Biodegradability and Compostability of Lignocellulosic Based Composite Materials

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ABSTRACT: Lignocellulosic composites have attracted interest from both academia and industry due to their beneficial environmental and sustainability attributes. The lignocellulosic industry has seen remarkable improvements in the development of composites for high performance applications. Both biodegradable as well as non-biodegradable polymers are used in the design and engineering of lignocellulosic composites. Biodegradability studies of lignocellulosic composites in soil and composting environments help in planning their end-life management. Biodegradability tests are complex and dependent on the environment in which the testing is carried out. Due to this, standards have been developed by international agencies such as the American Society for Testing and Materials (ASTM) and International Organization for Standardization (ISO) to adopt and test plastic materials in both composting and soil environments. The first part of this intended review article deals with the classification of lignocellulosic composites, biodegradation and composting concepts, biodegradability testing standards, and factors affecting biodegradation. A comparative analysis of ASTM and ISO biodegradability standards in terms of testing methodology and results interpretation is provided.. A special focus is given to the biodegradation mechanisms found in polymers and their composites. The second part of this review article is devoted to biodegradation studies of lignocellulosic composites under composting conditions and soil environments. The effect of filler type, environmental conditions, and compatibilization on the biodegradation of lignocellulosic composites is discussed in detail. Also, a special section on the biodegradability of lignin-based materials is given.

KEYWORDS: Biodegradation, compostability, lignocellulose, biocomposites

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1 INTRODUCTION

Lignocellulosic fibers are increasingly being used as fillers and/or reinforcing agents in the design and development of polymer composites. The renewability, low cost, rigidity and low abrasiveness of these fillers have made them attractive natural materials in the plastic industry. Furthermore, increased consumer demands for materials for high performance applications have led manufacturers to adopt technological advances to meet these demands. Rising oil prices, depleting natural resources and increased environmental concerns can be circumvented through the use of annually renewable materials. In this regard, lignocellulosic fibers have a major role to play, and the polymer composite industry is undergoing a remarkable transformation in adopting lignocellulosic fibers. This approach will also help the agricultural community, as most of the lignocellulosic fibers used in composites can be obtained from agricultural by-products. Forestry resources like wood also play a significant role in lignocellulosic fiber-based composite uses.

Lignocellulosic fibers can be classified as primary and secondary fibers based on their usage and utilization. Primary fibers are fibers from purpose-grown plants, including jute, sisal, kenaf, flax and hemp. Secondary fibers are fibers obtained as by-products of

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plants, including agro-residues, coir fibers, and pineapple fibers. There are several types of lignocellulosic fibers which are as follows: bast fibers (flax, jute, hemp, kenaf, etc.), leaf fibers (sisal, pineapple leaf fiber), seed fibers (cotton, coir), grass (switchgrass, miscanthus, etc.), reed fibers (wheat, corn and rice) and other types (wood and roots) [1].

Lignocellulosic polymer composites have advanced significantly from the times of simply mixing plastics and fibers and characterizing their properties. Today, more and more biobased plastics are being adopted in designing lignocellulosic composites, and these developments are mirrored by a corresponding growth in the market. The worldwide bioplastic industry capacity was projected by European Bioplastics to increase over a 5-year period to 5.8 million tons per year by 2016, a significant increase from the 1.2 million tons produced in 2011 [2]. Upcoming biobased plastics, such as bio-PE and biobased PET, account for a significant portion of the world's bioplastic production. The biobased PET makes up around 40% of the market, and is projected to control 80% of global production by 2016 [2].

Lignocellulosic fibers are naturally biodegradable in both soil and composting environments. A biodegradable material is one that is able to break down, or degrade, through the action of biological factors such as fungi and bacteria. However, their biodegradation rate depends on their composition. For example, a high lignin content is detrimental to the biodegradation of the fibers in composting environments. Biodegradation of plastics and lignocellulosic composites is a topic of interest for both industry and academia. There are a large number of factors that affect the biodegradability and chemical properties of such materials, and there exists a suitably extensive infrastructure to standardize the classification of these materials. Both of these factors will be discussed in great detail throughout this review article.

2 LIGNOCELLULOSIC MATERIALS AND THEIR COMPOSITES

Lignocellulosic materials are materials that contain both lignin and cellulose. They are also known as photomass, due to the fact that they are obtained as a result of photosynthesis. Wood, grasses, plant and agricultural residues are the most common resources of lignocellulosic materials. Lignocellulosic materials generally lack consistent, uniform and reproducible properties and composition. However, they offer many advantages in the design and engineering of polymer composites because of their renewability, low abrasiveness, load bearing capacity, and biodegradable nature [1]. In addition, these composites can be cost competitive and have applications in various sectors, including the production of durable and biodegradable products.

Lignocellulosic composites are defined as polymer composites in which lignocellulosic materials are major fillers. They are classified into two types based on their biodegradability in soil and composting facilities as shown in Figure 1 (as represented on left-hand side):

1. **Non-biodegradable lignocellulosic composites:** Lignocellulosic composites belonging to this category can be made up of both petro and/or renewable polymers, but they are not biodegradable in either soil or composting facilities. For example: polypropylene-natural fiber composites, biobased nylon-natural fibers composites, etc.
2. **Biodegradable lignocellulosic composites:** Lignocellulosic composites belonging to this category can be made up of both petro and/or renewable polymers. They are biodegradable in either soil or composting facilities. For example: poly(lactic acid)-natural fiber composites, poly(butylene adipate-co-terephthalate)-natural fiber composites, etc. It is well known that PLA is a renewable resource-based polymer, while PBAT is a petro-based polymer.

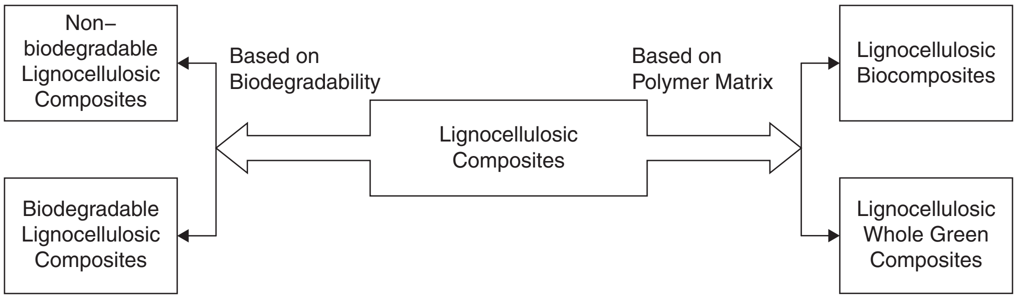

Figure 1 Classification of lignocellulosic composites.

In another concept on the origin of polymer matrix, lignocellulosic composites can also be classified into two types: lignocellulosic biocomposites and lignocellulosic whole green composites (Figure 1, right-hand side):

1. **Lignocellulosic biocomposites:** Lignocellulosic composites belonging to this category are made up of petro-based polymers, but their biodegradability in either soil or composting facilities depends on their polymer matrix. For example: polypropylene-natural fiber composites (non-biodegradable), poly(butylene adipate-co-terephthalate)-natural fibers composites (biodegradable), etc.
2. **Lignocellulosic whole green composites:** Lignocellulosic composites belonging to this category are made up of renewable resource-based polymers, but their biodegradability in either soil or composting facilities depends on their polymer matrix. For example: poly(lactic acid)-natural fiber composites (compostable), biobased nylon-based composites (non-biodegradable), etc.

This intended review article discusses the biodegradability of biodegradable lignocellulosic composites in both soil and compositing environments as mentioned below.

2.1 Biodegradable and Compostable Polymeric Materials

Because of the dependence of biodegradation on both polymer structure and environmental factors, a wide range of tests are required to fully quantify the biodegradability of a given plastic [3, 4]. To ensure a global standard of plastic biodegradability, standardized testing procedures have been defined by international organizations, such as the International Organization for Standardization (ISO) [5, 6] and American Society for Testing and Materials (ASTM) [7]. These organizations define and regulate a number of standard tests that are used to properly assess biodegradable polymers and encompass a variety of environmental factors. The definition of these biodegradability tests was established by the ISO [5, 6, 8]. For biodegradable plastics, these factors include humidity, temperature, pH level, oxygen presence, and most importantly, the presence of microorganisms (both in compost and soil). Each factor affects the rate of degradation, and changing them could have a major effect on the result of the experiment; as a result, these factors must be closely monitored for accurate testing.

Through the addition of natural fiber to the biodegradable polymer, the polymer properties and biodegradability of the resulting composites can be changed in comparison to the base polymer. Just as with pure polymer materials, the true biodegradability of polymer composites (in terms of rate of degradation and toxicity for safe disposal in environmental conditions) must be evaluated using specific standardized testing methods [5, 7]. These tests cover a broad scope of conditions in order to accommodate all types of plastics, and the various environmental conditions they could be exposed to as plastic waste. Thus, with continuous development and innovation in the biopolymer industry, these standard test methods for evaluating the biodegradability of polymeric materials are essential to the future of the plastics industry, and will be necessary to reduce the global issue of plastic pollution.

2.1.1 Definition and Concept

Biodegradation is defined as a process in which materials undergo chemical changes due to the action of enzymes that are secreted by living organisms (bacteria, fungi and algae). The process of biodegradation is comprised of two phases, including the initial phase (primary biodegradation) and the secondary phase (ultimate biodegradation). During the initial phase, a polymer undergoes significant weight loss, reduction in molecular weight and fragmentation, and is degraded into soluble low molecular weight compounds. The second phase of biodegradation is the ultimate conversion of primary degraded low molecular weight compounds into CO_2, water and cell biomass (in aerobic conditions), and CH_4, CO_2 and cell biomass (in anaerobic conditions). The primary and ultimate biodegradation must occur at specific times with specific rates under biological environmental conditions (soil, compost, aqueous, anaerobic digestate) in order to prevent the accumulation of plastics in the environment.

The American Society for Testing and Materials (ASTM) and the International Organization for Standardization (ISO) define:

i. ***Degradation*** as *"an irreversible process leading to a significant change of the structure of a material, typically characterized by a loss of properties (e.g., integrity, molecular weight, structure or mechanical strength) and/or fragmentation. Degradation is affected by environmental conditions and proceeds over a period of time comprising one or more steps"* [9, 10].

According to the ASTM definition [7]:

ii. ***Biodegradable plastic*** is *"a degradable plastic in which the degradation results from the action* *of naturally occurring microorganisms such as bacteria, fungi and algae."*

iii. **Compostable plastic** is *"a plastic that undergoes biological degradation during composting to yield carbon dioxide, water, inorganic compounds and biomass at a rate consistent with other known compostable materials and leaves no visually distinguishable or toxic residues."*

The aforementioned standard guideline definitions have been given to establish and identify biodegradable polymeric materials or products. Based on these guidelines, ASTM standards have provided various test methods to evaluate the potential biodegradability of polymeric materials and products in various disposal environmental conditions such as soil, compost, marine water and sewage. In the case of compostable plastics, the polymeric organic carbon must be converted into CO_2, water and cell biomass within 180 days [11, 12].

2.1.2 Factors Affecting the Biodegradability of Plastics

The rate and degree of biodegradation of plastics is mainly dependent on various factors such as active microbial species present in environments, temperature, humidity, oxygen presence, pH level, UV radiation and polymer properties (crystallinity, hydrophobicity, molecular weight, etc.). The biodegradation behavior of plastics is mainly associated with their chemical structure along with their physical and chemical properties. Both the physical and chemical properties of plastics can influence the polymer biodegradation mechanism. Such properties include surface area, hydrophobic and hydrophilic nature, chemical nature, molecular weight, molecular weight distribution, crystallinity, crystal structure, glass transition temperature, melting temperature and elasticity [3]. These physical and chemical properties play important roles in the polymer biodegradation process in different environmental conditions.

2.2 Biodegradation Mechanisms in Polymers

Polymers contain either a hetero-chain or carbon backbone. The degradation mechanism of hetero-chain backbone polymers is chemical degradation via hydrolysis, or enzyme-catalyzed hydrolysis [13]. Hydrolytic biodegradation mainly depends on the hydrolytic enzymes secreted by local microorganisms and the physico-chemical properties of the polymer. Through this process, polymer biodegradation may occur within a month [14]. The hydrolytic biodegradation lifetime of a polymer can be controlled with the use of additives or chemicals in order to suit various practical applications. Hydrolytic biodegradation is the mechanism for some naturally occurring biopolymers like polysaccharides and proteins, plant source polymers such as poly(lactic acid) (PLA), and poly(butylene succinate) (PBS) and microbially-synthesized polymers like poly(3-hydroxybutyrate-*co*-3-hydroxyvalerate) (PHBV) [13]. Many factors, such as chemical bonds, type of co-polymer, thickness, water uptake and morphology can influence the rate of hydrolytic degradation in enzyme-mediated or non-enzyme-mediated conditions. Comparing hetero-chain and carbon chain polymers, the carbon backbone polymers biodegrade more slowly [13]. The degradation mechanism of carbon backbone polymers is chemical degradation via oxidation or oxidative enzyme-mediated degradation. Several naturally occurring biopolymers, such as natural rubber and lignin, undergo oxidative biodegradation. The process of oxidative polymer biodegradation may take years, significantly more time than hydrolytic biodegradation.

2.2.1 Hydrolytic Biodegradation

Hydrolytic biodegradation occurs in polymers that contain hydrolyzable groups, such as polysaccharides, polyesters and polyamides, when they are exposed to moisture in biotic environmental conditions. The biodegradation of aliphatic polyesters is similar to the biodegradation of cellulose and chitin via enzymatic hydrolytic degradation. It has been reported that a group of esterase enzymes is responsible for the hydrolytic degradation of aliphatic polyester groups [15]. Esterase enzymes, such as lipase, have been well studied, and are more capable of hydrolyzing aliphatic polyesters than aromatic groups [16]. Esterase enzymes are proven to hydrolyze triglycerol into fatty acid and glycerol. The main reason for this kind of degradation is that the active site of lipase is more in contact with the main chain of the polymer, due to the hydrophilic nature of the aliphatic polyester, rather than the aromatic polyester, which is hydrophobic. These enzymatic reactions are heterogeneous; the hydrolytic enzymes adsorb onto the surface of the substrate polymer through the binding site of the molecules, then the hydrolytic enzymes directly contact the ester bond to break it down into functional group molecules. Each group of hydrolytic enzymes has a specific active binding site for substrate molecules, determined by the binding capacities of the substrate. These enzymes can hydrolyze polymers into low molecular weight compounds, which are then capable of undergoing bioassimilation processes with the aid of naturally occurring microorganisms. It has been shown in extensive studies that biobased polymers such as starch, cellulose, poly(lactic acid) (PLA), polyhydroxybutyrate (PHB), chitin, protein, and petro-based polyesters like poly(caprolactam) (PCL) and poly(butylene adipate-co-terephthalate) (PBAT) readily undergo biodegradation under composting conditions [17–19].

2.2.2 *Oxidative Biodegradation*

Oxidative degradation occurs when polymers containing oxidizable groups, such as lignin, natural rubber, poly(vinyl alcohol) and polyolefins, are exposed to either abiotic or biotic environmental conditions. The oxidized biodegradable compounds are further assimilated by microorganisms [20–22]. Lignin is an example of a natural polymer that, like *cis*-polyisoprene, cannot biodegrade through a hydrolytic process, but instead biodegrades slowly by the oxidative attack of extracellular peroxidases produced by fungi and *actinomycetes* [23].

Both hydrolytic and oxidative degradation share the same purpose: to break the polymer into fragments small enough to pass through the cell membrane of the active microorganism [19, 24–26]. These fragments enter the microorganism and are metabolized. In the case of aerobic biodegradation, this process yields a full decomposition of the polymer into carbon dioxide and water [3, 27]. In anaerobic biodegradation, carbon dioxide, water and methane are produced from the polymer. This process is called mineralization. It is the final step in biodegradation, and this means that the active microorganisms utilize carbon sources from the polymeric carbon as a nutrient.

2.3 Standards for Testing Biodegradability in Composting Environment

The standards relating to the compostable plastics have been developed by various international bodies such as CEN (European Committee for Standardization), ASTM (American Society for Testing and Materials), ISO (International Standards Organization) and the Japan BioPlastics Association. Some of the most widely adopted standards for biodegradation of polymers are EN 13432, ASTM D6400, ISO/DIS 17088 and JISK 6953. These standards from these organizations are intended to identify the polymeric materials and their related products which undergo biodegradation to their specified standards.

According to the European Directive on packaging and packaging waste [28], the packaging waste processed through composting must be biodegradable in nature and it should not hinder any unrecoverable materials.

According to the ASTM D6400 standard specification [7], in order for the compostable plastics and their products including packaging made from plastics to be considered "compostable in municipal and industrial composting facilities," the following key requirements must be satisfied for labeling and marketing products.

1. The polymeric carbon must be converted into CO_2, water and cell biomass by the action of natural microorganisms such as bacteria and fungi.
2. The biodegradation rate shall be comparable to the similar biodegradation rates of natural materials such as leaves, paper, grass and food scraps.
3. The compostable plastics should be disintegrated during the biological treatments.
4. The final quality of compost produced should not have any harmful effects.

According to ISO/DIS 17088 international standard, the biodegradable plastic is designed for recovering through aerobic composting process, and the following four criteria must be addressed:

1. Biodegradation (conversion carbon into CO_2, water and cell biomass by natural microorganisms).
2. Disintegration of compostable plastics during microbial treatment.
3. No negative impacts on these biological processes.
4. No toxic effects on the final quality of compost (including in the presence of metal and other harmful ingredients).

European standard EN 13432 is the most relevant standard in all European countries and is valid in all EU member states. This standard specifies that the compostability and anaerobic treatability of packaging waste including products produced from plastics must have the following characteristics:

1. Biodegradability
2. Mineralization during biological treatment (microbial enzymatic process)
3. No adverse effects on biological processing
4. No harmful effects on the final quality of compost

Biodegradable plastics in Japan are identified under the GreenPla identification system which is monitored by the Japan BioPlastics Association (JBPA). GreenPla is the nickname for biodegradable plastics. GreenPla

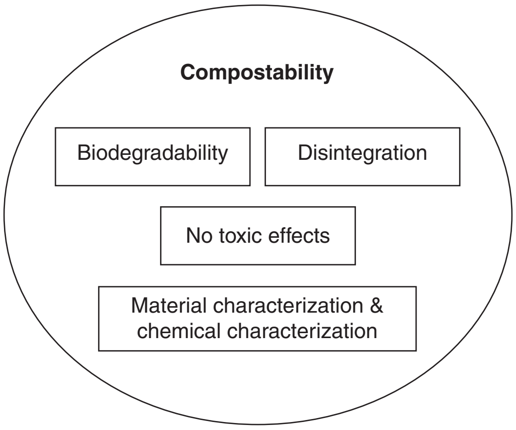

Figure 2 The criteria of compostability.

considers both the treatment and final disposal of the plastics in understanding and certifying the biodegradability. GreenPla is ultimately degraded into water and carbon dioxide. JIS K 6950, 6951, 6953, 6955 are the standards used by JBPA [29–33]. The following are the requirements for GreenPla certification:

1. All constituents of the product must be disclosed to JBPA and be on the positive list.
2. Product must pass the JBPA's standards.

In general, the criteria of compostability are similar in all standards and are represented in Figure 2.

According to the US Environmental Protection Agency (EPA), composting is a biological process decomposing of organic material in an aerobic environment to convert it into the form of humus-like material [34]. Organizations such as the ASTM, ISO and European Normative (EN) are involved in establishing the standard test methods for materials, systems, products and services for both producer and consumer consumption. These standard techniques mainly cover the required criteria to claim materials as environmentally biodegradable and compostable (Table 1). The ASTM standard D6400 [7] defines compostable plastic as one that undergoes biological degradation when exposed to controlled composting conditions to produce CO_2, H_2O and new cell biomass at a consistent rate, and leaves no toxic residues.

The ISO standards ISO 14855–1 and ISO 14855–2 [9, 10] define composting as "autothermic and thermophilic biological degradation of biowaste (organic waste) in the presence of oxygen under controlled conditions by the action of micro- and macro-organisms in order to produce compost." ISO 14855–1 and ISO 14855–2 are tests of biodegradation in compost; ISO 14855–1 analyzes the amount of CO_2 evolved using a general method, while ISO 14855–2 requires the evolved CO_2 to be measured gravimetrically. According to ISO 14855–1, a maximum of 100 grams of sample and 600 grams of inoculum are required for one-test sample measurements. Conversely, only 10 grams of sample are required according to ISO 14855–2. According to the ISO 17088 standard guidelines for a material to be considered a compostable plastic, the conditions of one of ISO 14855–1, ISO 14855–2 or ASTM D5338 must be satisfied (Table 1).

According to the European Norm (EN) standards 13432 and 15351 [28, 35], biodegradability is the ability of a compostable material to be converted into CO_2 through the action of microorganisms. This property is measured with the laboratory standard test method EN 14046. This standard test method is similar to the ISO 14855, another biodegradability test that applies controlled composting conditions. For a material to claim complete biodegradability, it must biodegrade by at least 90% within 180 days. In the same test, there must be evidence of no visual pollution, such as fragmentation and disintegrability, in the final compost within 90 days. Table 1 gives a comparison of ASTM, ISO, and EN compostability standards in terms of mineralization, disintegration and eco-toxicity.

2.3.1 *Composting Process and Methods*

The composting process of organic materials can be accomplished using three basic methods: 1) in-vessel methods, 2) aerated static pile methods, and 3) windrow methods [37, 38]. In the in-vessel methods, the organic materials are processed in a covered container or open channel process. These processes are closely monitored to ensure that they are well aerated with proper humidity conditions, and are mechanically agitated at a frequent, consistent time interval. The aerated static pile method uses forced air to form piles of compostable material. This pile is not mechanically turned or mixed. The windrow method uses elongated piles of compostable material which are regularly mechanically turned. In order to achieve a successful composting process, the composting microorganisms must proceed with the following phases: i) mesophilic phase, ii) thermophilic phase, and iii) cooling and maturation phase [39].

2.3.1.1 Mesophilic Phase

The mesophilic phase occurs in the beginning of the composting process and extends up to 45°C. Mesophile microorganisms are active during this

Table 1 Comparison of standards relating to specification for compostable plastics [6, 8, 9, 36, 37].

Standard	Test Results
ASTM D6400	*Mineralization:* • For products consisting of a single polymer (homopolymer or random copolymer), 60% of the organic carbon must be converted to carbon dioxide by the end of the 180 day test period when compared to the positive control. • For products consisting of more than one polymer (copolymer of either block copolymer or segmented copolymer, blends, and addition of low molecular weight additives), 90% of the organic carbon must be converted to carbon dioxide by the end of the test period within 180 days, when compared to the positive control. • Organic constituents present more than 1% shall be individually tested. *Disintegration:* • No more than 10% of polymer original dry weight may remain after sieving after 84 days in controlled laboratory conditions. *Eco-toxicity:* • There must be no adverse impact on the ability of compost (after testing) to support plant growth and low levels of heavy metals as described in these standards [28, 36].
ISO 17088	*Mineralization:* • For products consisting of a homopolymer, 60% of the organic carbon must be converted to carbon dioxide within 180 days. For all other polymers (e.g. copolymer or blends), 90% of the organic carbon must be converted to carbon dioxide within 180 days. *Disintegration:* • No more than 10% of its original dry mass remains after sieving on a 2 mm sieve after 84 days in controlled composting test. *Eco-toxicity:* • Low levels of heavy metals as described in these standards [28, 36]. • A minimum of 50% volatile solids (according to APHA 2540G standards). • Ecotoxicological assessment (plant growth test on two different plant species following modified OECD guideline).
EN 13432	*Mineralization:* • Biodegradation level of at least 90% must be reached in less than 6 months, either from biowaste and packaging like paper products and biodegradable plastics. *Disintegration:* • No more than 10% of the residues from the packaging waste fragments after sieving on a 2 mm sieve after 84 days in controlled composting test. *Eco-toxicity:* • Result should show low levels of heavy metals as described in these standards [28, 36]. • Should meet physical chemical analysis of the resulting compost and ecotoxicological assessment (plant growth test on two different plant species) according to OECD guideline as described in these standards [28, 36].

stage of composting. At this stage of the composting process, the active mesophilic bacteria and fungi are capable of degrading soluble low molecular weight organic compounds such as starch, monosaccharides and lipids. During this phase, the bacteria produce organic acids and the compost starts to slightly decrease in pH from 5.5 to 5.0. At the same time, the exothermic degradation process causes the compost temperature to rise rapidly. During this period, due to the protein degradation and release of NH_3, the pH level starts to rise to a maximum 8–9. The duration of this phase may occur between a few hours to a few days [13].

2.3.1.2 Thermophilic Phase

The thermophilic phase occurs when the compost reaches temperatures above 40°C. During the thermophilic phase, the higher temperatures accelerate the breakdown of proteins, fats and carbohydrates like cellulose, hemicelluloses and other complex structural compounds. In this phase only, thermophilic bacteria and fungi are more responsible for the higher degradation of organic materials. At temperatures exceeding 55–60°C the microbial activity and diversity decrease dramatically, due to thermophile microorganisms being unable to survive above this temperature. This phase can range significantly in length, from a few days, up to several months. During this phase, the pH is stabilized to a neutral level.

2.3.1.3 Cooling and Maturation Phase

As the higher energy compounds have been exhausted by the degradation of thermophilic bacteria, the compost becomes cool and mesophilic bacteria and fungi start to reappear to start the maturation phase. The mesophiles thrive in low temperatures and will become active once the compost becomes cool. During this phase, most of the species may differ from those present in the first mesophilic phase. The biological processes decline, but the compost continues to be humified and matures.

The above three phases have been identified in all composting processes. Based on these phases, the role of microorganisms and the conditions involved in organic carbon degradation under composting conditions have been evaluated. Based on these biological organisms, various standard test methods have been established for plastic waste disposal in municipal composting and industrial composting conditions. Among these conditions, studies have shown industrial composting conditions are more favorable for plastic waste disposal [14].

2.3.2 Microorganisms

During the composting process, different species of microorganisms dominate at different composting phases [39–41]. During the mesophilic phase, the microorganisms work to degrade any readily available low molecular-weight soluble compounds. At this stage, the temperature starts to rise and the bacterial growth rate declines. During this stage, mainly thermophilic bacteria of the genus *Bacillus* are activated [42]. When the temperature rises to 55°C and above, many human and plant pathogens are killed because of the high temperature. Fungi and bacteria persist as the majority of the compost microorganisms. Protozoa, larvae, and viruses also play a minor role in composting. Other heterotrophic bacteria, hydrogen-oxidizing bacteria, sulphur-oxidizing bacteria and denitrifying and nitrogen-fixing bacteria are also present in this stage. During the cooling and maturation phase, *Actinomycetes* grow rapidly. Anaerobic bacteria may also be present in small numbers in the compost environment. When the oxygen content is rapidly consumed, anaerobic microorganisms may develop during the thermophilic phase. It has been found that various protists and large species of microorganisms, including springtails, millipedes, mites, ants, beetles, centipedes, spiders, and worms are present in the maturation phase of the compost.

2.3.3 Composting Conditions

In order to monitor the biodegradability of plastic materials and their composite materials from natural and synthetic origin under composting conditions, the following optimization of compost is required for studying in a laboratory setting. The compost inoculum is screened to less than 10 mm by sieving and manually removing and discarding any large inert items (pieces of glass, stone, wood, etc.). Table 2 presents the required physical-chemical properties of compost. The total dry solids (weight %) is determined by the amount of solids present in the compost when measured after drying it at 105°C for 10 hours. The amount of total volatile solids is determined by heating the total dry solids after evaporating the moisture content at 550°C for 30 minutes. It is necessary to characterize the compost quality, including total nitrogen and carbon content, by elemental analysis. The C/N ratio in the mix can be adjusted with ammonium chloride if it is more than 40% [34].

Table 2 Physical-chemical properties of compost.

Analysis	ASTM D6400 requirements	Methods
Total Dry solids (%)	50–55	APHA 2540D and 2540E
pH	7–8	ASTM D12931
Moisture (%)	50–55	APHA 2540D and 2540E
C/N ratio	1:10–1:40	Elemental analysis
Volatiles solids (%)	More than 30	APHA test methods 2540 D and 2540E
mg CO_2 emission/g volatile solids	50–150	CO_2 evolution test

Table 3 Comparison standards relating to the specification for biodegradation of plastics in soil.

Standard	Test Method and Duration	Biodegradation condition (mineralization)
ASTM D 5988-96/2012	Analysis of evolved carbon dioxide from testing samples with time interval .The recommended frequency for analysis of carbon is every 3 to 4 days for the first 2 to 3 weeks and every 1 to 3 weeks thereafter. This is characterized by titrations over a 6 month period.	*Biodegradation:* No defined time duration for testing materials. *Validity of test:* More than 70% theoretical CO_2 evolution is observed for a reference material (starch or cellulose) within 6 months.
ISO - 17556	Test samples oxygen demand or analysis of evolved carbon dioxide over a 6 month period.	*Biodegradation:* No defined time duration for testing materials. *Validity of test:* (a) Degree of biodegradation is more than 60% of reference material at plateau phase or at the end of the test and (b) Biodegradation values of, or amounts of carbon dioxide evolved from, the two blanks are within 20% of the mean at the plateau phase or at the end of the test

2.4 Standards for Testing Biodegradability in Soil

Plastics that are biodegradable in soil are defined as "degradable plastics in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi and algae" (i.e., real soil conditions) [43].

Table 3 presents the ASTM and ISO standards that are available for testing plastics in a soil environment. These test methods cover the determination of both rate and degree of ultimate aerobic biodegradation of polymeric materials in soil.

ASTM D5988–12 specifies a "standard test method for determining aerobic biodegradation of plastic materials in soil," and deals with the extent of biodegradation over a time period in the microorganisms present in the soil-mineralized plastics [11]. This standard determines the extent of aerobic biodegradation in soil using a measurement of evolved carbon dioxide. Carbon dioxide is an end product of biodegradation and evolves over time. This standard requires the reference material to be at least 70% biodegraded within six months. Also, this test requires that the carbon dioxide output from blank samples should be around 20% during both the plateau phase and at the conclusion of the test. Such CO_2 emissions from blank samples in a composting test do not have threshold values at the conclusion of the test because validation is carried out within 10 days of the test. Any deviations from these criteria shall be regarded as an invalid test, and a new test must be conducted.

ISO 17556:2003 specifies "Plastics – Determination of the ultimate aerobic biodegradability of plastic materials in soil [44] by measuring the oxygen demand in a closed respirometer or the amount of carbon dioxide evolved." This test method is designed to achieve an ideal extent of biodegradation by providing sufficient aeration and humidity to the soil environment. This respirometric biodegradation test method is applicable to test all polymers from natural and synthetic origin, copolymers or mixtures that contain additives such as plasticizers, colorants or water-soluble polymers. Also, this test requires that the carbon dioxide output from the reference material should be more than 60% and for blank this should be 20% during both the plateau phase and at end of the test. Any deviations from these criteria shall be regarded as an invalid test and a new test must be conducted.

3 BIODEGRADATION OF LIGNOCELLULOSIC COMPOSITES

A significant amount of research studies have been conducted to investigate the biodegradability of lignocellulosic fiber-based biocomposites. In this research, both treated and untreated lignocellulosic fillers were used to change or modify the physico-chemical characteristics of the polymer biocomposites. However, research has not been well established in terms of the ultimate biodegradability (mineralization). Biodegradation end products such as CO_2, H_2O and cell biomass occur in aerobic conditions. In the case of anaerobic conditions, biodegradation end products like CH_4, CO_2, H_2O and cell biomass are produced [13]. Many studies have discussed the biodegradation of polymer composites in soil or compost, and reported their observation in terms of weight loss and change in mechanical properties. Although useful, such observations cannot provide an understanding of the complex processes involved in the biodegradation of these biocomposite materials. There has been a wide range of standardized test methods (ISO, ASTM and EN) established to estimate the mineralization (aerobic and anaerobic) of polymeric materials. These methods have emerged in part due to the development of more sensitive test equipment, such as respirometers, fully-closed circuit systems connected with CO_2 and CH_4 sensors which automatically measure the end products of these materials under aerobic and anaerobic conditions. Because of their suitability for the application, respirometers are very commonly used to test the biodegradability of biopolymers and biocomposites.

3.1 Effect of Fiber Content

Lignocellulosic materials derived from various agro-industrial, agri-food waste and agro-forestry feedstocks have been widely utilized in the development of polymer composites. These renewable resource-based materials can have a reduced environmental burden in comparison to non-renewable resource-based plastic materials. The assessment of overall biodegradability of these biocomposites can reveal the final environmental fate of their individual components during composting. Researchers investigated the aerobic biodegradability of chemically treated and untreated maple wood fiber in PLA-fiber composites (70–30 wt%), in compost conditions according to the standard procedure of ISO-14855 at a temperature 58 ± 2°C using a respirometric unit [45]. In this study, maple wood fibers were treated sequentially with several different treatments, including NaOH, NaOH then acetylation, or NaOH then silanation. These studies compared the CO_2 emission from untreated and treated wood fiber-PLA composites and characterized the composites using analytical techniques to understand the behavior of treated natural fiber in the PLA composite. All treated (NaOH, acetylation and silane) wood fiber with PLA composites showed a high extent of biodegradation (90% within 75 days), compared to the untreated PLA composite (90% in 85 days) and neat PLA (90% in 90 days). The author reported [45] that the higher rate of biodegradability observed in the treated PLA composites is due to an enhanced rate of hydrolysis of the PLA polymer matrix. The test samples were further characterized by gel permeation chromatography (GPC) analysis. In treated (NaOH, acetylation and silane) composites, increasing the porosity of the fibers resulted in an increase in the rate and degree of biodegradation in the fiber via the removal of surface waxes and internal hemicellulose structures. Most importantly, this study showed that both untreated and treated wood fiber-PLA composites are biodegradable in composting conditions. Funabashi and Kunioka [46] analyzed the biodegradation of composites prepared from 70% PLA and 30% cotton cellulose fiber in composting conditions at 58 ± 2°C with a microbial oxidative degradation analyzer (MODA) system, following the standard ISO-14855. The cotton fiber PLA composite showed higher rates of biodegradability (80% degradation in 25 days) and was approaching the plateau phase when the experiment concluded at 50 days, while the neat PLA sample attained 80% degradability in 50 days. This study indicated that the biodegradation of PLA samples was enhanced by the presence of cellulose fibers. In both studies [47, 48], PLA composites showed a higher rate of biodegradability than the neat PLA sample, but Funabashi and Kunioka [46] achieved neat PLA biodegradability in shorter times (80% 50 days) than Way *et al.* [45] (90% in 90 days). The analyzed results showed that the slower rate of biodegradation was due to a d-content of 4.5% and 1000 ppm Erucamide processing additive in the PLA of Way *et al.* [45], while the PLA used by Funabashi and Kunioka [46] did not have any additives. The added additive improves the adhesion of the matrix with fillers.

Pradhan *et al.* [49] studied and compared the aerobic biodegradability of injection molded PLA-wheat straw and PLA-soy straw biocomposites with 70–30% ratio of polymer to filler, as well as their constituent materials. The study was done under composting conditions, according to the standard ASTM D5338, by using an acid-base titration procedure. The PLA composites (PLA-untreated soy and PLA-wheat straw) showed 90% biodegradation in 70 days, while the natural biomass (untreated soy straw and wheat straw) attained 90% biodegradation in 45 days, and neat PLA reached 90% biodegradation in 100 days. This study showed that the PLA biodegradation was facilitated by the natural biomass. The authors suggested that these biocomposite strategies can help satisfy the ASTM D6400 standard requirements of 90% degradation in 180 days under composting conditions.

The comparative biodegradability of composites of PLA-soy straw (70–30%), PLA-wheat straw (70–30%), PCL-distiller's dried grains with solubles (70–30%) and PCL-soy meal (70–30%) were studied by Pradhan *et al.* [50] under controlled composting conditions, with the procedure adopted to follow ASTM D5338 using an acid-base titration procedure. This study showed PLA-soy straw and PLA-wheat straw composites attained 90% biodegradability compared to the neat PLA biodegradability that occurred 50% in 70 days. PCL-distiller's dried grains with soluble and PCL-soy meal composites showed 30% degradation in 65 days when compared to the neat PCL degradation which was 25% in 65 days. All of the biocomposite results showed improved biodegradation compared to the neat polymer used. The rate of biodegradability of the PLA- and PCL-based biocomposites was clearly shown to be better than those of neat PLA and PCL. An increase in the biodegradation rate was observed in their composites compared to the neat polymers PLA and PCL. This higher rate of biodegradation is attributed to the presence of readily biodegradable materials such as soy straw, soy meal, distiller's dried grains with solubles and wheat straw.

Biodegradability studies were carried out [51] on biocomposites prepared from 40% bio-flour (rice husk flour filler) filled with 60% of the biodegradable polymer, poly(butylene succinate) (PBS). The authors studied biodegradation test conditions simulated according to the ASTM D 6003–96 under soil burial and aerobic composting in municipal solid waste (MSW) for 80 days [51]. These biocomposite materials were evaluated in terms of percentage of weight loss, change in their mechanical properties before and after degradation, microbial colony count in test samples and surface morphology of the test samples. Based on the observations, by increasing the rice husk flour content from 20 to 40 wt% in the PBS matrix, the biodegradability rate of the biocomposites in terms of weight loss increased in both soil and compost environments, as compared to neat PBS polymer. This study showed that bio-flour-based PBS composites can be biodegraded faster in compost than in soil environmental conditions. These results were further proven by the microbial colonies present after the above biodegradation tests and morphology studies by scanning electron microscopy of these biocomposite samples. The observed results indicated that PBS biocomposites will have shorter survival time in compost rather than landfills.

Recently, studies were carried out by Muniyasamy *et al.* [47] to analyze the aerobic biodegradability of biocomposites prepared from the petroleum-based polymer, poly(butylene adipate-co-terephthalate) (*PBAT*), and bioethanol co-products such as water-washed distiller's dried grains with solubles (wwDDGS). The biocomposites were studied for their degradability under controlled composting conditions according to the standard ASTM D5338 by using a manual acid-base titration procedure. This study suggested that PBAT-wwDDGS (70–30 and 80–20 ratios) biocomposites were sensitive to hydrolytic biodegradation, and the material showed 90% biodegradability in 120 days compared to neat PBAT, which achieved 90% biodegradability in 180 days under the composting conditions used. The rate of biodegradation of PBAT-wwDDGS is similar to that of cellulose and wwDDGS. In addition, thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FT-IR) analysis revealed that the biodegradation mechanisms of these PBAT-wwDDGS biocomposites were hydrolytic. The decrease in thermal stability and ester group broadening of the PBAT matrix after composting incubation was due to enzymatic hydrolysis and biodegradation. The mineralization of the PBAT, wwDDGS, and PBAT-wwDDGS composites under composting conditions were compared with standard cellulose as per ASTM D5338. The mineralization of cellulose has validated the test and compost quality. These results indicated that the use of wwDDGS as a filler in a PBAT matrix can enhance the biodegradability of the resulting biocomposites relative to PBAT polymer matrix (Figure 3).

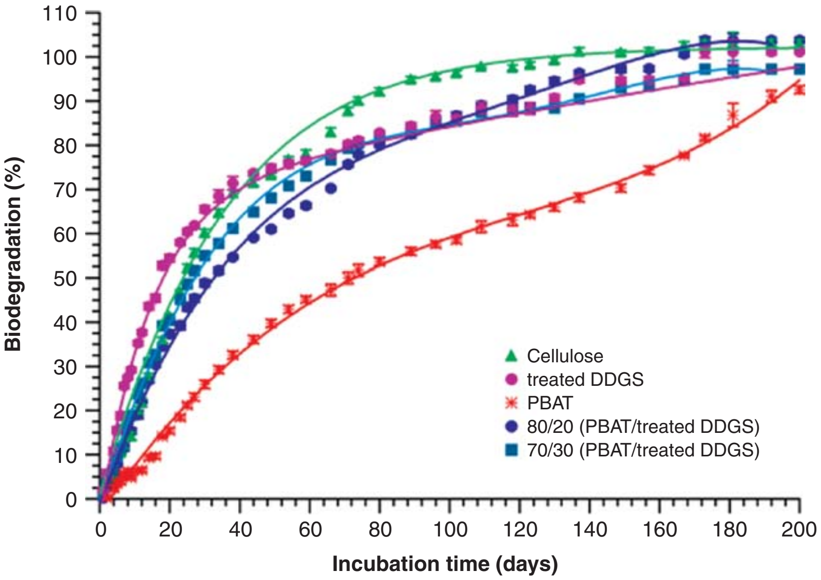

Figure 3 Biodegradation profiles of hydro-biodegradable plastic materials and filter paper in respirometric compost tests. (From reference [47] with permission).

In another study, Chiellini *et al.* [48] showed that composite samples based on starch and poly(vinyl alcohol) (PVA) presented a lower ultimate mineralization in soil and compost experiments. This reduction was caused by the presence of the synthetic PVA polymer. Interestingly, this lower biodegradation was not observed in composites of starch/cellulose fiber (55/45 wt%) and starch/PVA/cellulose fiber (45/10/45 wt%). These biodegradations of biocomposites were evaluated using micro-oxymax respirometer system (Columbus Instruments, Columbus, OH). The micro-oxymax respirometric system is an automated method to measure gas emission from the test samples contained in a closed chamber. This instrument is highly adaptable to suit different standards (ASTM, ISO and EN). Measurement of the biodegradation rate in compost (Table 4) confirmed a higher mineralization rate for samples containing cellulose fiber (55% biodegradation in 75 days), and a lower biodegradation rate for samples prepared without cellulose fibers (40% biodegradation in 75 days). The authors concluded that the biodegradation of PVA is due to the presence of cellulose, which acts as a promoting agent for PVA biodegradation.

Table 4 Biodegradability of natural filler-based biocomposite materials.

Principal components (wt%)	Conditions for biodegradation	Results	Reference
PLA/maple wood fibre (70/30)	Compost ISO-14855	Acetyl treatment of the maple wood fibers increases their porosity, which enhanced hydrolytic biodegradation of the PLA.	[45]
PLA/cotton cellulose fibers (70/30)	Compost ISO-14855	Cotton cellulose fibers accelerated the biodegradability of PLA.	[46]
PLA/soy straw (70/30)	Compost (ASTM D5338)	Untreated soy straw and wheat straw promoted the biodegradability of PLA.	[49]
PLA/soy straw (70/30); PLA/wheat straw (70/30);PCL/distiller's dried grains with soluble (DDGS) (70/30); PCL/soy meal (70/30)	Compost (ASTM D5338)	All the biocomposites showed improved degradation compared to the neat biodegradable polymer used.	[50]
Distiller's dried grains with soluble (DDGS)/(aliphatic and aromatic co-polyester) PBAT (30/70)	Compost (ASTM D5338)	PBAT/DDGS biocomposite showed similar biodegradability to DDGS and cellulose. PBAT degradability increased with the incorporation of water washed DDGS into biocomposite system.	[47]
Poly(ethylene sebacate)/cellulose fiber (85/15)	Compost (ASTM D5338)	Within 30 days, the biocomposite products showed 100% biodegradability	[52]
49% Starch /45% corn fiber/5%inorganic fillers/1% Arabic gum	Compost (ASTM D5338)	Approached plateau phase after 65% biodegradation in 75 days.	[48]
45% Starch/7% poly(vinyl alcohol)/45% corn fiber/3% inorganic filler	Compost (ASTM D5338)	PVA biocomposite approached a plateau phase after 55% biodegradability in 75 days.	[48]
80% Starch/16% poly(vinyl alcohol)/3% inorganic filler/1% Arabic gum	Compost (ASTM D5338)	Approached a plateau phase after 40% biodegradability in 75 days.	[48]
96%Starch/3%inorganic filler/1% Arabic gum	Compost (ASTM D5338)	Approached a plateau phase after 40% biodegradability in 75 days.	[48]
49% Starch /45% corn fiber/5% inorganic fillers/1% Arabic gum	Soil (ASTM D5988)	90% biodegradability was observed within 130 days and these materials satisfied the soil biodegradability criteria as per ASTM standard requirements.	[48]
45% Starch/7% poly(vinyl alcohol)/45% corn fiber/3% inorganic filler	Soil (ASTM D5988)	90% biodegradability observed within 130 days and these materials satisfied soil biodegradability criteria as per ASTM standard requirements.	[48]

Fernandes *et al.* [52] studied the biodegradation of biocomposite materials prepared from hexanoylated cellulose fibers and the biodegradable polyester matrix, poly(ethylene sebacate) (PES). The biodegradation studies were carried out according to the standard methods ISO 14855 and EN 14046 to determine the ultimate aerobic biodegradability of these biocomposites in controlled composting conditions. In these test conditions, PES showed 100% biodegradation within 15 days, while the unmodified cellulose fibers required about 25 days to degrade 100%. Composite samples of PES/15% hexanoylated cellulose fibers were completely degraded, i.e., 100% degraded within 28 days of testing, while a slower biodegradation rate of 60% was observed in the hexanoylated cellulose fiber alone within 30 days of testing. The results concluded that PES/15% hexanoylated cellulose fibers experienced slower degradation compared to PES matrix due to the effect of surface acylation by hexanoyl chloride, which imparted a comparatively nonpolar characteristic to the surface of the cellulose fiber as compared to untreated cellulose fiber.

Alvarez *et al.* [53] prepared biocomposites of sisal fiber (15%) into Mater-Bi®. Mater-Bi is a commercially available biodegradable material and is a proprietary thermoplastic starch. These materials with and without sisal fiber were tested in soil burial conditions for 400 days. Degradation was measured in terms of their weight loss followed by microscopic analysis. Researchers noticed that the sisal fiber played a secondary role in this soil burial test, thereby decreasing the biodegradation rate. In this test Mater-Bi and composite showed 40 and 33 wt% degradation, respectively. The authors concluded that Mater-Bi favors microbial degradation compared to the Mater-Bi/sisal fiber biocomposite, and the higher weight loss of Mater-Bi is mainly due to its amorphous structure (destructed starch), which is easily accessible to the matrix by microorganisms. The sisal fiber plays a minor role, which slows down the biodegradation of biocomposite materials.

Bastioli [54] studied how the presence of starch influenced the rate of biodegradability of Mater-Bi in a composting environment using ASTM D 5338–92. Contrary to the previous research [53], e.g., biocomposites of sisal fiber (15%) in Mater-Bi when exposed to soil burial, only partial degradation was observed. These experimental results clearly indicated that a composting environment is more suitable to enhance the biodegradability of such materials soil environments. Imam *et al.* [55] studied the biodegradation of biocomposite materials with a poly(vinyl alcohol) matrix polymer, and starch and orange fiber fillers, using a soil burial test. The authors concluded that starch was a more biosusceptible material than orange fiber. When the above research studies were concluded, the orange fibers appeared to be more stable for years, and the authors argued that these natural fibers may slowly degrade and favor the hydrolytic degradation of the polymer matrix. These suggestions comply with the other study carried out by Modelli *et al.* [56], in which the biodegradability of chemically-modified flax fibers and starch powder were tested in soil conditions. The chemically-modified flax fibers showed slow biodegradation compared to the starch powder in 180 days; after this long induction time, they started to mineralize faster as they underwent hydrolytic biodegradation.

3.2 Effect of Compatibilizer

Natural fiber reinforced polymer composites require adequate fiber-matrix adhesion to realize their full potential of superior performance [57]. Poor interfacial adhesion results in poor mechanical properties in the final products. In order to overcome this weakness, suitable compatibilizers can be used in engineering high performance biocomposites. Compatibilizers are also referred to as coupling agents or additives that are capable of modifying interfacial adhesion during melt blending of the polymeric matrix with hydroxyl groups on the surface of fiber. The use of compatibilizers in lignocellulosic polymer composites may affect their rate of biodegradability. In this regard, researchers investigated [24] the biodegradation of PLA/corn starch/natural fiber (coir)-based composites with and without the coupling agent, maleic anhydride (MA) (1% wt). The biodegradation of the PLA composite was studied in the presence of compost for a 90 day incubation period, with the conditions of the procedure following the test method ISO 14855. The CO_2 emission was evaluated through a manual titration method. At the end of the 90 day incubation, the biodegradability of the PLA biocomposite, PLA and corn starch were assessed. Corn starch attained a higher level of biodegradation (87%) than neat PLA (55%) in 90 days. The compatibilized composite (PLA/corn starch/coir/maleic anhydride) had lower biodegradation (54%) than the uncompatibilized composite (which showed 59% biodegradation) in a 90 day test period. The researchers concluded that the natural fiber plays a secondary role to the corn starch in the biodegradation of the PLA composite. The presence of the maleic anhydride inhibits the microbial growth in the compatibilized composite, compared to the uncompatibilized composite. The research also concluded that compost is the most suitable environment for the biodegradation of these PLA/cornstarch/natural fiber-based composite materials. Tserki *et al.* [58] studied the commercial polyester Bionolle® 3020, obtained from the Showa Highpolymer Company, Japan, containing succinic acid (40%), adipic acid (10%) and 1,4 butanediol (50%), grafted with 7phr maleic anhydride (MA). The biodegradation experiments of Bionolle-grafted-MA/cellulose fiber composite were carried out in a soil burial environment for 90 days. This test was approached to study the effect of compatibilizers and the thickness of the test specimens (1 and 3 mm) in soil burial conditions. Based on the weight loss observation of the compatibilized composites of varying thickness, the experimental results suggested that thickness was a very predominant factor in controlling the degradation rates. The weight loss of 1 mm thick polyester Bionolle 3020 was 28% over 90 days, compared to only 10% in the 3mm thick Bionolle 3020 over the same period. The 1 mm thick Bionolle-grafted-MA/40% cellulose fiber composite showed a 48% weight loss, which was larger in comparison to the 3 mm Bionolle-grafted-MA/40% cellulose fiber composite, which experienced only 29% weight loss in the same 90 day period test duration. The authors concluded that with the incorporation of 40% cellulose fibers into the polymer matrix, the biodegradation rate was steadily increased. These results also suggested that sample thickness plays a major role, because as the sample thickness increased, the rate of biodegradation decreased due to the lower ratio of surface area to mass. The authors also concluded that the compatibilizer did not have much influence on the biodegradation of compatibilized composites.

The compatibilization of lignocellulosic polymer composites is mainly used to increase potential durability and mechanical performance. The potential toxicity of compatibilized biocomposites has not been well studied in natural environmental conditions such as soil, compost, and aqueous solutions [59]. Further detailed study is needed to ensure that the degradation products and intermediates of such biocomposites will not have any negative effect on living organisms in these environments. The potential ecotoxicity of biocomposite degradation products can be verified using plant growth tests or seed germination tests as prescribed in ASTM and ISO standards.

3.3 Effect of Environmental Conditions

According to an investigation carried out on the biodegradation of PLA filled with cornstarch (up to 60%) [60], there was no degradation observed in the PLA matrix in a soil burial test. The only weight loss that occurred in the composite was due to the degradation of the starch. In another study [61], the PLA biodegradation test was studied and compared to reference cellulose materials in soil and compost conditions at room temperature (30°C) conditions for 200 days. The authors found that there was no PLA biodegradation in the soil or compost at room temperature conditions, while the reference cellulose materials achieved 75% biodegradation in 45 days in both conditions. When the same PLA test materials were exposed to industrial composting conditions at temperature 58°C, 90% biodegradation was achieved within 100 days. Therefore, it was concluded that industrial composting provides a much more suitable environment for the thermophilic bacteria and fungi that are involved in the biodegradation of PLA compared to soil burial, home compost and aqueous conditions, where mesophilic bacteria and fungi are predominant.

Researchers studied the biodegradability of poly(butylene succinate) (PBS) biocomposites with a rice husk flour (RHF) filler in soil and composting conditions [51]. They observed a higher biodegradability in the PBS biocomposite than in the neat PBS, and the biodegradation rate was enhanced with the addition of the rice husk flour. The PBS/RHF biocomposite showed 18% weight loss in compost, while the PBS/RHF biocomposite had 13% weight loss in soil conditions in 80 days. The neat PBS showed 6% weight loss in compost, while the neat PBS experienced weight loss of 3% in soil in 80 days. These comparison studies demonstrated that the RHF promoted the biodegradation of the PBS matrix in both the soil and compost environments. A comparison of the effects of environmental conditions revealed a higher rate of biodegradation in compost compared to soil, due to the larger amount of microbial colonies present in the compost. Biodegradation studies of aliphatic polyesters (PCL, PHBV, PBS, and PLA) with abaca fibers were investigated by Termite *et al.* [62] using a soil burial test. In this study, there was no weight loss observed for PLA in 180 days. The other composites demonstrated biodegradability, in the order of PCL>PHBV>PBS in 180 days. In another study, composites of the biodegradable polyester PBS filled with agricultural residues, such as rice husk [63], were exposed to biodegradation tests in an *Azospirillum brasilense* BCRC 12270 liquid culture medium. It was observed that the degree of biodegradation increased with an increasing rice husk content. To conclude, these various biodegradability studies demonstrated that a composting environment is most suitable for the disposal of bioplastic materials in a short time (within six months). Figure 3 summarizes the conditions in which bioplastics biodegrade in different types of biological waste treatments. It is considered that there are four types of waste treatments: home composting, industrial composting, anaerobic digestion and incineration. The ultimate mineralization of bioplastics is mainly dependent on the environmental conditions (temperature, oxygen and active microorganisms) and the waste treatment options

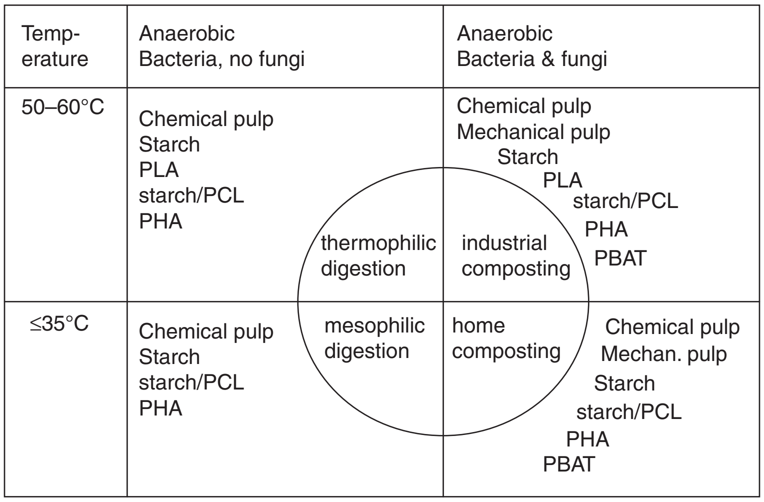

Figure 4 Materials which are biodegradable in four types of biological waste treatment. (From reference [64] with permission).

(composting and anaerobic digestion), which are represented in Figure 4.

4 BIODEGRADATION OF LIGNIN-BASED MATERIALS

The biodegradation behavior of plant mass is very much dependent upon its lignin content, and the microbial population of its environmental systems. Lignin provides physical, chemical and biological protection for the growing of plant wood, straw and husks. Lignin does not degrade easily by either abiotic or biotic processes due to its inertness, and it tends to accumulate in the environment. However, lignin undergoes slow biodegradation under composting conditions. The biodegradation of lignin occurs in a two-step process. The first step involves the reduction of the molecular weight of the lignin, leading to biodegradable low molecular weight compounds [65, 66]. The second step is the microbial assimilation of the low molecular weight compounds produced in the first step. The first step requires a specific microbial enzymatic process, including peroxides in order to breakdown the C-C chains of lignin through non-specific free radical reactions, and produce low molecular compounds which are biodegradable by microorganisms. It has been shown that a large number of peroxide group enzymes are capable of removing lignin from lignocellulosic materials without affecting their cellulose components. *Manganese peroxidase* (MnP) has been implicated as a particularly important enzyme formed by white-rot fungus during the delignification of kraft pulps [61]. This enzyme forms a complex with acids and diffuses away from the enzyme to oxidize lignin. However, the redox potential of MnP is lower than that of lignin peroxidase [65]. Until recently, white-rot fungi were the only organisms known to be capable of biodegrading lignin efficiently by their active extracellular enzymatic process [11]. Currently, research on lignin biodegradation is mainly focused on the following aspects: lignin biodegradation by white-rot and litter-decomposing fungi, and the biochemical pathway mechanisms of peroxidases, purification, production and characterization of ligninolytic and cellulolytic enzymes secreted by white-rot fungi.

Sudhakar *et al.* [13] studied and made a comparison of the CO_2 emission profiles of oxo-biodegradable materials. These materials included natural rubber, pine nut shell lignin (PNSL) with synthetic linear low density polyethylene (LLDPE), and a low density polyethylene (LDPE)/lignin composite. The analysis was conducted according to the ASTM D-5988 biodegradation test in soil. The study revealed that oxo-biodegradable materials undergo slow rates of biodegradation (conversion of organic carbon to CO2) in soil. The authors suggested that the biodegradation processes of the LDPE/lignin composite in soil involve microbial populations and biochemical pathways that are similar to those observed in lignin and natural rubber biodegradation.

The degradation of wood in natural environments such as soil and other outdoor places is mainly caused by fungi. The degradation of wood hard substances is mainly through the fungi organized hypae, which usually helps fungal penetration capacity. It has been identified as *Trichoderma reesei,* which is capable of producing cellulases which hydrolyze the cellobiose and other short cello-oligosaccharide into glucose. In spite of this, *Trichoderma reesei* does not have the capability to produce other enzymes to degrade lignin substances. The best known family of white-rot fungi, such as *Trametes versicolar,* are capable of degrading both lignin and carbohydrates components of wood in the same proportional rate [67].

The degradation of poly aromatic hydrocarbon (PHAs) is mainly caused by litter composting fungi in soil conditions. The PHAs are natural and anthropogenic hydrocarbon which contains two or more fused benzene rings. Due to their large presence, bioaccumulation and carcinogenic nature, PHAs are a serious concern as an environmental burden. The following lignolytic fungi are known to biodegrade PHAs: *Phanerochaete chrysosporium*, *Bjerkandera adusta*, and *Pleurotus ostreatus*. The enzymes involved in the biodegradation of PHAs include lignin peroxidase, versatile peroxidase, manganese-peroxidase, and laccase [68].

The organic material can contaminate it with dioxins and dibenzofurans, extremely toxic dioxins and dibenzofurans which are formed during bleaching in pulp industries. It is common and among the most toxic pollutants encountered in the soil environment. The white-rot fungi *Phanerochaete sordida* has been considered the most efficient organism at mineralizing these toxic compounds in nature. As a result, white-rot fungi are attractive candidates for use in bioremediation applications [69].

Interestingly, in nature, white-rot fungi possess the unique ability to degrade lignin to carbon dioxide [65] in order to have access to the cellulose molecule, which is a carbon source for white-rot fungi. Recently, researchers have started to understand the enzymatic mechanism by which the degradation of lignin is accomplished [65]. Furthermore, ^{14}C-labeled techniques have been used to better understand new features in lignin biodegradation. This technique has helped make it possible to determine the fate of the mineralized components of lignin, and lignin solubilization after microbial assimilation. The ^{14}C-labeling techniques also help in replacing carbons on either aromatic rings or propane side chains [67].

Nonisotopic methods are limited to qualitative lignin degradation studies due to their limited ability to provide rough estimation of degradation [67].

Besides white-rot fungi, there are many types of *actinomycetes* and eubacteria that are able to degrade extracted lignin. Most of the bacterial strains belonging to these genera are only capable of solubilizing and modifying lignin and have limited ability to mineralize the lignin [70].

5 BIODEGRADATION OF NATURAL RUBBER

The microbial biodegradation of natural rubber (NR) occurs in active microbial environmental conditions. Studies have shown [71] that specific microbial actinomycetes species and fungi are capable of degrading 55% mass loss of rubber sheets in 70 days. Tsuchii *et al.* [72] found that the actinomyces, *Nocardia* sp. (strain 835A) is capable of degrading NR rubber efficiently in the synthetic medium in the absence of any other carbon sources. Ikram *et al.* [73] studied the soil biodegradation of natural rubber gloves at ambient temperature. They showed NR gloves reached 54 and 94% weight loss after 4 and 48 weeks respectively. The authors later concluded that NR biodegradation is mainly dependent upon the soil nutrients such as nitrogen and phosphorous. The natural rubber in a higher content of nitrogen (100 mg/l), phosphorous (150 mg/l) system showed a 61.5% weight loss, whereas in the low content of nitrogen (10mg/l), phosphorous (15mg/l) system showed only 23.6% after 24 weeks. Moreover, the microbial colonies on the natural rubber also decreased in the lower content of nitrogen and phosphorous present as expected. These authors have identified a higher number of bacterial population (12317/mg) on the NR gloves than fungi (441/mg) and actinomycete population (297/mg). In studies, Linos *et al.* [74, 75] compared the biodegradability of natural rubber and synthetic rubber in the presence of *Pseudomonas aeruginosa* under aqueous medium. Results showed natural rubber was 26% mineralized in six weeks, compared was 21% mineralization for synthetic rubber. The authors concluded that natural and synthetic rubbers undergo a similar rate of biodegradation, and the slight changes may be due to the antioxidants used in the synthetic rubber. The microbial species *Nocardia* and *P. aeruginosa* were shown [74, 75] in which the breakdown of cis-polyisoprene chain was found due to oxidative microbial enzymes. The evidence suggests that bacteria initiate free-radical chain peroxidation by oxidative biodegradation mechanisms.

6 CONCLUSION

Lignocellulosic composites made from both bioplastics and fossil plastics are advancing in a multitude of applications, including in both durable and single-use products, due to their renewability, low cost, low relative density and biodegradability (in the case of some lignocellulosic composites). The end life management of these lignocellulosic composites depends on their biodegradability in their optimal disposal environment. The biodegradability of a lignocellulosic composite mainly depends on its polymer matrix. Many studies have shown that lignocellulosic composites are biodegradable if they are made up of a biodegradable polymer matrix. The composition of lignocellulosic fibers used as fillers also influences the biodegradation behavior of their composites. Also, the rate of biodegradation of lignocellulosic composites depends on many other environmental factors, such as moisture, humidity, temperature and microbial population. This review article has emphasized the variability of the biodegradation behavior of lignocellulosic composites in composting and soil environments. The international standards concerning testing and labeling of biodegradable plastics in composting conditions and soil environments were also explained, and are essential to ensure that biocomposite materials are developed intelligently. The validation criteria for biodegradability of lignocellulosic composites and polymeric materials such as mineralization percentage, disintegration and ecotoxicity as per different testing methods vary between different standards.

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GLOSSARY:

Abiotic: Components of an environment which are non-living chemical and physical factors that affect the ecosystem. For example: sunlight, temperature, oxygen, etc.
Adsorption: The adhesion of particles from a fluid to a surface due to attractive forces**.**
ASTM: American Society for Testing and Materials
Biocomposite: Polymer composite consisting of either a filler or polymer matrix that is derived from biological resources.
Biodegradability: The ability of a material to undergo degradation by the action of biological agents such as bacteria, fungi, etc.
Biodegradable Composite: A polymer composite that degrades by the action of biological agents such as bacteria, fungi.
Biotic: Components of an environment which are living and affect the ecosystem. For example: bacteria and fungi.
Compostability: The ability of a material to undergo degradation by the action of biological agents such as bacteria, fungi, etc., at the same rate as that of known compostable materials, such as cellulose, and without leaving toxic residues.
Composting Rates: The rate of degradation required by polymers as per the compostability testing standards. For example, ASTM D6400 requires 60% of the organic carbon present in single polymer to be converted to carbon dioxide by the end of the 180 day test period.
Composting Pile: The pile of organic masses in which the aerobic breakdown of the substrate occurs.
Inoculum: Medium containing microbial species.
ISO: International Organization for Standardization
Lignocellulosic: Plant biomass composed of cellulose, hemicellulose and lignin.
Mesophile: Microorganisms that grow best in moderate temperature, neither too hot nor too cold, typically between 20 and 45°C.
Mesophilic Digestion: Digestion of waste at elevated temperatures up to 45°C in the presence of mainly mesophilic microorganisms.
OECD: Organization of Economic Co-operation and Development
Oxygen Demand: The amount of dissolved oxygen needed by microorganisms in soil biodegradation testing.
PLA: Poly(lactic acid)
PHAs: Polyhydroxyalkanoates
PHB: Polyhydroxybutyrate
PHBV: Poly(hydroxybutyrate-co-hydroxyvalerate)
PCL: Polycaprolactone
PBAT: Poly(butylene adipate-co-terephthalate)
PBS: Poly(butylene succinate)
PE: Polyethylene
PP: Polypropylene
PET: Poly(ethylene terephthalate)
NR: Natural rubber
Thermophile: Microorganisms that live and grow in hot environments that would kill most other organisms.
Thermophilic Digestion: Digestion of waste at elevated temperatures of up to 70°C in the presence of mainly thermophilic microorganisms.
Whole Green Composites: Polymer composites that are made up of renewable resource-based polymers and fillers. Their biodegradability depends on the type of polymer matrix used.

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