

Preparation and Evaluation of Biodegradation Performance of Potato Starch Mixed LDPE Polymer Composites

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Authors' contributions

This work was carried out in collaboration among all authors. Author SD wrote the protocol and the first draft of the manuscript. The lab work was carried out by authors SD and LN. Authors SP and SD managed the literature searches and analysis of the study. Author MYM designed the study and was the academic supervisor for the work. All authors read and approved the final manuscript.

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ABSTRACT

The potential of biodegradable polymers has long been appreciated. In this work, an attempt has been made to synthesize biodegradable polymer composite from potato starch and low density polyethylene (LDPE) with different ratios of starch (0%-15% w/w) by using an extruder. The structure and morphology of film surfaces was studied using X-ray diffraction and scanning electron microscopy (SEM) respectively. The physical (density, water absorption), mechanical (tensile, flexural) and thermal (TGA and DTA) analyses of starch/LDPE bio-composites were evaluated through standard methods and instruments. Biodegradation tendency was investigated utilizing soil burial and Rockwell micro-hardness test. The results revealed that the density and water absorption of polymer blends increased with increasing the starch content in starch/LDPE composites. The tensile strength and elongation at break decreased with starch content whereas the elastic modulus, flexural strength and flexural modulus rose. The biodegradability of composites enhanced by increasing the starch content and the result was backed by weight loss

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during burial of the samples in soil for 90 days. Microhardness test also supported the biodegradation probability as hardness found to reduce extensively after soil burial. However, further study to decide the optimum starch loading alongwith some modifications to starch and LDPE is highly suggested to have a biodegradable LDPE polymer composite in a realistic time frame.

Keywords: LDPE; starch; biocomposite; biodegradable polymer; soil burial; micro-hardness.

1. INTRODUCTION

Polyethylene (PE) is one of the largely produced non-degradable polymers with extensive usage in various fields such as packaging materials in food and pharmaceutical industries, disposable containers, agriculture mulch films, composites, and carrier bags [1]. Various types of polyethylene such as high-density PE (HDPE), low-density PE (LDPE), or linear low-density PE (LLDPE) have several advantages and applications [2]. Because of such widespread uses and non-biodegradability, they cause environmental problems when disposed, submerged in the sea, or dumped in the riverside soil. To overcome these hurdles associated with disposal of synthetic plastics, plastic degradability has been considered as a prominent approach. Making PE biodegradable need to improve its crystalline level, molecular weight and mechanical characteristics that are accountable for PE defiance towards degradation. This can be obtained by developing PE hydrophilic level and/or shortening its polymer chains length by oxidation to be approachable for microbial degradation [3]. Although there are several methods like ultraviolet light, heat, or oxygen to start the oxidation of PE, there is actually only one method to sustain it. That is mixing specific additives such as starch to the PE which may help to enhance PE chain oxidation reactions; microbial consumption of starch also raises the PE surface area by generating pores and improves oxygen based reaction [3].

Starch is a hydrocolloid biopolymer that can be obtained from a large number of agricultural feedstocks such as wheat, corn, rice, beans, and potatoes [2]. It is natural polymer consisting of repeating 1-4- α -D glucopyranosyl units (often called anhydroglucose units) and a mixture of linear and branched components amylose and amylopectin [2,4]. The linear constituent, amylose, is the minor component, generally ranging from 20 to 30%, is of a shape in the form of a helix [3]. The branched component, amylopectin, is the major component, whose

average chain length is 20–30 glucose units, and the branching in amylopectin occurs at C-6 of the glucopyranosyl units [4].

The incorporation of starch to various synthetic plastics like poly (vinyl alcohol) [5], high density polyethylene [6] and linear lowdensity polyethylene [7-9] has been known to increase their biodegradability. Some investigations have also been done to develop biodegradable polymer by mixing starch of various sources with LDPE [1-4,10-14]. The mechanical characteristics and biodegradation studies of prepared starch-LDPE blends have revealed improved biodegradation of LDPE on addition of starch. Mixing of dry starch to low density polyethylene (LDPE) follows a common trend of filler effect on polymer characteristics [4].

In this research, we have developed various composite blends incorporating small amount of potato starch into LDPE matrix and investigated several physico-mechanical properties that can explain the possible reason behind biodegradability of starch/LDPE blends.

2. EXPERIMENTAL DETAILS

2.1 Materials

Low-density polyethylene (LDPE) and potato starch were collected from Pilot Plant & Product Development Center, Bangladesh Council of Scientific and Industrial Research (BCSIR), Dhaka.

2.2 Polymer Sample Preparation

Four types of LDPE polymer samples were prepared in combination with various percentages of starch following the method mentioned by these literatures [2,14]. LDPE and starch were weighed by an electric balance (KERN, RH120-3, Max 120gm) with various percentages of LDPE and starch as reported in Table 1. At first, the starch was kept in vacuum oven at 80° C for at least 24 hrs to keep the starch powder dry. Then, LDPE and starch were

Table 1. Relative percentage and weight of starch and LDPE in starch/LDPE polymer samples

Starch content (%)	LDPE content (%)	Amount of starch in (gm)	Amount of LDPE in (gm)
0	100	0	60
5	95	3	57
10	90	6	54
15	85	9	51

fed into the extruder (Dynisco, Japan, Orifice 1/8" Stand Header, 30rpm) through a feed hopper. Extrusion temperature and screw speed was 170° C and 30 rpm respectively and extruded strands of starch/LDPE composite were found through the orifice of the extruder. The extruded strands of starch/LDPE composite were chopped into small pieces (about 10 mm). After that, starch/LDPE strands were placed into a square shaped stainless steel frame (height: 2 cm, diameter: 8 mm). Polyethylene terephthalate (PET) sheets were used in between polymer surface and steel plate to prevent clinging of the polymer samples to the steel plate. And later were heated up to 150° C temperature under pressure of 50 kN for 1-2 min. and allowed to cool at 30° C temperature. Cooling was done by tap water through the outer area of heat plates of the Paul-Otto Weber machine and the cooling time was around 11-15 min.

2.3 Shape Preparation

Dumb-Bell-Shaped Cutting. Upon finishing of synthesis process, the polymer samples were cut into dumb-bell-shaped by a dumb-bell cutter (model SDL-100; Dumb-Bell Co. Ltd.). The dumb-bell-shaped samples had smooth surface specifically in the neck section to avoid stress concentration during mechanical tests.

2.4 Characterization

2.4.1 Bulk density

The density of the synthesized composite is obtained by dividing its mass by its volume following the procedure mentioned here [14]. Three different density analyses were performed for each sample. Samples were taken of approximately same weight (about 2 gm) and measured by a digital weighing balance with four decimal.

2.4.2 Water absorption property

Water absorption test was carried out according to the method mentioned in this report [14]. The samples were dried for 6 hours at 50° C. Then,

they were cooled and weighed. The samples were soaked in distilled water at 23±1° C temperature for 30 days. The samples were periodically weighed every 2 days to record any change in their weights until an equilibrium state is observed. The percentage of the water absorption by composite was then calculated using the following formula [12]:

$$\text{Water absorption, } W_f = \frac{W_m - W_n}{W_m} \times 100 \%$$

Where, W_f is the final increased weight percentage, W_m is the wet weight and W_n is the conditioned weight of the testing samples respectively.

2.4.3 Morphology analysis

The developed bio-composite samples were characterized for their structural and nanostructure properties. The structures of powder starch, pure LDPE and starch/LDPE composites were investigated by using X-ray diffractometer (Ultima IV, Rigaku Corporation, Japan) with Cu-K α radiations ($\lambda = 1.5406 \text{ \AA}$) in 2 θ range from 10° to 90° operated at a voltage of 40 kV and current 40 mA with scan speed of 3°/min. The same procedure of literature is followed here [3]. Surface morphology was investigated using scanning electron microscopy, SEM (JEOL JSM-6490LA, Tokyo, Japan) following the mentioned method here [4,13].

2.4.4 Mechanical properties

Tensile test was carried out to investigate the influence of starch content on LDPE. Tensile test of the composite specimens were carried out using UTM (Universal Testing Machine, Canver. INC.USA, Wobash, Indiana as per ASTM-D 638 standard) [1] at test speed 100 mm/min, gauge length 50 mm. Flexural test was done by ASTM-D 790 standard [1], at test speed 2.8 mm/min, gauge length 100 mm respectively. Tensile and flexural specimens were dumb-bell and rectangular in shape respectively.

2.4.5 Thermal properties

For thermo-gravimetric analysis, TGA (TGA-50, Shimadzu, Japan) of the prepared samples the thermograms were recorded under nitrogen atmosphere at a purge rate of 200 ml/min in an alumina cell at a temperature rate of 10° C/min with the temperature range being 25° C to 800° C. TGA analysis was done according to the adopted procedure of the study [12].

2.4.6 Biodegradability study (Soil-Burial Test)

The in vitro degradation study was conducted on the dumb-bell-shaped LDPE polymer samples having various percentages of starch (0 %, 5 %, 10 %, and 15 %) by burying the polymer samples at the exterior under the soil at a depth of approximately 10 cm. The samples were removed after 90 days and washed well with demineralized water and dried at 40° C – 45° C in a vacuum oven for 24 hrs. Then, weight of each sample was measured and the weight loss due to biodegradation was observed for duration of 90 days. This study was done primarily to study the variation of degradation kinetics because of change in starch content. The degradation phenomenon was measured through mass loss and change in surface morphology. Prior to burial, the polymer samples mass were weighed and recorded using an electronic balance. After three months of burial, the samples were removed and washed with demineralized water to remove soil/mud. Samples were then kept in an area with adequate ventilation for natural drying. The dried degraded samples were again weighed using the same electronic balance as was done before starting degradation. Consequently, the

percentage of mass loss of respective sample was measured as follows [2,13]:

$$\% \text{ Mass loss, } = \frac{M_1 - M_2}{M_2} \times 100$$

Where, M_1 is the mass before degradation and M_2 is the mass after degradation respectively.

2.4.7 Micro-hardness test (Rockwell hardness):

The results of a hardness test can be utilized to give crucial material activity information and understanding to the durability, strength, flexibility, and performances of various constituent types from starting materials to synthesized samples, and end products. Digital Rockwell hardness tester with HRL indenter is used to determine the micro hardness value of the synthesized starch/LDPE composite samples before and after soil burial for 90 days. The test was done following ASTM D 785 standard [15].

3. RESULTS AND DISCUSSION

3.1 Bulk Density

The effects of starch loading on bulk density of LDPE are shown in below Fig. (Fig. 1). The Figure shows that the bulk density increases with the increase of starch loading. It may be because rising of fillers concentration create an improved interfacial bonding between LDPE and starch which leads to increased density [14]. And, the lower density values were assigned to the existence of void within filled composites.

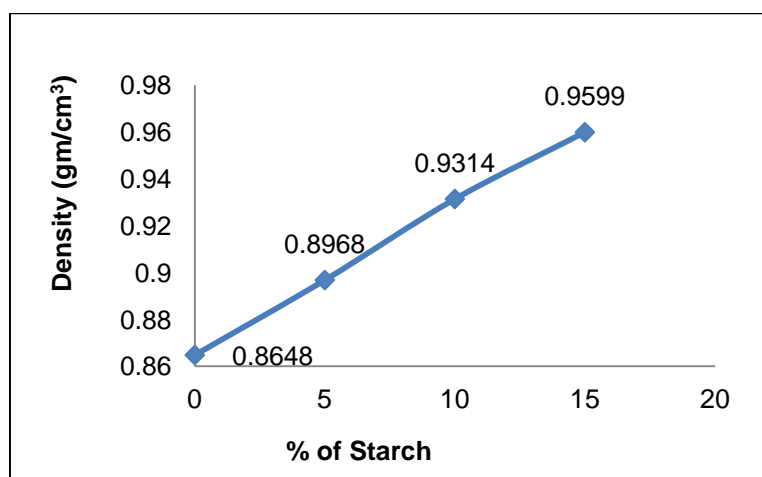


Fig. 1. Bulk density of starch/LDPE bio-composites

3.2 Water Absorption

Below figure (Fig. 2) reveals that, pure LDPE shows no water absorption while it rises with raising potato starch content. The absorption of water is linked to its rate of diffusion into the composites. Starch based synthetic materials prone to absorb water because the hydroxyl group of starch can form a hydrogen bond with water [14]. Since starch is hydrophilic in nature, it has a high tendency to attract water molecules [12,14].

3.3 Morphology Analysis

It is important to study the structure and morphology of the polymer blends since the mechanical properties depend on it. In these blends, the major component (LDPE) forms the matrix and the minor component (starch) forms the dispersed phase. The degree of crystallinity of pure LDPE, pure starch and 10% starch blended 90% LDPE was determined by X-ray diffraction. The XRD spectra (Fig. 3) of sharp

distinguished peaks at 21.18 and 23.7 of the angular position 2θ are of pure LDPE [3]. However, XRD pattern of the 10% starch/LDPE blend gives peaks at the same angular position 2θ of pure LDPE but of lower intensity (Fig. 3). This clarifies that the crystal sizes of starch/LDPE blends plummets due to starch incorporation into LDPE matrix.

Scanning electron micrographs of 100% LDPE, 5% starch, 10% starch and 15% starch loaded LDPE blends are shown below (Fig. 4. a-d). It can be seen that, the distribution of starch on LDPE matrix is more visible with increasing amount of potato starch in composites. Flat and smooth surface of LDPE (Fig. 4. a) is affected by powdered starch addition (Fig. 4. b-d). Uniform distribution of powdered starch did not happen in the composites rather lumps are found here and there throughout the surfaces (Fig. 4. d). This is due to lack of compatibility between hydrophilic biopolymer starch and the typically hydrophobic thermoplastic matrix LDPE [4].

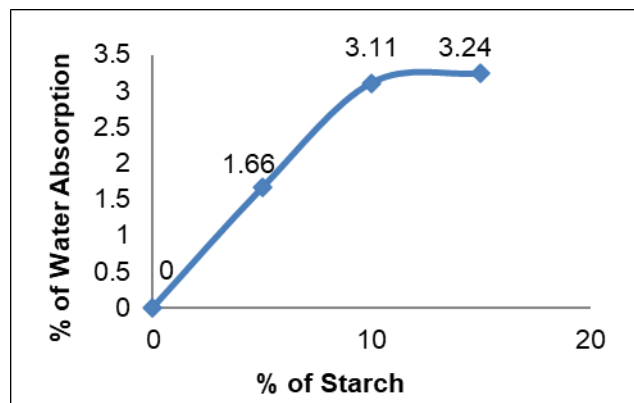


Fig. 2. Percentage of water absorption of starch/LDPE bio-composites

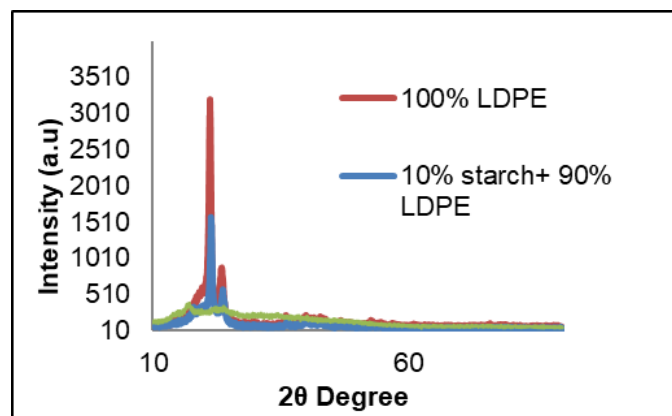


Fig. 3. XRD of starch/LDPE bio-composites

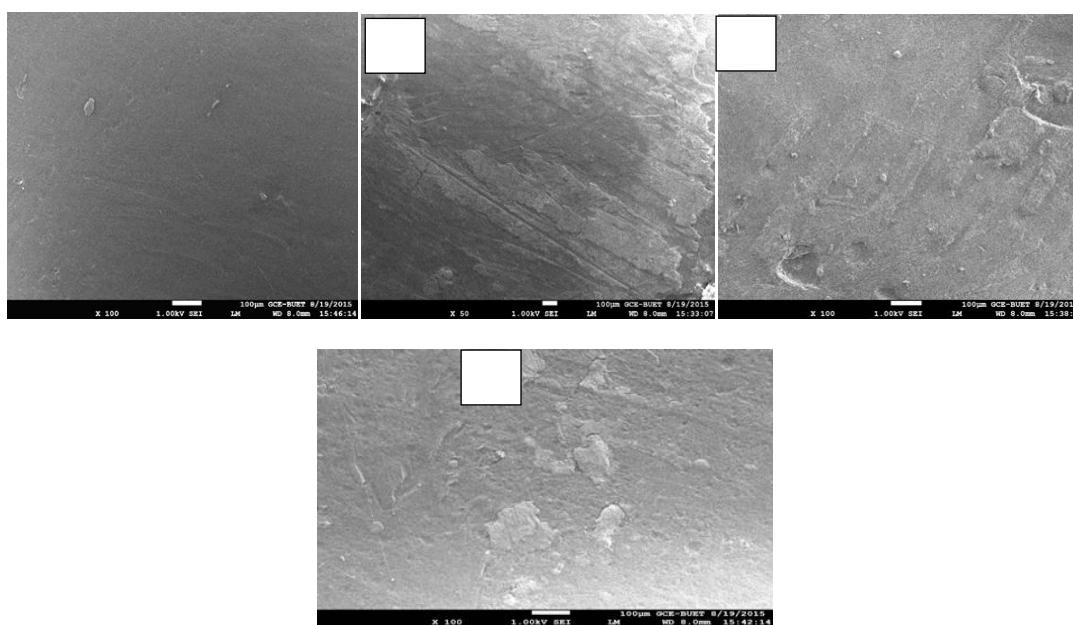


Fig. 4. SEM images of starch/LDPE bio-composites; (a) 0% starch, (b) 5% starch, (c) 10% starch and (d) 15% starch

3.4 Mechanical Properties

3.4.1 Tensile strength

The mechanical experiment (tensile test) primarily investigated the effect of starch content on mechanical property of LDPE. Tensile strength decreases with increase in potato starch concentration (Fig. 5 .a). The tensile strength which fell with the addition of starch content can be assigned to the phenomenon that the starch granule containing hydroxyl groups on its surface is highly hydrophilic, whereas LDPE is nonpolar. In such case, strong interfacial bond (e.g., hydrogen bond) does not occur between LDPE and starch. Also, the presence/absorption of moisture by the starch at the starch/LDPE interface weakens the interfacial adhesion. Thus, tensile strength gets reduced. Besides, it was found that the samples have tendency to absorb water. Thus, it may cause the possibility of decrease in tensile strength because of the presence of moisture at the starch LDPE interface [14].

LDPE bio-composites of variable fiber content illustrate considerable changes in their elastic modulus which is a proof of stiffness and resistance to stress (Fig. 5. b). Unlike tensile strength, elastic modulus rises as starch content increases. This is because filler-filler interaction becomes more distinct than filler-matrix interaction when filler content increases. As the

starch contents rises, particles are agglomerated and reduced the particle-matrix interaction. The films became more rigid and stiffer than films without starch as starch granules are stiffer than the LDPE matrix [2,14]. Rigid filler is typically supposed to elevate the modulus of a blend even in the case where actual reinforcement does not happen [2]. Therefore, the rise in modulus of the polymer blend with the rise of starch content is suggested to be due to the higher stiffness of the starch granules.

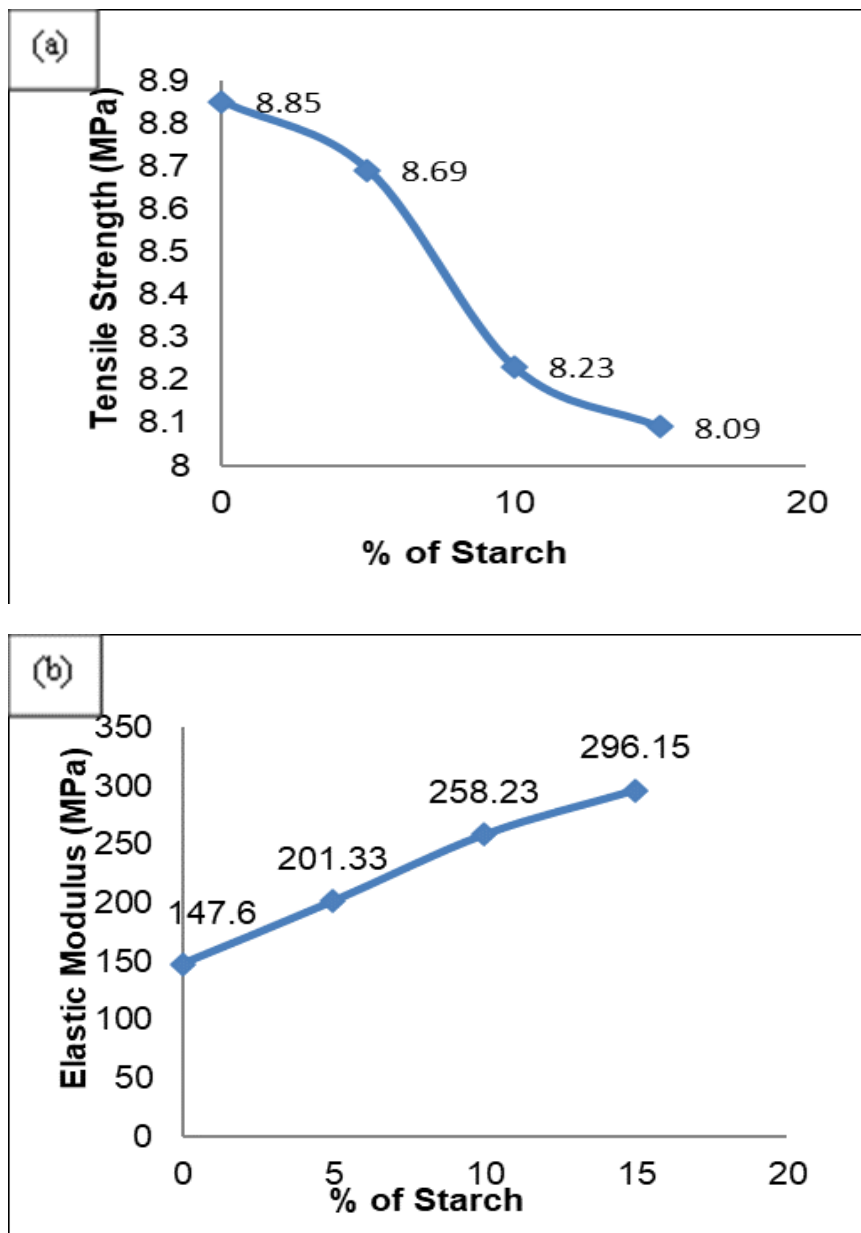
The changes in elongation with increase in starch content are shown below (Fig. 5. c). It is noticeable that elongation falls with the rise in potato starch content. Similar to tensile strength, decreasing of elongation is because of the weakness of interfacial adhesion between starch and LDPE. In synthetic polymer blends, the incorporation of the immiscible element to a ductile matrix normally reduces the elongation characteristics at break [14]. Therefore, the starch granules do not elongate along with the LDPE and thus give rise to easy crack initiation and/or propagation finally causing fracture of the polymer sample at lower values of elongation [2]. Also, the more noticeable filler-filler interaction reduces the effective cross-sectional area of the polymer sample caused by the existence of starch particles. The applied stress is not thus transferred thoroughly from the polymer matrix to the rigid starch particles, and hence the effective

stress felt by the matrix is necessarily larger [2,14].

3.4.2 Flexural strength

The flexural strength and flexural modulus of all starch/LDPE blends are higher than native LDPE (Fig. 6). For pure LDPE the flexural strength is 11.26 MPa and 5%, 10% & 15% starch loaded LDPE composites show rise in flexural strength with the addition of starch (Fig. 6. a). Maximum flexural strength is observed in case of 15% starch with 14.5 MPa. And, the flexural modulus of pure LDPE is 278.58 MPa whereas for 5%, 10%, and 15% starch it is observed 322.68 MPa,

251.73 MPa and 346.25 MPa respectively (Fig. 6. b). The increase in flexural strength with corresponding increase in starch content is because it may act like a binder which stiffens the elasticity of the polymer matrix and increase the ability of the composite to absorb and dissipate energy. It was recommended that the weak interfacial bonding between starch and LDPE matrix cause disorientation of void and dispersion in starch/LDPE blends, resulting in reduced degree of molecular alignment of composite [16]. Our findings are in agreement with findings of other works [14,16].



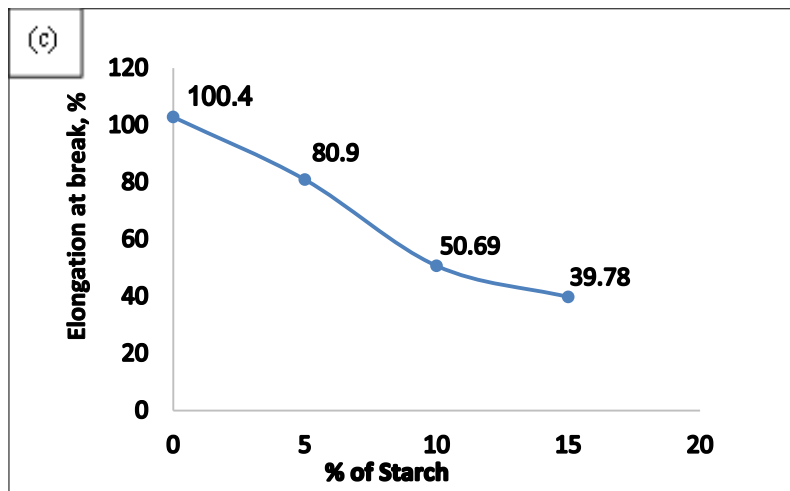


Fig. 5. Tensile properties of starch/LDPE bio-composites; (a) tensile strength, (b) elastic modulus and (c) elongation at break

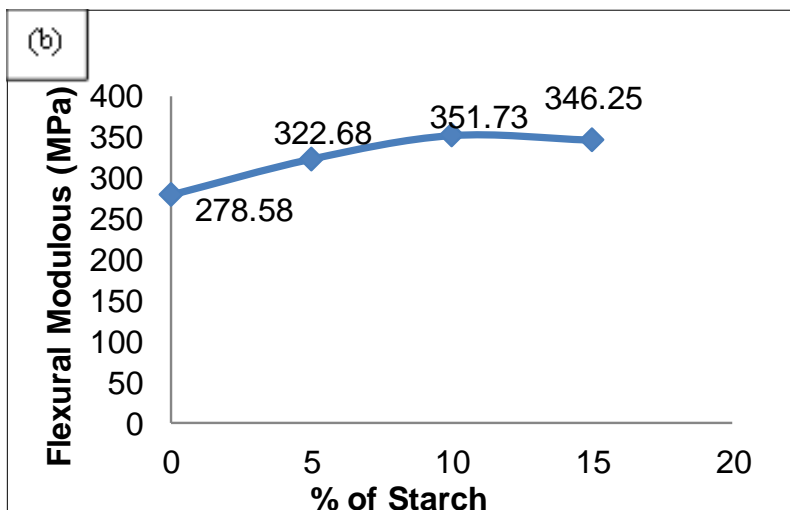
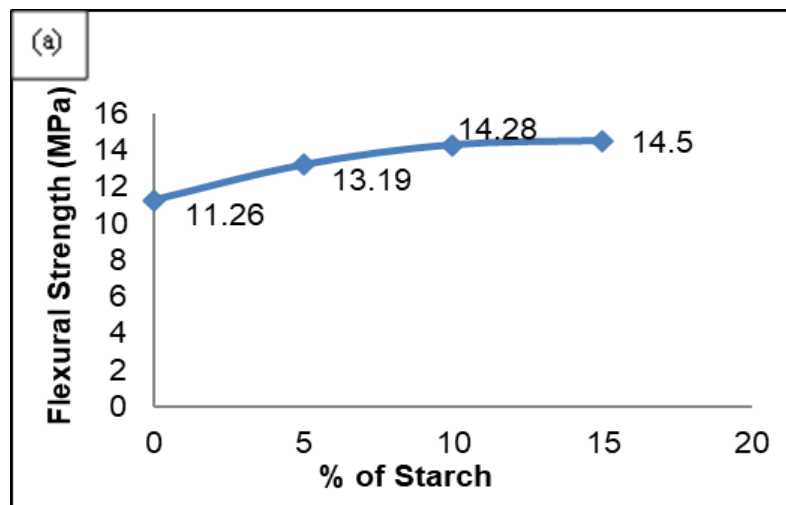


Fig. 6. Flexural properties of starch/LDPE bio-composites; (a) flexural strength, (b) flexural modulus

3.5 Thermal Analysis

The effect of the potato starch contents on the thermal degradation behavior of starch/LDPE bio-composites is shown in the following figure (Fig. 7. left). It could be seen that the initial decomposition temperature of the composite blends reduced with increasing potato starch concentration. Admittedly, the starch/LDPE blends degraded in two stages. The first one around 300°-340° C was due to starch decomposition [4]. And in the second stage, also known as main stage, thermal degradation observed at around 450° C, linked to the highest weight loss, is due to thermal degradation of the LDPE backbone [4,14]. This higher thermal decomposition is because of the starch content. It is observed that less than 5% residue is left at 510° C.

The DTA curve (Fig. 7. right) shows three endothermic peaks for all composite blends due to moisture loss, melting point of starch and LDPE polymer degradation respectively. Also, it is found that the endothermic peaks of starch/LDPE composites transferred to the left side gradually with the increase of starch concentration.

3.6 Biodegradability Test

The evaluated masses before and after degradation of the starch/LDPE composite samples is given in Table 2 from which eventually the mass loss (i.e., degradation) is measured. Pure LDPE (100%) showed no apparent mass loss after 3 months of burial. And low starch loaded composites also gave poor degradation performance. This result is in agreement that the presence of very low starch concentration cannot offer the polymer to degrade [2].

The LDPE composite sample containing 15% starch provides mass loss of 2.1% proving that higher starch content increases the degradation kinetics and thus enhances mass loss. This can be due to the hydrophilic nature of starch that has been recommended in another study [2]. The hydrophilic nature of starch tends to absorb moisture that renders the degradation of the polymer composites. Therefore, the greater the starch content in the composite, the higher the moisture content that offers faster degradation through microorganism growth.

3.7 Micro-Hardness Properties after Biodegradability Test

Reduction in the micro hardness properties of the resulting polymer blends is reported as a desirable effect of the addition of starch to polymers. The effects of soil burial test on the micro-hardness properties of potato starch/LDPE blends that were exposed to simple soil environment for the periods of 90 days are visible (Fig. 8). Even after soil burial, pure LDPE didn't show any change in hardness (Fig. 8) which is in agreement with our finding of mass loss measurement. Hardness loss after soil burial indicates the starch removal from the polymer matrix by soil microorganisms. Typically, starch loading is supposed to increase hardness in composite specimens as is found in other studies [17-18]. But, here (Fig. 8) we got an opposite trend which may be due to the stacking of the filler starch fostering confined surface non uniformity which ultimately creates the film brittle alleviating its load standing capacity and its application opportunities. Similar finding was also reported by others [17]. Also, we got supporting evidence from our SEM images where agglomeration was found in the composite samples.

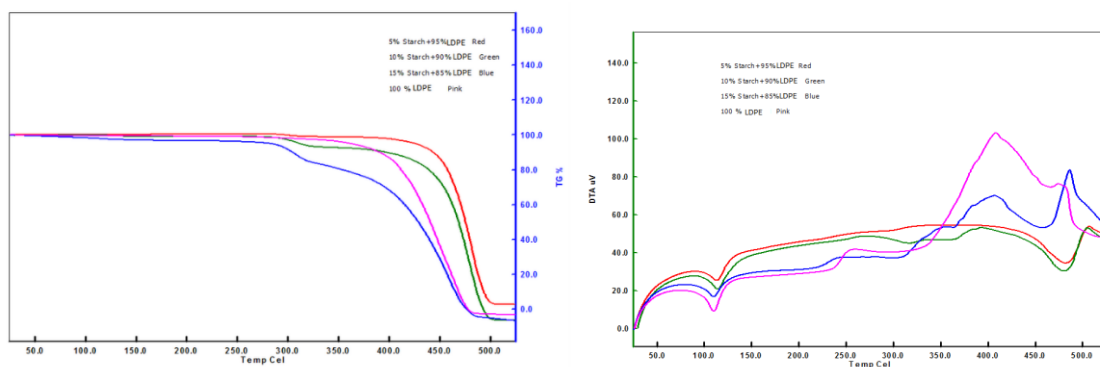
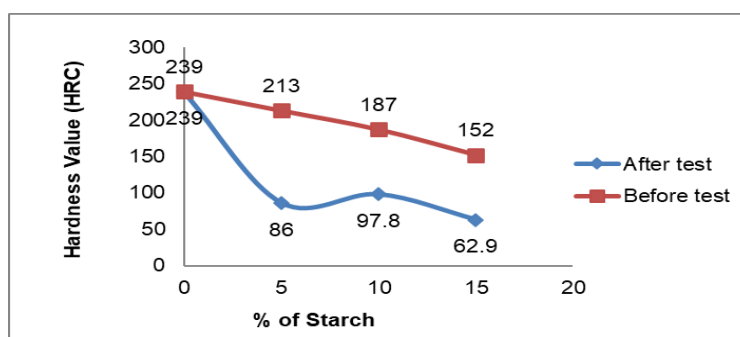


Fig. 7. (Left) TGA and (right) DTA curves of starch/LDPE bio-composites

Table 2. The mass of starch/LDPE bio-composites (before and after soil burial of three month)

Starch Content (%)	Mass (before degradation) (g)	Mass (after degradation) (g)	Mass loss (%)
0	1.998	1.998	0
5	1.899	1.896	0.3
10	1.868	1.857	1.1
15	1.943	1.922	2.1

**Fig. 8. Micro-hardness of starch/LDPE bio-composites before and after soil burial**

4. CONCLUSION

The starch/LDPE bio-composite was successfully synthesized incorporating potato starch of varying concentration into polymer matrix. The results indicate that the addition of hydrophilic starch into hydrophobic LDPE tend to increase the hydrophilicity and biodegradability of the overall polymer. Bulk density, water absorption capacity, elastic modulus, flexural strength and flexural modulus are found to be rising with rising filler concentration whereas tensile strength, elongation at break and hardness are observed to fall with starch addition. Two stages for thermal degradation are found from TG analysis indicating both starch and LDPE degradation respectively. Soil burial test provides proof of biodegradation probability through mass loss and hardness declination. However, it will be more accurate to say that the bio-composites actually biodeteriorate on exposure to a soil environment. Therefore, the degradation characteristic of the potato starch mixed LDPE polymer should be modulated by balancing the starch content and accommodating possible modifications to the filler and matrix. Indeed, the polymer should be developed with necessarily a controlled degradation characteristic while managing the needed strength of the polymeric object during its designed life time for specific application.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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