Formulations.

Cassava starch	LDPE (wt.%)
5	95
10	90
15	85
20	80

2.3 Characterization Result of Samples (Cassava Blend)

Alongside *degradation by mass technique*, another analysis was carried out on the samples namely, microhardness, compression, tensile, thickness analyses, and micro structural characterization. An Instron 3380 universal testing equipment was used to perform compression tests following ASTM E2954. Compressive and tensile characteristics were determined using two samples of each combination. The thickness of each composite sample set was measured using a micrometer scale manufactured by Mitutoyo. The composite sample was placed between their inside teeth until the readings stabilized. The SEM/EDS analyses were carried out using a field emission JSM-7600F (Schottky model) equipped with energy-dispersive X-ray spectroscopy.

3. RESULTS AND DISCUSSIONS

3.1 Mechanical Properties of Composite Samples: (Cassava Blend)

A. Results from compression analysis

The results of compression tests are used to calculate the maximum compressive strength and applied loads.

Table-2 shows the result for the 100 wt.% LDPE sample, followed by the data for the blends.

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Weight % of starch/LDPE	Compressive strength (MPa)	Load at compressive strength (N)
0/100	0.209	9.648

Table-3 demonstrates that the 20/80 wt.% CaS/LDPE mix had the highest compressive strength and load at maximum compressive strength when evaluated to the other blends. A proportional trend was revealed as an increase in powder content raised the maximum compressive strength as well as the loads at maximum compressive strength.

Table-3. Compressive strength result.

Weight % of starch/LDPE	Compressive strength (MPa)	Load at maximum compressive strength (N)
5/95	0.022	1.279
10/90	0.028	1.617
15/85	0.034	1.954
20/80	0.063	2.871

It was observed in Table-4 that the 5/95 wt.% CaS/LDPE blend had higher values for both maximum compressive strength, and load at maximum compressive strength when related to the other blends. The maximum compressive strength is reduced as the starch content increase, and this indicates an inversely proportional trend. The same was the circumstance for the loads at maximum stress. With increasing starch content, the load required to generate maximal stress decreased.

 Table-4. Compressive strength for palm oil plasticized

 CaS /LDPE blends.

Weight % of starch/LDPE	Compressive strength (MPa)	Load at maximum compressive strength (N)
5/95	0.122	6.941
10/90	0.076	4.347
15/85	0.031	1.752
20/80	0.026	0.892

B. Results from tensile evaluation

The maximum tensile strength and applied loads at maximum tensile strength are the factors considered in the tensile test findings.

Table-5 shows the 100 wt.% LDPE sample, followed by the results of the mixed samples.

Weight % of starch/LDPE	Tensile strength (MPa)	Load at maximum tensile strength (N)
0/100	1.385	76.360

Table-5. Tensile result.

Table-6 shows that the 20/80 wt.% CaS/LDPE blend had the highest values for both maximum tensile strength, and load at maximum tensile strength compared to the other blends. The maximum tensile strength increased with increasing starch content, showing a proportional trend, and the same could be said for the loads that were subjected to the most stress. The load needed to produce maximum stress reduced as starch content increased.

 Table-6. Tensile result for Glycerol-plasticized

 CaS/LDPE blends.

Weight % of Starch/LDPE	Tensile strength (MPa)	Load at maximum tensile strength (N)
5/95	0.216	15.329
10/90	0.349	19.882
15/85	0.670	38.212
20/80	0.992	56.542

Table-7 revealed that the 5/95 wt.% CaS/LDPE blend had the highest values for both maximum tensile strength, and load at maximum tensile strength compared to the other blends. The maximum tensile strength decreased as the starch content increased, following an inversely proportional trend, as did the loads at maximum stress. The load needed to produce maximum stress reduced as starch content increased.

Table-7. Tensile result for palm oil plasticizedCaS/LDPE blends.

Weight % of Starch/LDPE	Tensile strength (MPa)	Load at maximum tensile strength (N)
5/95	0.944	50.39
10/90	0.704	40.12
15/85	0.463	27.43
20/80	0.223	12.68

3.2 Characterization Results of Samples: Chemical Properties

A. Results from energy dispersive X-ray Spectroscopy

The EDX graphs below indicate the chemical and weight % of each constituent element with peak levels where they are detected. Figure-2 shows that the elements found in the control sample - Starch/LDPE (0/100) were oxygen, carbon, nitrogen, calcium, sodium, sulphur, and

chlorine. The element with the highest weight was carbon, at 50.95%.



Figure-2. EDX graph showing 100 wt.% of LDPE.

There was no exclusive element in the CaS-G samples in Figures 3 and 4, and the mutual elements found in the samples were oxygen, carbon, phosphorus, calcium, sodium, nitrogen, potassium, and sulphur. The element with the highest weight was carbon, at 91.64% in the CaS-G sample, and 83.51% in the CaS-P sample.



Figure-3. EDS graph showing 10/90 wt.% of glycerolplasticized cassava starch/LDPE.







Figure-Fehler! Kein Text mit angegebener Formatvorlage im Dokument.. EDX graph showing 10/90 wt.% of palm oil-plasticized cassava starch/LDPE.

B. Microstructural characteristics of polymer surfaces

Figure-5a depicts a micrograph of an LDPE sample with homogenous characteristics. Figures 5 (b and c) illustrate SEMmicrographs of the 10/90 glycerolplasticized cassava starch/LDPE blend and 10/90 palm oilplasticized cassava starch/LDPE blend, respectively, and demonstrate no growths in the sample. Larger grain sizes can be seen in the blend in Figure-5b which was plasticized by glycerol than in Figure-5c which was plasticized by palm oil. In the latter, the microstructure has a threadlike and dendritic appearance showing inter-matrix growth [14-15]. The grains are more abundant and much smaller, showing less of a plasticizing effect in the blend with palm oil. Therefore, it can be concluded that the glycerol had a better effect on the surface morphologies of the blends than the palm oil [16, 22-23]. Tables 8 and 9 show the average thickness for the formulated blends for glycerol-plasticized blends and palm oil-plasticized blends respectively.



Figure-5. Scanning electron microscopy images (a) 100 wt.% LDPE,(b) 10/90 wt.% glycerol- plasticized cassava starch/LDPE blend and (c) 10/90 wt.% palm oil-plasticized cassava starch/LDPE blend.

Table-8. Thickness values for glycerol-plasticiz
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	95	90	85	80	100
Cassava	1.54	1.04	1.18	1.23	1.56

Table-9. Thickness values for palm oil-plasticized.

	95	90	85	80	100
Cassava	0.90	0.79	1.01	1.12	1.56

3.3 Biodegradation Analysis

The composite samples were split based on the plasticizers used, then buried for three (3) weeks, and taken out weekly for measurement. Every time the samples were dug up, they were cleansed of sand and other debris to avoid measurement errors. The samples were then weighed and documented. Tables 10 to 11 show the weight values at various times during the burial period.

A. Degradation weights of blended glycerol buried in



sandy-loamy soil

Weeks	95	90	85	80	100
Start	0.318	0.367	0.254	0.353	0.297
1	0.311	0.354	0.241	0.345	0.297
2	0.301	0.321	0.232	0.307	0.297
3	0.288	0.301	0.221	0.289	0.297

Table-10. Shows the biodegradation of LDPE/cassava starch blend.

B. Degradation weights of palm oil blends buried in sandy-loamy soil

		-			
Weeks	95	90	85	80	100
Start	0.498	0.339	0.422	0.527	0.297
1	0.492	0.334	0.396	0.523	0.297
2	0.489	0.314	0.298	0.518	0.297
3	0.479	0.302	0.200	0.513	0.297

Table-11. Shows the biodegradation of LDPE/cassava starch blend.

3.4 Percentage Weight Loss

The values of the wt. % lossare obtained from the equation below for comparison of degradation of the sample.

Percentageweight
$$\frac{\text{loss}}{\text{degradation}} = \frac{\text{Wi} - \text{Wc}}{\text{Wi}} X \, 100$$

Where Wi = Initial Weight Wc = Current Weight

A. Weight reduction % of combined glycerolplasticized buried in sandy-loamy soil

The degradation of glycerol LDPE/Cassava starch (CaS) blends is shown in Table-12 and Figure-6. 95% of the LDPE has degraded by 2.26%, 5.44%, and 9.5% after 7, 14, and 28 days respectively. These show an almost linear decline, with a high value of 9.5 percent. Thus, weeks 1 and 2 have a value of 9.5 percent and a substantial percentage weight reduction.

As indicated in the graph, 90% of LDPE had the largest and most consistent degradation throughout the burial stage, with 3.7%, 12.55%, and 18.05% degradation during weeks 2 and 3. This indicates a high percentage of weight loss at its peak. Small holes and cracks, as well as defragmentation, were discovered, indicating the presence of microbial activity in the burial site [23-25]. The linear degradation value of 85%-LDPE increased with time, peaking at 13.04%. 80% -LDPE showed the same degradation as 90%-LDPE alongside its observations.

Table-12. Weight reduction of glycerol LDPE/cassava blends degradation.

Weeks					
	95	90	85	80	100
1	2.26	3.70	5.24	2.23	0.00
2	5.44	12.55	8.71	13.06	0.00
3	9.50	18.05	13.04	18.18	0.00





Figure-6. Biodegradation of glycerol LDPE/cassava blends.

B. Weight reduction % of combined palm oilplasticized buried in sandy-loamy soil

The graph depicts that 95% of the LDPE degraded by 1.22%, 1.81%, and 3.92% after 7, 14, and 28 days respectively, as shown in Figure-7. This shows a nearly linear decline, with a high value of 3.92 percent and a maximum percentage weight reduction between weeks 1 and 2.

From indication, 90%-LDPE degraded constantly the most, all through the burial period; 3.7%, 12.55%, and 18.05%. The evidence of slight hoes and cracks, and

defragmentation shows percentage weight loss was at its peak for the period of weeks 2 and 3. This is the initial suggestion of microbial activity in the burial ground.85 %-LDPE has a fast-growing linear degradation value with a high of 52.57 %. This occurred because of regular defragmentation, which occurred every seven days and demonstrated significant microbial activity in the soil. [26-28]. Aside from its excessive microbial activity, which was visible as holes on the samples, however, the degradation of 80% -LDPE was related to that of 95%-LDPE.

Table-13. Weight reduction of palm oil LDPE/cassava blend degradation.

Weeks					
	95	90	85	80	100
1	1.22	1.71	6.02	0.85	0.00
2	1.81	7.66	29.26	1.80	0.00
3	3.92	11.19	52.57	2.69	0.00



Figure-7. Biodegradation of palm oil LDPE/Cassava Blends.

CONCLUSIONS

The advent of bioplastics is key to the presentday mitigation of pollution from plastic all over the world, which also leads to achieving better sustainable production and consumption. In this work, cassava starch was used as a biodegradable material, whereas glycerol and palm oil were used as plasticizers. Glycerol was employed in the compressive strength investigation, and it was discovered that when the starch content increased from 0.02245 to 0.03428, the maximum compressive strength increased proportionally. In addition, when the starch content rose from 1.27983 to 1.95387, the load necessary to cause maximal stress increased. The maximum compressive strength in palm oil decreased as the starch concentration increased from 0.12177 to 0.03074, indicating an inversely proportional relationship. When the starch content was increased from 6.94105 to 1.75211, the load at maximum strength decreased. Also, when the starch concentration

grew, the maximum tensile strength increased from 0.34881 MPa to 0.99196 when glycerol MPa was used to investigate the tensile strength showing a proportional trend. This demonstrated the loads at maximum strength, which ranged from 19.88216 N to 56.54196 N. As the starch concentration increased, the maximum tensile strength decreased from 0.70383 MPa to 0.02225 MPa when palm oil was used. This tendency is inversely proportional. As starch content increased, the load needed to attain maximum strength was reduced from 40.12 N to 12.68 N.

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