

Biodegradability of Starch and Starch Blends

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Starch is one of the most abundant biodegradable biopolymers from renewable sources; it also contains tunable thermoplastic properties suitable for diverse applications in agriculture. Functional performances of starch such as physicomechanical, barrier, and surface chemistry may be altered for extended agricultural applications. Furthermore, starch can be a multidimensional additive for plasticulture that can function as a filler, a metaphase component in blends/composites, a plasticizer, an efficient carrier for active delivery of biocides and so on.

biodegradable polymers

starch

1. Introduction

Fossil-derived plastics take more than 100 years to break down in the environment ^[1]. Even though various plastic waste-management systems have been proposed for mitigation, execution is somewhat challenging in plastic recycling, incineration, and disposal into landfills at the end of their service life. Mismanaged plastics create adverse impacts on the environment due to the generation of pollutant gases and toxic substances such as dioxins, furans ^[2], and endocrine disruptors ^[3], along with the production of leachate consisting of heavy metals that pollute water and soil. As a result, there is an alarming necessity for biodegradable plastics and expanding investigations on understanding the biodegradation pathways of biopolymers ^{[4][5]}. Petrochemical plastic production is over 400 million tons as of 2020 ^[6], and global bioplastic production is expected to exceed 7.5 tons in 2026 ^[7].

It is crucial to evaluate the biodegradability of agricultural polymers before using them in various processes and industrial applications. The American Society for Testing and Materials (ASTM), the European Committee for Standardization (EN), and the International Standards Organization (ISO) have established standardized tests to assess biodegradability and the degree of biodegradability of polymers ^{[8][9]}. Aerobic and anaerobic digestions are the main methods to define biodegradation assays and microbial activity that impact the decomposition rates into environmentally friendly components such as carbon dioxide, methane, water, biomass, and inorganic elements (sodium, potassium, phosphorous, and calcium) ^[10]. Another biodegradation assay evaluates ecotoxicity in various plants and animal species such as cress and earthworms ^[9]. Moreover, other standard methods evaluate biodegradability by using material exposure to specific microorganisms ^[11]. Such methods may be subjected to at least one or a few of the following evaluation methods of samples after the assay ^[9]: (a) molecular weight, (b) molecular weight distribution, (c) carbon dioxide and/or methane, (d) weight loss of the material, and (e) the visual observations of changes. In addition to the aforementioned items, different analytical techniques can be used to assess biodegradability, such as Fourier transform infrared spectroscopy, differential scanning calorimetry (DSC),

nuclear magnetic resonance spectroscopy (NMR), X-ray photoelectron spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and X-ray diffraction. After the biodegradation, the tensile properties of polymers (tensile strain, tensile strength, elongation, and tensile modulus) are evaluated before and after comparison [11].

The degradation of starch and starch blends under natural conditions is a complex process that may be facilitated by water adsorption, hydrolysis, and polymer biodegradation [12][13]. There are three stages involved in biodegradation: biodeterioration, biofragmentation, and assimilation [14]. In soil and natural conditions, biodegradation is promoted by microorganisms and enzymes. Complete justification must be given to blends, composites, and surface-modified biodegradable materials due to the tendency to behave differently in biodegradation (**Figure 1**).

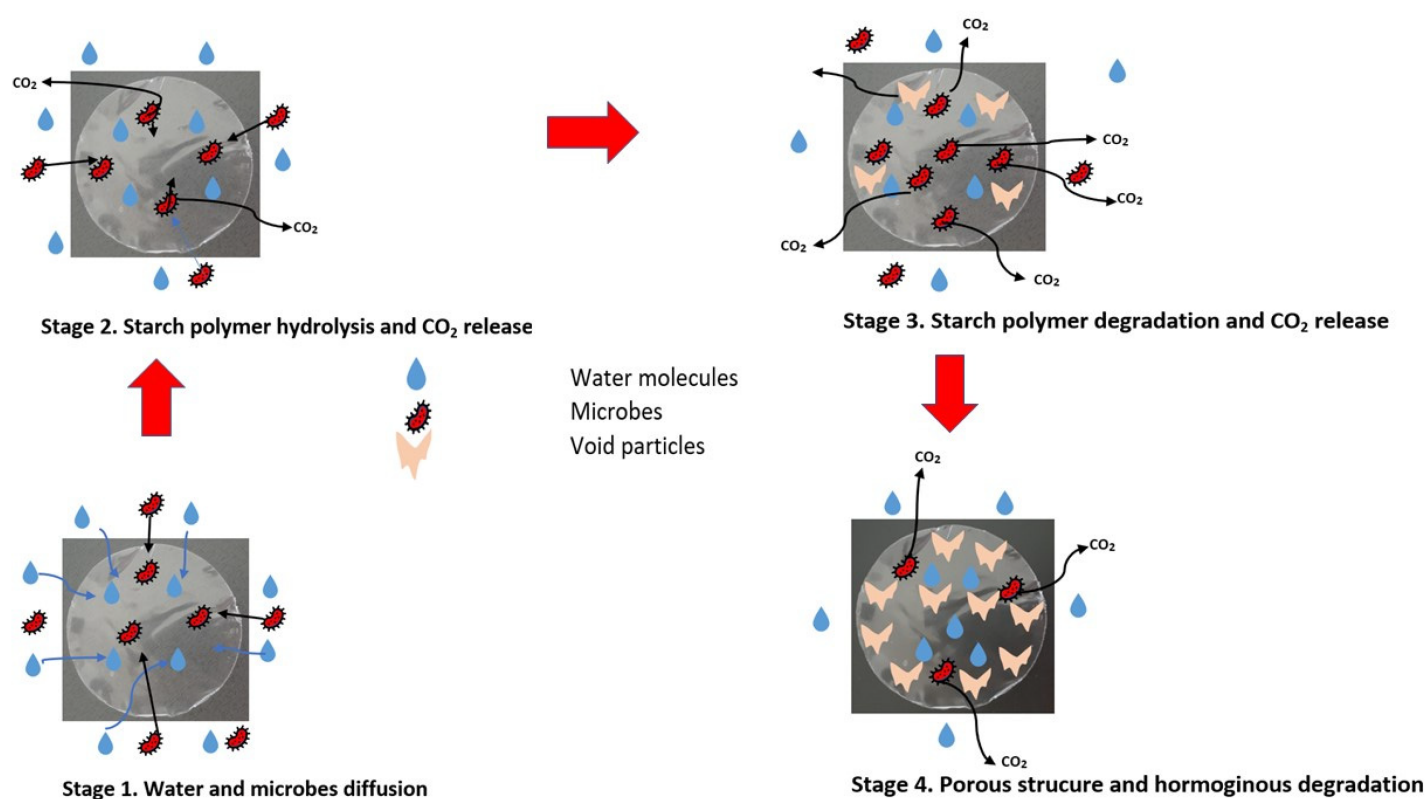


Figure 1. Degradation of biodegradable cassava starch film in soil.

2. Biodegradation of Starch/PVA

Starch/PVA blends are of great importance in packaging and agricultural applications due to their high compatibility and excellent film properties [10]. Several have been conducted to investigate the biodegradability of starch/PVA blends while preparing various wheat starch/PVA/glycerol blends following the solution cast technique in complying with ISO 14855; they learned composting under degradation for 45 days. After allowing the blends to compost for 45 days, it was observed that starch and glycerol were degraded, leaving the PVA fraction intact. Moreover, the blend characteristics were enhanced without interfering with the biodegradation of starch from surface modification

with chitosan [11]. Another one evaluated the biodegradation of PVA/starch blends using a 180-day assay and explained the biodegradation in terms of the changes in molecular weights. These blends had various amounts of cross-linked starch (CLS) compared to PVA blends with acid-modified starches (AMSs).

In similar research, the biodegradation of PVA/AMS blends improved with the increase in AMS percentage. PVA/AMS samples demonstrated a higher degree of biodegradation than PVA/CLS blends [12]. Degradation properties of blow molded PVA/starch films in aqueous anaerobic digestion were learned using sludge from municipal wastewater treatment. This was highlighted that the degradation of PVA blended with native or plasticized starch was significantly increased in terms of degradation rate and elevation, even at a low starch level of 5 wt% [13]. Furthermore, it was reported a higher degree of biodegradation, up to 60%, with loading starch from 21% to 42%, and mechanical properties of starch-modified PVA were declined. Others further assessed the anaerobic degradation of glycerol-plasticized and biopolymer such as starch, gellan gum, and xanthan [14].

Parallel research has further evaluated the biodegradability of some starch/PVA-blended films in soil environments using a 6-month soil burial test in which the weight loss over time was measured [15]. The results showed a better degradation of the citric acid-added films than those with glycerol added, and 80% of the total film degradation occurred with an increasing degradation rate while the rate of degradation was slow [15]. This was revealed that the biodegradability of starch/PVA-blended films that incorporated glacial acetic acid (crosslinking agent) in moist soils might take up to 30 days. It was explained that the biodegradation rates of PVA-blended films were governed by moist vs. dry soil and the molecular weight (31×10^3 to 205×10^3 g/mole). It has been reported that degradation was initiated within 3 days in the moist soil, while in dry soils the initiation time of biodegradation increased from 10 to 14 days [16]. The effect of starch content on biodegradability starch/PVA films prepared using melt processing was further discussed in recent research [17]. The evaluation was carried out by determining the weight loss of specimens buried in soil for 30 days. With increasing starch fraction, the weight loss increased under the same burial test conditions as the highest value of weight loss of the films was obtained at the highest loading of starch in blends. Other researchers affirm the same trend that loading starch in PVA-blended films favors the biodegradation rate; up to 28% to 38% increase in biodegradation rate was achieved at 0–30 wt% loading of starch in PVA blends tested from soil burial method for 45 days [18].

Blends of starch/PVA films from solvent casting resulted in higher susceptibility towards enzymatic degradation in both soil and compost with increasing corn starch loading, which led to achieving up to 85% increase in biodegradation when the burial time is extended in both soil and compost for 8 weeks [19]. The same research further justified that the strength of the blends decreased as the percentage of corn starch was increased. It has been reported that microorganisms from different sources can degrade starch/PVA blends at various degradation rates. Bacteria and fungi species isolated from municipal sewage sludge successfully digested starch components, the amorphous regions of PVA, plasticizer in starch/PVA blends, and glycerol [11]. In similar contexts, two fungi species *Penicillium* and *Apergillus flavus*, isolated from aerobic compost, were able to digest PVA/starch films, resulting in nearly 71% weight loss after 300 days [20]. The same one was described that when two of these fungi activated separately, a higher degradation rate was observed, up to 60% in the actual compost over the same period. Parallel investigations have been executed to assess the biodegradability of PVA/starch blends that have

undergone modifications. Fibrous composites of PVA and natural lignocellulosic fibers from orange wastes incorporated with and without cornstarch biodegraded within 30 days in the soil in 80% mineralization [21]. It has been reported that PVA degradation was enhanced with the addition of fibers while both starch and lignocellulosic fiber degraded faster than PVA.

In contrast, nanoparticles may have a lower impact on the biodegradation of composites. It was concluded that in nano-SiO₂-reinforced starch/PVA nanocomposite films, the nanoparticles have no significant effect on the biodegradability of the films as the reference films prepared (without nanoparticles) resulted in a total weight loss of up to 60%. Biodegradation of metaphase films prepared from clay/starch/PVA has been reported to depend on the characteristics of nanoparticles in a composite, such as the type, content, and composition [22]. Some were represented that the starch/PVA blends are not readily biodegradable and subjected to exposed environmental conditions. This fact can be supported by the following comparative research, which indicated the achievement of a certain degree of biodegradation in solvent-casted starch/PVA films exposed to manure soil. It was further confirmed the similar trend observed, obtaining a significant increment in biodegradation rate with the increase of the starch content. Moreover, the ultimate weight loss obtained from starch/PVA films did not exceed 40% over three months, confirming that starch/PVA blend films are not easily biodegradable in natural conditions [23]. In contrast, it was also reported that the rate of degradation increased with the addition of starch that followed China National Standards (CN:14432). It was analyzed the biodegradability of starch/PVA blends using bio-reactivity kinetic models. According to first-order kinetics, the microorganism's growth rate increased with the loading of more quantity of starch in blended film preparation. Thereby, the decomposition rate of the the starch/PVA blend reached only around 36.66% after 180 days. It is conclusive that starch/PVA blends may not undergo complete biodegradation within a short period under natural environments [12].

3. Biodegradation of Starch/PLA

Following ISO methods, the biodegradability of co-extruded starch/PLA blends in different environments such as liquid, inert solid, and composting media were learned. In the given ISO method, the minimum required mineralization percentage for a compound to be classified as a biodegradable compound is 60%. Concerning starch/PLA blends, it was reported that the percentage of mineralization was higher than 60% and found that starch/PLA blends can be considered biodegradable. Furthermore, this research highlighted that the rate of biodegradation is enhanced with the addition of starch in the liquid medium [24]. In contrast, another one was used the standard procedures described under ASTM D 5209–92, 5338–92, ISO/CEN 14852, and 14855 to measure the biodegradability of the starch/PLA films which were exposed to ultraviolet light at 315 nm prior

Biodegradation of PLA blends with starch and wood flour was learned following the ISO 14855 standard for compost. Biodegradation rates were increased by about 80% by increasing the starch loading up to 40% and were discovered to be relatively lower than those of pure PLA compared to starch/PLA and PLA/wood flour blends [27]. The compostability of pure PLA was further researched using starch/PLA blends at different loadings of starch. Based on the visual inspections, it was observed that all the test samples were completely biodegraded without leaving any residue after 30 days [28]. The same one justified the environmental impact and safe use in the

ecosystem by ecotoxicity test of pure PLA and starch/PLA blends. Another group of researchers learned the biodegradation and degradation rates of the PLA–starch blends using cellulose as the control material in a controlled environment. PLA blends that incorporated chemically modified TPS (CMPS) were extruded, and within 42 days the biodegradability of the blends increased with increasing CMPS content in the blends, and neat CMPS was fully degraded [29]. The degradation of PLA and TPS blends in simulated soils was further investigated using stimulants such as tert-butyl hydroperoxide, myoglobin, and peroxide-activated myoglobin, in which TPS enhanced the degradation of degradation blends in all systems [30]. Several have been conducted using PLA together with starch and different compatibilizers or other substances to evaluate soil biodegradation kinetics and rate. Injection-molded tensile specimens prepared using various combinations of native cornstarch, PLA, and polyhydroxyester-ether (PHEE) were buried in soil for one year to assess the effects of starch and PHEE loading on biodegradation rate [31]. It was reported that the weight loss elevated with increasing starch and poly (hydroxyester-ether) (PHEE) loading in blends [31]. In a comparison one of PLA/starch blends vs. PLA/acrylic acid (AA) grafted starch composite (PLA-g-AA/starch), within 3 months, the starch in the composite was able to entirely degrade in the soil environment [32]. Loading more starch onto composites, the tensile strength at the breakpoint decreased and PLA-g-AA was not degradable as there was not a significant weight observed within 7–12 weeks [32]. Maleic anhydride (MA) has been used as an efficient compatibilizer for PLA/starch blends. The results demonstrated that MA compatibilized blends show better biodegradability than the reference starch/PLA blends in which biodegradability was indicated to be increased with the loading of more starch [33]. A similar one justifies the biodegradability of neat PLA and corn starch/PLA composites with/without lysine di-isocyanate that was examined following enzymatic degradation using Proteinase K and burial tests. According to the results, the degradation rate increased by incorporating more corn starch, and all the corn starch/PLA composites were gradually degraded over the given time period, except pure PLA [34]. In contrast, based on a five-month soil burial experiment designed to interpret the effects of adding PEG to PLA/TPS blends on biodegradation, the mixing of PEG gave an elevated degradation rate, a considerable change in weight loss, and improvement in mechanical properties. It is worth noting, further, that the degradation of blends was increased by incorporating more TPS. It was observed that blends with PEG showed more significant weight loss and enhanced biodegradation of TPS/PLA blends [35].

4. Biodegradation of Starch/PCL Blends

Modern research has conducted numerous experiments to evaluate and examine biodegradation and properties of starch/PCL blends. Biodegradation of starch/PCL blends was evaluated from weight loss and the amount of adipic acid immersed from PCL in two types of starch/PCL blends which are distinguished from the starch sources dried granulated sago starch and undried thermoplastic sago starch (TPSS). The biodegradation rate was enhanced by loading more sago starch, indicating a positive trend in mechanical and biodegradation properties in dried granulated sago starch added to PCL blends. Adipic acid liberation is a direct indication of PCL degradation. Granulated sago starch blends liberated more adipic acid as PCL and TPSS decreased biodegradability [36]. Different aerobic environments such as activated sludge and compost, in the presence of *Pseudomonas putida*, the biodegradation of three different types of films formulated from 100% PCL, a blend of 50% modified starch with 50% PCL, and a blend of 50% unmodified starch with 50% PCL blends were learned. Based on the results, there is

no significant impact on degradation by *P. putida*. At the same time, considerable deformation in every film was observed within the first 7 days—in both activated sludge and compost the environment may accelerate biodegradation—and after 15 days, all the films had completely degraded [37].

Another one was investigated the biodegradability of PCL blends with various starches in anaerobic aqueous environments specific to mesophilic sludge from municipal wastewater treatment [38]. In this one, native corn starch, genetically modified corn starch, gelatinized corn starch, and amaranth starch were used to prepare PCL blends. Then properties of these films were compared with a series of starch/PCL blends that incorporated glycerol. The results demonstrated that the blends that contained glycerol showed better mechanical properties and a higher degree of biodegradation. The biodegradability of the starches may range between 70% (maize starch) and 81% (amaranth starch), while the biodegradation of PCL was reported to be very low, only up to 2% [38]. Other were assessed the biodegradation of metaphase PCL/starch blends included in various components under compost and soil burial tests.

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Biodegradability-modified PCL following the reaction melt processing using glycidyl methacrylate (GMA) and benzoyl peroxide were learned using the compost method [39]. Two types of blends were not significantly degraded after 8 weeks, while higher degradation was achieved in the blends with lower GMA content. The application of azodicarbonamide (ADC) has been learned in simulated soil to accelerate the biodegradability of PCL/corn starch blends. Various proportions of ADC were incorporated into pure PCL and PCL/cornstarch (50/50) blends [40]. It was reported minimal or no significant weight loss and measurable degradation reported only after 100 days [41]. Compared to pure PCL, the highest biodegradation was recorded in the 50:50 PCL/cornstarch blend. ADC showed no impact on the biodegradation of the blends, which might have inhibited the biodegradation of pure PCL [42]. Another one was [43] used three types of PCL blends, high amylose starch [44], and CAB, to evaluate the biodegradation rates. Inside a mature compost made from autoclaved municipal solid wastes, these three blends were buried in a compost seed mixture of compost made from garden waste, which showed the degradation decreased with the decreasing of starch content [43]. The biodegradability of melt-blended PCL/corn starch nanocomposites that had introduced fatty hydroxamic acid to modify sodium montmorillonite (Na-MMT) was learned under the ASTM D5338-92 standard, which reported a higher degree of weight loss after 60 days in PCL nanocomposites than PCL/CS blends [44].

Another was focused on various blends of TPS/PCL and PCL modified with an added MA compatibilizer in a soil environment after 21 days and reported that pure TPS was fully degradable. The rate of degradation was elevated with increasing TPS loading in the blend. The lowest biodegradation rate was demonstrated in the blend, which contained 5 wt.% of PCL-MA, and it was further concluded that the rate of biodegradation is independent of the TPS quantity [45]. Sisal fiber-reinforced PCL/starch blends were evaluated over 9 months using the soil burial test to evaluate biodegradation. Results indicate that the biodegradation was increased by adding fibers into starch/PCL blends [46]. Moreover, the researchers also investigated the biodegradation of twin-screw extruded TPS/PCL blends with 5% and 10% of sisal fiber loading and reported that the degradation declined with the incorporation of fibers in blends. At the same time, higher proportions of TPS could enhance the biodegradation of PCL. Furthermore, degradation kinetics of co-extruded TPS and TPS/PCL blends with introduced sisal whisker loadings of 5 and 10 wt.% has been learned. As per the findings, the addition of the whiskers improved the biodegradation of the TPS and the TPS/PCL matrices; also, PCL in TPS/PCL blends accelerates the biodegradation of TPS [47]. Sisal fibers may retard the biodegradation, and the fibers slowed down the biodegradation. Hence, incorporating fibers should be done under many considerations such as application, matrix, and loading level.

5. Biodegradation of Starch/PHB-V

Limited ones have investigated degradation kinetics and biodegradation of starch/PHB-V blends using a soil compost test that varied the temperature between 100 °C and 140 °C. These blends were tested for 192, 425, and 600 h to induce thermal aging of extrusion-blended corn starch with poly(3-hydroxybutyrate)-co-poly(3-hydroxyvalerate) (PHB-V) or PCL. Biodegradation of starch/PHB-V at 25% starch loading of the starch impacted thermal aging while demonstrating a higher biodegradation rate within ten months compared to PCL blends. Moreover, the biodegradability of the starch PHB-V blends was increased by loading 50% of the starch into PHB-V, making its degradation time half of that of the PHB-V blend without starch [48]. Three types of TPS blends with potato starch, corn starch, and water-soluble potato starch were used in a similar one with two degrees of gelatinization of PHB under the soil burial test. The results indicate that weight loss decreased as PHB loading increased. In addition, weight loss increased as time and the glycerol content increased [49]. The biodegradability of melt-blended 1:1 PHB-V, and glycerol-TPS with m-MMT were investigated following the soil burial method [50]. The results justified an enhancement of mechanical properties in the blends in comparison to pure TPS and a faster degradation rate than pure PHB/V. Furthermore, biodegradation was accelerated up to 90% with increasing m-MMT loading in blends [50].

6. Biodegradation of Starch/PBS and Starch/PBSA

Aerobic and anaerobic biodegradation of cornflour/PBSA and plasticized blends indicated that the biodegradability of blends decreases with incorporating PBSA [51]. In contrast, PBS/starch, PBS, and PLA biodegradation rates were further examined using powdered bioplastics from the soil burial method. According to the observations, the physicochemical structures of PBS and PBS/starch were comparatively more favorable for biodegradation than

PLA under the same given test conditions. PBS/starch blends demonstrated the highest degradability and degradation rates, faster in PBS and PBS-starch than neat PLA [52].

7. Biodegradation of Ternary Blends

Limited research has been published on the biodegradation of ternary blends. The biodegradability and kinetics of TPS blends of PLA, PCL, and starch were explored. These blends were melt-blended and formulated by introducing acrylic acid grafted by melt blending. As per the results, these blends were rapidly degraded within the first 8 weeks under given soil burial test conditions [53]. Biodegradation of binary and ternary blends of PLA, TPS, and glycidyl methacrylate grafted poly (consider ethylene octane) was assessed following compost testing in complying with ISO 14855. The results indicate that the samples with 40% starch loading underwent more than 80% biodegradation within 10 weeks, compared to blends with 10–20% starch loading, which can only degrade up to 40% under given test conditions. Furthermore, higher biodegradation rates were observed in the blends with GPOE compared to those without GPOE [54].

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