EFFECT OF A BIODEGRADATION PROMOTING ADDITIVE ON POLYETHYLENE TEREPHTHALATE IN ANAEROBIC DIGESTION

By

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ABSTRACT

EFFECT OF A BIODEGRADATION PROMOTING ADDITIVE
ON POLYETHYLENE TEREPHTHALATE IN ANAEROBIC DIGESTION

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Polyethylene terephthalate sheet (PET) with a biodegradation promoting additive, provided by ENSO Plastics, was made and evaluated in an anaerobic environment with three different inoculums: landfill leachate, wastewater treatment residue, and liquid from an anaerobic digester. As bioreactors, 125 mL glass bottles with closures were prepared, and test samples, fresh cow manure, and inoculums were placed into the bioreactors. The bioreactors were kept in a 35 ˚C incubator. Gas production from the samples was measured for 90 days. Cellulose samples, prepared as positive controls, showed higher gas production than the other samples, and its biodegradation extent reached 32.4 %, 51.6 % and 36.5 % in each inoculum, respectively. However, PET with additive samples did not show higher gas production than either blank or neat PET in landfill leachate, wastewater treatment residue and anaerobic digester inoculums. Statistical analysis of the gas production data showed that only cellulose was significantly different than the other samples in landfill leachate, wastewater treatment and anaerobic digester inoculums ($\alpha=0.05$). In conclusion, no significant difference was observed in PET with biodegradation promoting additive in this test environment. According to ENSO Plastics, the cloudiness of the PET sheets made for this experiment indicated insufficient dispersion of the additive: the company states that excellent dispersion is required to enhance biodegradation.

To my family

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KEY TO ABBREVIATIONS

ABS	Acrylonitrile butadiene styrene
ASTM	American Society for Testing and Materials
$CaCO_3$	Calcium carbonate
Ce	Cerium
Co	Cobalt
Cu	Copper
CH_4	Methane
CHN	Carbon, hydrogen and nytrogen
CO_2	Carbon dioxide
COD	Chemical oxygen demand
DSC	Differential scanning calorimetry
EVA	Ethylene vinyl acetate
EVOH	Ethylene vinyl alcohol
Fe	Iron
FTIR	Fourier transform infrared spectroscopy
IPCC	Intergovernmental Panel on Climate Change
ISO	International Organization for Standardization
LDPE	Low density polyethylene
Mn	Manganese
MSW	Municipal solid waste
NaOH	Sodium hydrate
Ni	Nickel
OECD	Organization for Economic Cooperation and Development
PBAT	Polybutylene adipate-co-terephthalate

PBS	Polybutylenesuccinate
PBSA	Polybutylene succinate-co-adipate
PCL	Polycaprolactone
PE	Polyethylene
PET	Polyethylene terephthalate
PHAs	Polyhydroxyalkanoates
PHB	Polyhydroxybutyrate
PLA	Polylactic acid
PP	Polypropylene
PS	Polystyrene
PU	Polyurethane
PVC	Polyvinyl chloride
RH	Relative humidity
SEM	Scanning electron microscope
STP	Standard temperature and pressure
T_g	Glass transition temperature
TGA	Thermal gravimetric analysis
T_m	Melting temperature
UV	Ultraviolet
WTE	Waste to energy
WVTR	Water vapor transmission rate

CHAPTER 1

INTRODUCTION

1.1 Background

As societies become more advanced, consumption is promoted, and municipal solid waste (MSW) is becoming a serious concern all over the world. Higher income countries are suffering from large amounts of wastes, and lower income countries are suffering from inappropriately treated wastes. According to the MSW fact sheet in U.S., MSW generated was 88.1 million tons in 1960, but this increased to 250.9 million tons in 2012 [1]. Based on the available data in 2012, 20 countries in the Organization for Economic Co-operation and Development (OECD), which are mostly developed countries, generated 572 million tons of solid waste per year, and this was 44 % of the waste in the world. Based on the same data, 1,289 million tons of solid waste per year is produced currently in the world, and it is estimated to almost double, to 2,215 million tons of solid waste per year, by 2025 [2]. Not only is the amount of waste, but also waste management a serious problem. Landfill is the most common method; it was 44 % of all waste disposal in the world. The most desired method, recycling, was relatively low; 17 % in the world (Figure 1-1). In the U.S., landfill was 53.8 % followed by 34.5 % recovery and 11.7 % energy recovery in 2012 [2]. In Europe, landfill was approximately 45 % followed by 35 % recycling and 20 % incineration in 2011 [3]. In developing countries, landfill is operated poorly, and it is appropriate to call it controlled dumping. In undeveloped countries, open dumping and open burning are the dominant methods.

Looking at the composition of the waste, plastic has been significantly increasing since its innovation. Figure 1-2 shows the increase of plastic production in the world. In the U.S., plastic constituted 12.7 % of MSW generation in 2012, and in the world, it constituted 10 % of MSW generation [1] [2]. Investigating further, packages generate a large percentage of plastic

waste. In Europe, packaging constituted 39.4 % of total plastic demand in 2011 [4], and in the U.S., plastic waste of containers and packages was 13.78 million tons and constituted 43.4 % of total plastic waste generation (31.75 million tons) in 2012 [1].

To achieve a sustainable system, plastic recycling is important. For the package industries, the recycling rate of polyethylene terephthalate (PET) bottles is valuable since PET is one of the most widely used materials for packages. In the U.S., the recycling rate of PET bottles was 31.2 % in 2012, an increase from 19.6 % in 2003 [5]. In Europe, the recycling rate of PET bottles was 52.3 % in 2012 [6]. Some countries have achieved relatively high recycling rate. For example, in Japan, the recycling rate of PET bottles was 85.8 % in 2013 [7].

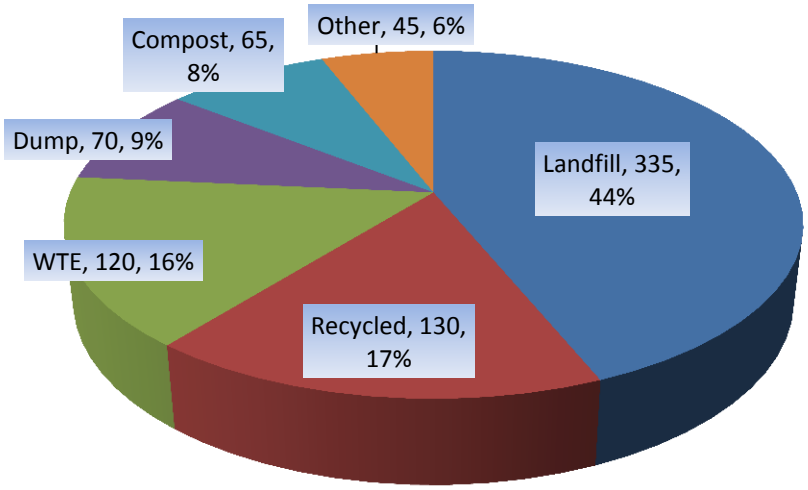

Figure 1-1 Waste management in the world, adapted from [2].

Note: unit is million tons.

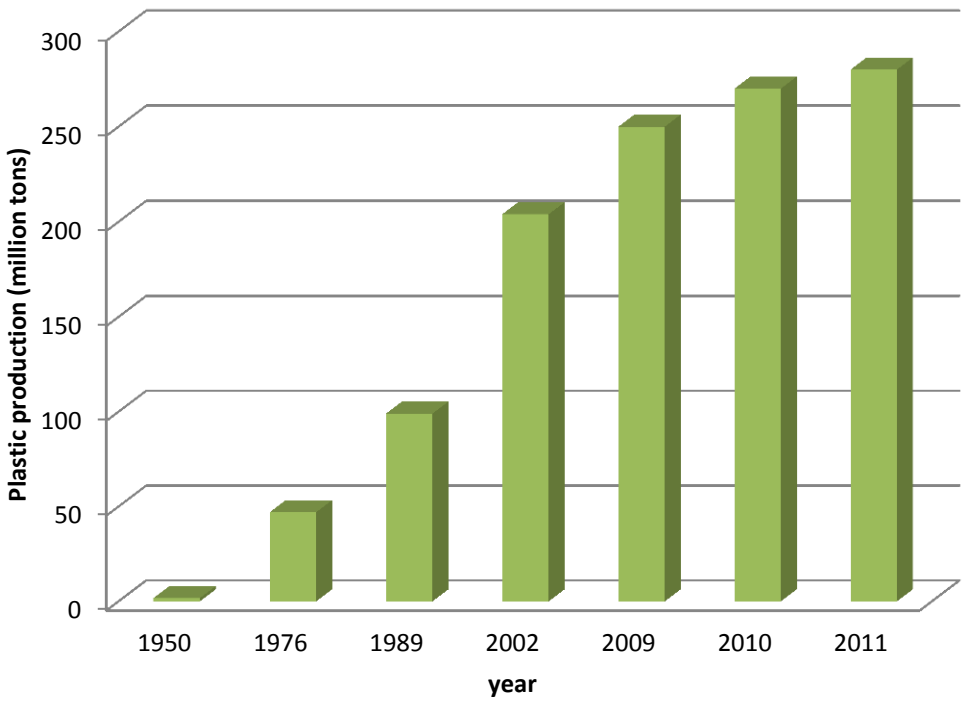

Figure 1-2 Plastic production in the world, adapted from [4].

1.2 Motivation

To reduce environmental impact and maintain sustainability, recycling should be the highest priority. However, the recycling rate is relatively low all over the world, as the data indicated in chapter 1-1. Unfortunately, the most common disposal method currently is landfill. Therefore, another way to reduce environmental impact of disposal is required. One answer is expanding the biodegradable plastic applications. There are two approaches to try to make biodegradable plastic; one is to use originally biodegradable plastic, such as polylactic acid (PLA) and polycaprolactone (PCL), and another way is to make conventional plastic biodegradable with prodegradant additives. Both ways have advantages and disadvantages. An

advantage of originally biodegradable plastics is their relatively high biodegradability. However, typically they are inferior to conventional plastics in properties, applications, or costs. Conversely, although conventional plastics with prodegradant tend to have relatively low biodegradability, they have large potential applications once sufficient biodegradability is achieved.

1.3 Goal and objectives

In this study, to overcome the environmental problem of plastic package disposal, especially targeting PET packages disposed in a landfill environment, the effect of a biodegradation additive for PET was investigated in anaerobic conditions. PET was selected as the test object because it is one of the most widely used materials in packaging, and anaerobic was selected as a test condition because it simulates a landfill environment, which is the most common disposal method.

CHAPTER 2

LITERATURE REVIEW

2.1 PET

PET is one of the most widely used plastics for packages, especially in the beverage industry as soft drink bottles. The structure of PET is shown in Figure 2-1. PET is produced by a condensation reaction between dimethyl terephthalate or terephthalic acid and ethylene glycol. The dimethyl terephthalate and terephthalic acid are converted from para-xylene. The ethylene glycol is converted from ethylene. PET is mainly processed by injection blow molding or injection stretch blow molding. Injection blow molding is used for production of small bottles such as pharmaceutical bottles. Injection stretch blow molding is used for the majority of PET bottles because the biaxial orientation improves mechanical properties. Extrusion blow molding is not suitable for PET due to its low melt strength. PET has acceptable barrier properties for oxygen and carbon dioxide [8]. Due to the light weight trend in many industries, the barrier properties of PET containers are being reduced, so barrier technologies such as coatings are being developed. Table 2-1 shows the major properties of PET.

To reduce the environmental impact of PET production, bio-based PET is being developed. The development of bio-based ethylene glycol has succeeded and is now used commercially for several companies' products, for instance, Dasani water bottles, produced by Coca-Cola Co [9]. PET consists of 30% ethylene glycol, and the remaining 70% is terephthalic acid. Commercialization of bio-based terephthalic acid has not yet succeeded, and is still in the pilot production level [10].

In 2012 in the U.S., 2,790 thousand tons of PET bottles and jars were produced, 860 thousand tons (30.8 %) were recycled, and 1,930 thousand tons were discarded [1]. In 2013,

1.1 trillion units of beverage packages were produced worldwide; 404.9 billion units were rigid plastics, and 93 % of the rigid plastics were PET bottles [11].

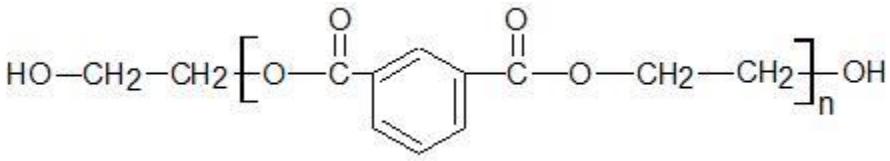

Figure 2-1 PET structure.

Table 2-1 Major properties of PET, adapted from [8].

T_g	73-80 ˚C (163-176 ˚F)
T_m	245-265 ˚C (473-509 ˚F)
Density	1.29-1.40 g/cm^3
Typical yield, 25 μm (1 mil) film	30 m^2/kg (21,100 in^2/lb)
Tensile strength	48.2-72.3 mPa (7.0-10.5 x 10^3 psi)
Tensile modulus	2,756-4,135 mPa (4-6 x 10^5 psi)
Elongation at break	30-3,000 %
Tear strength, film	30 g/25 μm (0.066 lb/mil)
WVTR	390-510 g μm/m^2 day at 37.8 ˚C, 90 % RH
	(1.0-1.3 g mil/100 in^2 24 h at 100 ˚F, 90% RH)
O_2 permeability, 25 ˚C	1.2-2.4 x 10^3 cm^3 μm/m^2 d atm
	(3.0-6.1 cm^3 mil/100 in^2 24h atm)
CO_2 permeability, 25 ˚C	5.9-9.8 x 10^3 cm^3 μm/m^2 d atm
	(15-25 cm^3 mil/100 in^2 24h atm)
Water absorption, 0.32 cm thick, 24h	0.1-0.2 %

2.2 Biodegradation

A biodegradable plastic can be defined as “a degradable plastic in which the degradation results from the action of naturally-occurring micro-organisms such as bacteria, fungi, and algae”, as stated in ASTM D883 [12]. The microorganisms degrade plastic to carbon dioxide, methane, or other small molecules. These transformations are caused by chemical reactions with enzymes which are produced by microorganisms. The biodegradation process can be divided into two steps, primary degradation and ultimate degradation. In the first step, primary degradation, chain scissions of the main backbone of the polymer occur and the polymer is converted into short polymer chains by hydrolysis and oxidation reactions. In the second step, ultimate degradation, the short polymer chains are converted into carbon dioxide, biomass and water [13]. Biodegradation occurs in two situations, aerobic, the main reaction in soil and compost, and anaerobic, primarily in landfill.

There are many factors which affect the biodegradation process. Table 2-2 shows a summary of the factors [14]. Increasing the temperature and moisture affects the biodegradation and hydrolysis reaction rates. However, temperature affects microorganism activity too, and if the temperature is too high, the activity is decreased; therefore, biodegradation has an optimal temperature range. pH affects the hydrolysis reaction rate and microorganism activity too. In addition, polymer characteristics are important for biodegradation. For example, in general, increase of molecular weight and crystallinity decrease biodegradability. The existence of crosslinking decreases biodegradability.

Table 2-2 Factors affecting Biodegradation, adapted from [14].

Factors affecting biodegradation	Exposure conditions	abiotic	Temperature
			Moisture
			pH
			UV radiation
		biotic	Extracellular
			Hydrophobicity
			Biosurfactants
	Polymer Characteristics		Flexibility
			Crystallinity
			Morphology
			Functional groups
			Crosslinking
			Molecular Weight
			Copolymers
			Blend
			Tacticity
			Additives

2.3 Anaerobic digestion

Anaerobic digestion consists of three steps. In the first step, the complex organic matter is hydrolyzed into soluble molecules by fermentative bacteria. In the second step, acid forming bacteria convert these molecules to simple organic acids, carbon dioxide and hydrogen; the principal compounds produced are acetic acid, propionic acid, butyric acid and ethanol. In the third step, methane is formed by methanogenic bacteria, either by breaking down the acids to methane (CH_4) and carbon dioxide (CO_2), or by bonding carbon dioxide with hydrogen to create methane and water [15]. Research on anaerobic digestion is conducted mostly in two environments: mesophilic conditions (approximately 35 ˚C), which simulates a landfill environment, and thermophilic conditions (approximately 55 ˚C), which simulates an anaerobic fermentation plant.

Yagi et al. conducted anaerobic digestion tests in both mesophilic and thermophilic conditions with four bioplastic powders: PCL, PLA, polyhydroxybutyrate (PHB) and polybutylenesuccinate (PBS). In mesophilic conditions (37 ˚C), the biodegradation rate was in the order of PHB >> PLA > PCL. PHB biodegraded 90 % in 9 days, PLA biodegraded 29 % and 49 % in two different runs at the same conditions in 277 days, and PCL biodegraded 3 % and 22 % in two different runs at the same conditions in 277 days. PBS did not degrade [16]. In thermophilic conditions (55 ˚C), the biodegradation rate was in the order of PHB >> PLA > PCL. PHB biodegraded 90 % in 14 days, PLA biodegraded 80 % in 50 days, and PCL biodegraded 75 % in 75 days. PBS did not degrade [17]. Yagi et al. also investigated the effect of sample size of PLA on biodegradation rate in thermophilic condition (55 ˚C), and found that small pieces of PLA film (25 μm) biodegraded more slowly than large pieces of PLA film, and PLA film biodegraded faster than PLA powder (125-250 μm) [18]. The authors explained that small pieces floated in the sludge, and if the activity of the upper layer of the sludge was lower than the bottom, the biodegradation rate of the small pieces could be slower. Also, the total surface area of PLA film was larger than PLA powder, so the biodegradation rate of the film was higher than the powder.

Hubackova et al. compared various starch types for PCL/starch blends in mesophilic conditions (35 ˚C), and concluded that PCL with starch plasticized with glycerol demonstrated a higher rate of biodegradation than PCL with pure starch [19]. Hermanová et al. researched polyethylene terephthalate-co-lactate copolyesters, produced from PET waste beverage bottles and L-lactic acid, and found that the biodegradability of the samples reached 34-69 % at 394 days in thermophilic conditions (55 ˚C) [20].

2.4 Landfill

Landfill can be said to be the bottom of the solid waste management hierarchy and the least desirable disposal method, but it is the only choice for residues from more effective ways, such as incineration. In addition, it is still the least expensive disposal method in countries which have large available area such as the U.S., and countries which do not have sophisticated disposal systems such as developing countries [21]. Landfilling has been conducted since the beginning of civilization. At first, there was no serious concern because the amount of waste was significantly less than in the modern era, and waste materials were organic. However, as industries expanded, the amount of waste, non-organic waste and non-biodegradable waste has increased. Pollution from landfill sites received much attention as environmental awareness grew. The lack of knowledge of treating landfill leachate allowed contamination of underground water with toxic substances, such as heavy metals. Generation of methane gas is hazardous because of its risk of explosion. Methane is also known as a greenhouse gas and the Intergovernmental Panel on Climate Change (IPPC) estimates its effect is 23 times greater than that of the same volume of carbon dioxide [22]. To overcome these concerns, modern landfill technology was developed [23].

Modern landfills have three features: a liner system, leachate collection system and methane collection system. First, to separate the trash and subsequent leachate from the ground water, a liner layer is made for protection on the bottom. The liner is made of clay, plastic (PE, PVC), or a combination of both. Second, to collect landfill leachate, perforated pipe is buried and the leachate is pumped up to a tank or pond. The leachate comes from the liquid content of garbage and ingress of water such as rain, and collects contaminants including hazardous substances as it percolates through the garbage. Therefore, leachate is strictly regulated by law and requires proper treatment. Typically, collected leachate is monitored to collect contaminant data and is recirculated or treated by wastewater treatment facilities. Third,

methane gas is generated by anaerobic bacterial activity and collected through pipes. Methane gas is used as energy for boilers and electricity generators, or simply flared [24][25].

Figure 2-2 shows the components of MSW after recycling and composting in the U.S. in 2012 [1]. Biomass materials, which were paper and paper board, yard trimmings, wood, and food waste, constituted 52.8 % of the MSW. Petrochemical, which was plastics, constituted 17.6 %. Rubber, leather, and textiles, which constituted 11.2 %, could be either biomass material or petrochemical. The average molecular structure of organic compounds in MSW can be shown as the molecular composition of $C_6H_{10}O_4$ [26]. After the waste is landfilled, organic components start to degrade to primary CH_4 and CO_2 by anaerobic biodegradation. This process can be shown in two representative reactions [15].

Acetogenesis

$$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2$$

Methanogenesis

$$CH_3COOH \rightarrow CH_4 + CO_2$$

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

More simply, the maximum amount of gas from organic compounds in anaerobic digestion can be shown in the following equation [15].

$$C_6H_{10}O_4 + 1.5H_2O \rightarrow 3.25CH_4 + 2.75CO_2$$

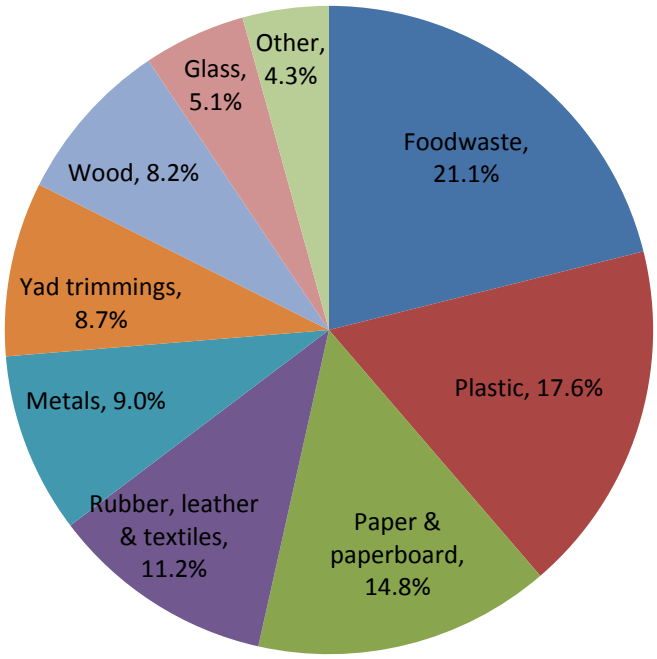

Figure 2-2 The components of MSW after recycling and composting in 2012 in the U.S., adapted from [1].

2.5 Biodegradable plastics

Research on biodegradable plastics has been an active topic since the 1970's, motivated by the increase of plastic waste and environmental concerns. In the early years of the research, biodegradable plastic was not the primary goal, but disintegration of plastic was the goal to save landfill space. Copolymers of conventional polyolefins with starch, metal oxides, or metal salts were studied, but these can only disintegrate into small chips. As plastics became a serious concern, truly biodegradable plastics, such as PCL, PBS, polybutylene succinate-co-adipate (PBSA), and polybutylene adipate-co-terephthalate (PBAT) were studied. Biodegradable plastics can be categorized to three groups by their origins: naturally occurring

biodegradable polymers, biodegradable polymers derived from renewable resources, and biodegradable polymers derived from petroleum [27].

Naturally occurring biodegradable polymers have a long history. They have been used from ancient times as skin of animals, plant fibers, silk, etc. At first, these polymers were not used because of high cost compared with conventional plastics. However, as the awareness of environmental pollution and depletion of fossil oil increased, the research expanded. Starch, cellulose, soy protein plastics, and sugar beet pulp plastics can be categorized in this group. Especially, starch-based plastic has the second largest share in the world - 41 % in 2012 - in the biodegradable plastic market, and it was the largest share in Europe with 62 % of the market [28]. For instance, starch-based plastic was used in plastic bags and cushion packaging.

In the group of biodegradable polymers derived from renewable resources, PLA and the polyhydroxyalkanoates (PHAs) are the most important polymers. These polymers are not found in nature or not available in commercially beneficial form or quantity, but can be produced from naturally occurring bioresources. Especially, PLA is the most widely used and studied biodegradable polymer due to its thermal characteristics, which make it possible to use existing process equipment, and the cost is competitive when compared with petroleum-based polymers. PLA had the largest share (47%) in the biodegradable plastic market in 2012 [28].

Typically, synthetic polymers are resistant to biodegradation, though natural polymers are relatively susceptible to biodegradation. However, there are some polymers that are biodegradable and petroleum-based. Examples include PCL, PBS, and PBAT. PCL and PBS are synthetic aliphatic polyesters, and PBAT is an aliphatic-aromatic copolymer [27].

2.6 Prodegradant additives for biodegradation

Since conventional plastics are usually not biodegradable, many attempts have been made to make synthetic plastic biodegradable using additives. Prodegradant technology can be categorized into three groups: transition metal salts, carbonyl containing copolymers, and chemo-taxis approaches [29]. Most prodegradants use an oxo-biodegradation mechanism. Oxo-biodegradation is a combination of biodegradation and oxidation by mostly photo degradation or thermal degradation. By photo or thermal degradation, it is claimed that the molecular weight of the polymer is reduced and the polymer becomes easy to biodegrade with enzyme reactions by microorganisms.

Transition metal salts are the most widely used prodegradants for polyolefins. Commercially available prodegradants in this group include TDPA® [30], Reverte™ [31], AddiFlex® [32], d_2w [33] and P-Life [34]. TDPA® (EPI Environmental Products Inc., Vancouver, Canada) works by two stages of oxo-biodegradation process. According to the company's claim, in the first stage, the long polymer molecules are reduced to shorter lengths by oxidation with heat, UV light and mechanical stress. With oxidation, the molecules become hydrophilic and small enough to be ingested by micro-organisms. In the second stage, biodegradation occurs by microorganism digestion. TDPA® can be used for polyethylene (PE), polypropylene (PP) and polystyrene (PS) [30]. Active components of TDPA® are metal stearates (Fe, Ce, Co) and citric acid (typically Co) [29]. Reverte™ (Wells Plastics Ltd, UK) also works by an oxo-biodegradation process. According to the company's claim, in the oxidation phase, polymer molecular weight is reduced and oxygen is introduced into the structure. In the biodegradation phase, the lower molecular weight polymer is converted into biomass, CO_2 and H_2O by microorganisms. Reverte™ is designed for PE, PP and PET [31]. Active components of Reverte™ are an undisclosed photo-inhibiting package, a metal ion prodegradant package, and biodegradation promotors (micronized cellulose) [29]. AddiFlex® (Add-X Biotech AB, Sweden) also works by

oxo-biodegradation and is used for PE according to the company's claim [32]. Active components are metal carboxylate (Fe, Mn, Cu, Co, Ni), starch, and calcium carbonate ($CaCO_3$). Especially, $CaCO_3$ plays an important role and increases UV degradation by up to 66% [29]. d_2w (Symphony Environmental, UK) is an oxo-biodegradable additive which breaks molecular chains by a process of oxidation, accelerated by light, heat and stress, according to the company's claim. Applications are PE and PP [33]. The active components are metal stearates and stabilizers (typically Mn) [29]. P-Life (Programmable Life Inc., U.S.) is an oxo-biodegradable additive based on a manganese salt and used for low density polyethylene (LDPE) [34]. Jakubowicz et al. used P-Life for their research on thermally oxidized LDPE in soil (23 ˚C) and in a compost environment (58 ˚C) , and found 91 % mineralization in soil and 43 % mineralization in a compost test after two years [35]. They explained that the reason that higher mineralization was found in the lower temperature (soil) was due to the difference of microorganism population between the test environments.

Carbonyl containing copolymers can be divided two groups: carbon monoxide copolymers and vinyl ketone copolymers. The carbon monoxide is known as poly(ethylene- co-carbon monoxide), which is used for six-pack carrier rings for beverage cans and bottles. The carbonyl group absorbs UV light and breaks polymer chains to short segments by a Norrish II reaction [29]. Vinyl ketone copolymer is commercially available as Ecolyte (Ecoplastic ltd, Ontario, Canada) [36]. Just like carbon monoxide, the carbonyl group in ketone copolymer absorbs UV light and induces photodegradation. However, these are not claimed to work for biodegradation.

The chemo-taxis approach uses organic additives, and it is claimed to attract microorganisms by providing food in the additive to digest the polymer more quickly. Commercially available additives in this group are Eco-One™ [37], EcoPure® [38], Omnidegradable™ packaging [39], and ENSO RESTORE™ [40]. Eco-One™ (Ecologic LLC, WI,

U.S.) is an organic additive, and according to the company's claim, the ingredients in Eco-One™ allow microorganisms to form a coating (biofilm) on the surface of the plastic, and other ingredients in Eco-One™ work together to expand the molecular structure to make room for microorganisms. The microorganisms attract additional microorganisms and break down the chemical bonds of the polymer. Eco-One™ attracts oleophilic bacteria, which exist in landfills. Eco-One ™ is compatible with PE, polyurethane (PU), PP, PET, PS, nylon, ethylene vinyl acetate (EVA), acrylonitrile butadiene styrene (ABS), polyvinyl chloride (PVC), polycarbonate, and ethylene vinyl alcohol (EVOH) [37]. EcoPure® (Bio-Tec Environmental LLC, NM, U.S.) is an organic additive, and according to the company's claim, the additive acts as a catalyst when it is exposed to enzymes, so that microorganisms will penetrate the plastic. Other ingredients expand the molecular structure, and make room for microorganisms, and microorganisms attract other microorganisms by a chemical signal, quorum sensing. At the signal, microorganisms gather to a food source and break down chemical bonds. EcoPure® can be used for EVA, PET, PE, PP, PS, and nylon [38]. According to Anne et al. [29] and US patent 2008103232 [41], assignee Bio-Tec Environmental LLC, the ingredients of chemo attractants are based on furanone, and the swelling agents are natural fibers or cultured colloids. In addition, there are essential components of glutaric acid, a hexadecanoic acid compound, and polycaprolactone in a carrier resin (EVA). Furanone compounds are 3,5-dimethyl-pentenyl-dihydro-2(3H)-furanone isomer mixtures, emoxyfurane and N-acylhomoserine lactones (Fig 2-3). Furanones contain carbonyl structures and can act as UV absorbers. Halogenated furanones are excluded from furanone compounds because they act as quorum sensing inhibitors. Furthermore, non-esterified starch is also listed as a chemo attractant. Omnidegradable™ packaging (TekPak Solutions,Ontario, Canada) is an organic additive, and according to the company's claim, it works in landfill, soil or water. ENSO RESTORE™ (ENSO Plastics LLC, Mesa, AZ) is an organic additive and it is claimed to induce the production of an extra-cellular enzyme from certain microorganisms. The enzyme works as the catalyst for depolymerizing the

plastic material, and makes polymers biodegradable. ENSO RESTORE™ is designed for PET, HDPE, LDPE, PE, PP, EVA, PS, nitrile, rubber and latex [40].

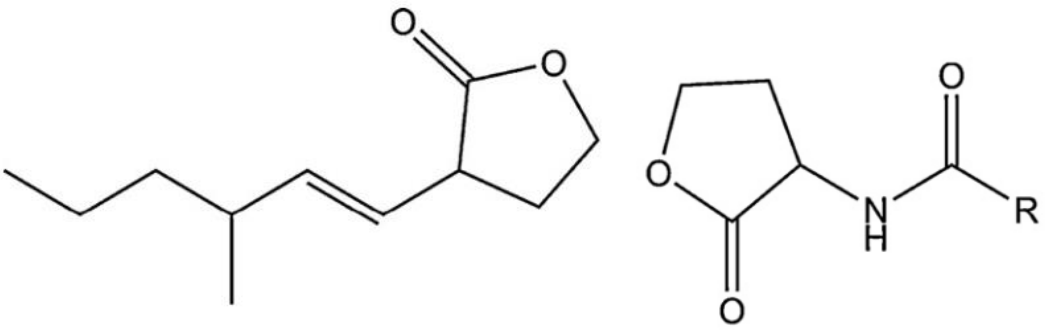

Figure 2-3 Chemical structure of furanone compounds, 3,5-dimethyl-pentenyl-dihydro-2(3H)-furanone (left) and N-acrylhomoserine lactone (right).

2.7 Test standards for anaerobic digestion

Several test standards about anaerobic digestion are defined by ASTM and ISO. The followings is a list of standards, and table 2-3 shows a summary of the ASTM standards.

- ASTM D5210 − 92: Standard Test Method for Determining the Anaerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge [42].
- ASTM D5511 – 12: Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under High-Solids Anaerobic-Digestion Conditions [43].
- ASTM D5526 – 12: Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under Accelerated Landfill Conditions [44].
- ASTM D7475 – 11: Standard Test Method for Determining the Aerobic Degradation and Anaerobic Biodegradation of Plastic Materials under Accelerated Bioreactor Landfill Conditions [45].

- ISO 13975:2012: Plastics -- Determination of the ultimate anaerobic biodegradation of plastic materials in controlled slurry digestion systems -- Method by measurement of biogas production [46].
- ISO 14853:2005: Plastics -- Determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system -- Method by measurement of biogas production [47].
- ISO 15985:2014: Plastics -- Determination of the ultimate anaerobic biodegradation under high-solids anaerobic-digestion conditions -- Method by analysis of released biogas [48].

Table 2-3 Summary of the ASTM standards for anaerobic tests.

ASTM	D5210-92	D5511-12	D5526-12	D7475-11
Simulated environment	Anaerobic digester municipal sewage sludge	High-solids anaerobic-digestion	Accelerated landfill conditions	Change of environment from aerobic to anaerobic as depth of landfill increases
Temperature	35 ± 2 ˚C	37 ± 2 or 52 ± 2 ˚C	35 ± 2 ˚C	35 ± 2 ˚C
pH	-	7.5 - 8.5	7.5 - 8.5	7.5 - 8.5
Solid content	At least 1 to 2 %	Over 20%	35, 45, and 60%	35, 45, and 60%
Inoculum	Anaerobic-sludge digester	Anaerobic digester with pretreated household waste as a sole substrate	Anaerobic digester with pretreated household waste as a sole substrate	Anaerobic digester with pretreated household waste as a sole substrate
Other components	Stock solution	-	Pretreated household waste	Household waste and pretreated household waste
Digester	Serum bottle (approximately 160 mL)	Erlenmeyer flask	Pressure resistant glass vessels (4L to 6L)	Pressure resistant glass vessels (4L to 6L)
Gas measurement method	Volume	Volume	Pressure increase	Pressure increase
Incubation	In the dark	In the dark or diffused light	In the dark	In the dark
Blank	Inoculum medium	Inoculum only	Inoculum with pretreated household waste	Inoculum with pretreated household waste
Negative control	-	Polyethylene (optional)	Polyethylene (optional)	Polyethylene (optional)
Positive control	-	Cellulose	Cellulose	Cellulose
Frequency of measurements	Sufficient number	Five times per week	At least weekly	At least weekly
Number of replicates	Three	Three	Three for each solid content (9 vessels for each samples)	Three for each solid content (9 vessels for each samples)

CHAPTER 3

MATERIALS AND METHODS

3.1 PET production process

Neat PET sheet, PET sheet with 1 wt% additive, and PET sheet with 5 wt% additive were prepared for this study. PET resin, Laser+® W 4000 (K42A) grade, which is designed for water bottles, was obtained from DAK Americas LLC (Chadds Ford, PA). The masterbatch of the biodegradation promoting additive was obtained from ENSO Plastics (Mesa, AZ), and ENSO RESTORE™ PETG was used for this study. The required amount of PET resin and additive were mixed and placed into a vacuum oven. The vacuum oven was set at 120 ˚C and -30 inHg. Resins were dried for 24 hours under this condition, then cooled to room temperature and kept in vacuum conditions until they were used.

PET sheet was produced using a Microtruder model RCP-0625 extruder (Randcastle Extrusion Systems, Inc., Cedar Grove, NJ) (Figure 3-1). The temperature profile of the extruder was 282-310-310-310-310 ºC (540-590-590-590-590 ºF) for zone 1, zone 2, zone 3, transfer tube, and die, respectively. The screw speed of the extruder was 500 rpm. To reduce crystallinity of the sheets, the chill roll temperature was controlled at 21 ºC (70 ºF) and placed close to the die exit so that the sheets were chilled rapidly. The speed of the chill roll was 20 rpm. Table 3-1 shows the thickness of the sheets.

The composition of the test sheets was measured by CHN analyzer (Perkin Elmer, Waltham, Massachusetts). The data are shown in appendix A.

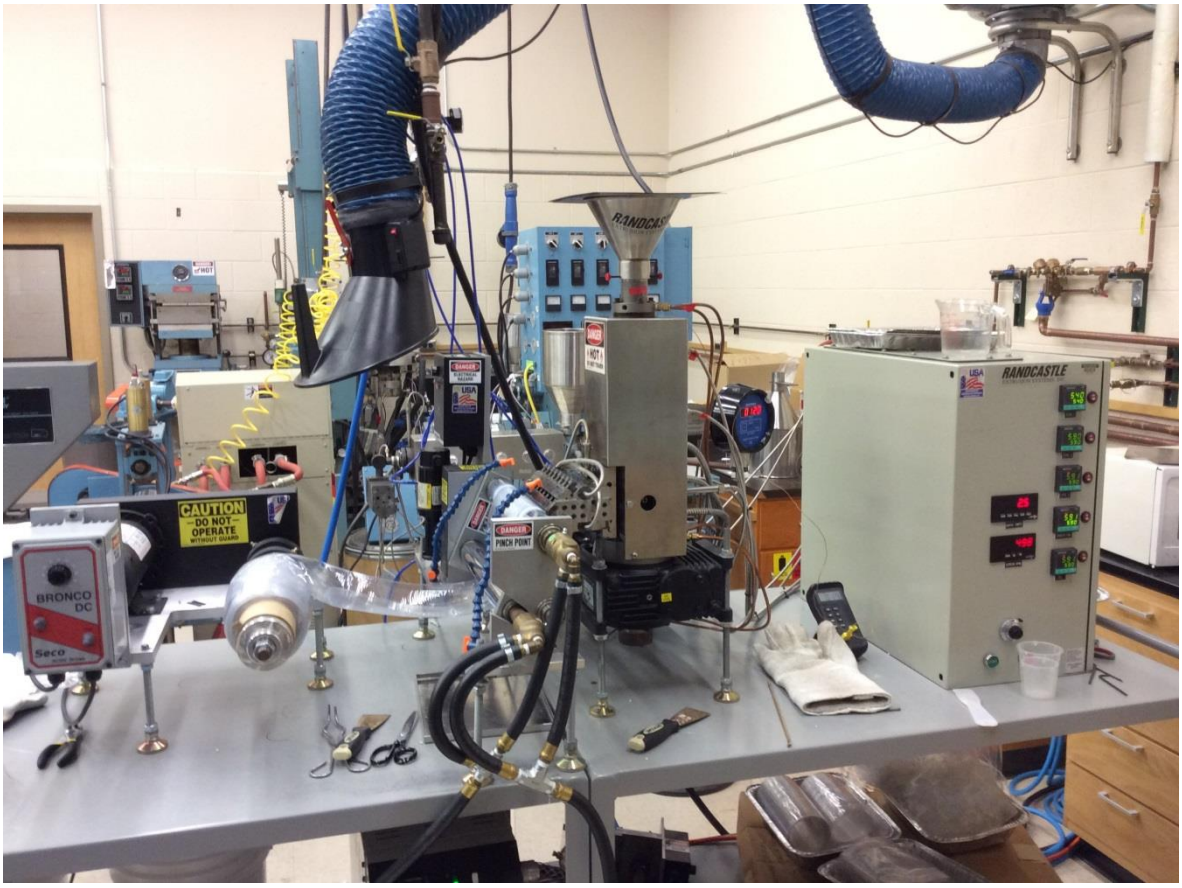

Figure 3-1 Cast film extruder in the School of Packaging lab.

Table 3-1 Thickness of test sheets.

Thickness (mil)	Average	Minimum	Maximum	σ
Neat PET	4.3	3.4	4.9	0.41
PET with 1% additive	4.4	3.5	5.5	0.62
PET with 5% additive	5.2	4.0	6.1	0.70

3.2 Anaerobic digestion system

A simulated landfill anaerobic digestion system was established for this experiment. 125 mL glass bottles were selected as bioreactors. The caps for the bottles had chlorobutyl rubber plugs in the center of the tops. Needles could be inserted into the bottles through the rubber plug to add or remove contents without exposing the contents to the outside air. Therefore, inside the bottle was maintained as an anaerobic environment throughout the experiment.

Next, inoculum, manure and test samples were placed in the bottles. The inoculum was used as a seed source of anaerobic microorganisms. Three different inoculums, obtained from landfill leachate, wastewater treatment residue, and an anaerobic digester, were used in this experiment to investigate how the microorganism population affected biodegradability of the test samples. The landfill leachate inoculum was obtained from a landfill site of Granger LLC (Lansing, MI). The wastewater treatment residue inoculum was obtained from Delhi Charter Township Wastewater Treatment Plant (Holt, MI). The anaerobic digester at Michigan State University provided the anaerobic digester inoculum. Fresh cow manure was obtained from the Michigan State University dairy farm. The purpose of the manure was to provide necessary nutrition for microorganism activity. Liquid manure (5 wt % total solids content) was used for this experiment, since a lower solids content would lead to higher gas generation [49]. Chemical oxygen demand (COD) of the inoculums and manure were measured by a DR 2800 Portable Spectrophotometer and Digestion Solution for COD 20-1500 mg/L Range (HACH Company, Loveland, Co). Two measurements were conducted for each sample. Table 3-2 shows the COD values.

After the inoculum, manure, and test samples were placed in the bottles, the air in the headspace of the bottles was replaced by nitrogen gas to make the environment anaerobic. The bioreactors were maintained at 35 ºC to simulate actual landfill temperatures. Throughout the

experiment, microorganisms degraded the manure and test samples, and produced gas which was mainly CO_2 and CH_4. The produced gas was collected by a needle which was inserted into the bottle, so that the inside of the bottle maintained the anaerobic environment.

Table 3-2 COD values.

		COD (g/L)	Average COD (g/L)
Manure (5 wt % solid content)	1	32.0	28.7
	2	25.4	
Landfill leachate	1	1.00	1.00
	2	0.99	
Wastewater treatment residue	1	23.2	23.0
	2	22.7	
Anaerobic digester	1	49.5	48.8
	2	48.1	

3.3 Sample preparation

PET sheets were cut into 0.5 in x 0.5 in (1.27 cm x 1.27 cm) squares with a sample cutter and scissors. Five test standards, which are negative control, positive control, Neat PET, PET with 1 % additive, and PET with 5 % additive, were prepared. The negative control contained only manure and inoculum, and was used as a blank. For the positive control, cellulose powder, which was obtained from Sigma-Aldrich Co. LLC (St. Louis, MO), was used as a test sample. The purpose of the positive control is to confirm the biodegradation capability of the system, since cellulose is known to be a biodegradable polymer. Manure was mixed with distilled water to 5 wt % solid content and homogenized by a blender. Appendix B shows the water content and organic content of the original manure. Next, three different inoculums were placed in the bioreactors with test samples and manure. Three bioreactors were prepared for

each test sample. Tables 3-3 and 3-4 show the amount of material and number of test samples. In total, 45 bioreactors, which included five test samples and three different inoculums, were prepared.

After the bioreactors were closed tightly, the air in the head space of the bioreactors was replaced with 100% nitrogen gas through two needles, which were inserted into the bottles through the caps. One needle was connected to a nitrogen cylinder and the other was for the gas to exit. Next, the bioreactors were placed in the oven, which was maintained at 35 ± 2 ºC throughout the experiment. Appendix C shows the temperature control capability of the oven. After an hour, the bioreactors were removed from the oven, and the inside gas was released through the needle to neutralize the initial pressure difference, which was caused by the increase of the temperature of the bioreactors from room temperature to 35 ºC. Then, the experimental measurements began.

Table 3-3 Amounts of materials in a bioreactor.

	Inoculum (mL)	Manure (mL)	Cellulose (g)	PET sheet (g)
Blank (Negative control)	7.5	75.0		
Cellulose (Positive control)	7.5	75.0	0.550	
Neat PET	7.5	75.0		3.00
PET with 1 % additive	7.5	75.0		3.00
PET with 5 % additive	7.5	75.0		3.00

Table 3-4 Number of samples.

	Inoculum		
	Landfill leachate	Wastewater treatment residue	Anaerobic digester
Blank (Negative control)	3	3	3
Cellulose (Positive control)	3	3	3
Neat PET	3	3	3
PET with 1 % additive	3	3	3
PET with 5 % additive	3	3	3

3.4 Gas measurement

The generated gas in the bioreactors was measured by a syphon system. Figure 3-2 shows the gas measurement system for the experiment. A 500 mL glass bottle was prepared and filled with water to about 80 % of maximum volume. The same cap as the bioreactor was used for the 500 mL glass bottle. Two needles were inserted into the 500 mL bottle. One was a short needle and the tip was in the head space of the bottle. The other was a long needle and the tip was on the bottom of the glass bottle. Both needles were connected to plastic tubes and the other end of the short needle tube had another needle which was inserted into the bioreactors. The other end of the long needle tube was inserted into a graduated cylinder. The internal pressure of the bioreactor was higher than atmospheric pressure because of the generated gas, so once the needle was inserted into the bioreactor, the gas moved to the head space of the 500 mL glass bottle. The head space of the 500 mL glass bottle was pressurized by the increase in the volume of gas, and pressurized the water in the bottle, which moved

through the long needle and came out into the graduated cylinder. This continued until the internal pressure of the bioreactor became equal to atmospheric pressure, and at this point, the volume of the generated gas was measured in the graduated cylinder as volume of the water. The measurement takes approximately three to five minutes, depending on the volume of the generated gas. The 500 mL glass bottle was refilled after the water volume became less than 40 % of the maximum volume. To reduce total measurement time, three sets of the same system were established and run simultaneously. Three bioreactors were removed from the oven at one time to avoid decrease in temperature. Gas leakage was checked by pouring detergent liquid on the connections such as between the rubber plug of the cap and the needles, and the needles and tubes so that if there was a leak, it was detected as bubbles.

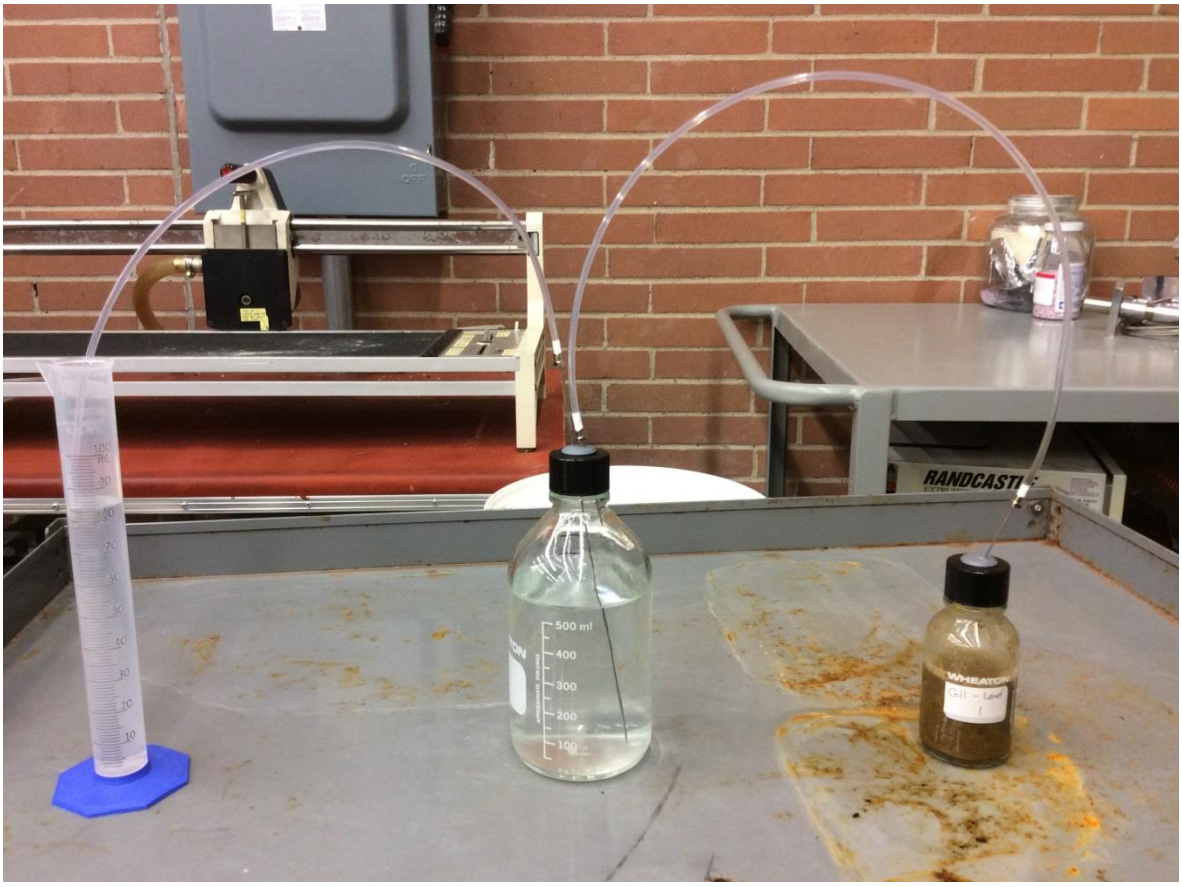

Figure 3-2 Gas measurement system.

3.5 pH adjustment

The pH of the bioreactors was checked and maintained close to pH 7 throughout the experiment. The initial pH of the inoculums, manure, and bioreactors were measured using a pH meter (PHB-212, OMEGA Engineering Inc., Stamford, CT). Table 3-5 shows the pH data for each of these test materials. During the experiment, pH could not be measured using a pH meter because opening the cap would destroy the anaerobic environment. Therefore, pH was measured by a syringe and pH strips (Hydrion® pH 6.0-8.0, Micro Essential Laboratory Inc., Brooklyn, NY). Approximately 0.1 mL of liquid was removed from the bioreactor using a 1 mL syringe, and deposited on a pH strip. For each measurement, one of three bioreactors in each test set was selected as a representative and measured. Once the pH strip indicated the pH had fallen below 6, a 10 wt % sodium hydroxide (NaOH) solution was added to the bioreactors. According to a titration test conducted before the experiment, from pH 6 to pH 7, approximately 1 mL of 10 % wt NaOH solution was required for the 82.5 mL bioreactors. Appendix D shows the pH adjustment record throughout the experiment. The maximum amount of NaOH added to the sample was 2.5 ml, which equaled 3.03 g/L. This value was less than the half maximal inhibitory concentration (IC_{50}), 5.6 to 53 g/L, which reduces cumulative methane production by half [50].

Table 3-5 Initial pH of samples.

	pH
Manure (5 wt % solid content)	7.79
Landfill leachate	7.21
Wastewater treatment residue	7.18
Anaerobic digester	7.25
Manure (5 wt % solid content) + landfill leachate	7.77
Manure (5 wt % solid content) + wastewater treatment residue	7.75
Manure (5 wt % solid content) + anaerobic digester	7.61

3.6 Estimated gas production

The estimated gas production can be calculated with two methods: from COD values and CHN values. By using COD values in table 3-2, estimated gas production was calculated with the following equation,

$$\mathrm{V_{gas}\ (mL)} = \frac{COD\ \left(\frac{\mathrm{g}}{\mathrm{L}}\right) \times COD\ reduction\ (\%) \times 0.395\ \frac{L\ \mathrm{CH4}}{1\ \mathrm{g\ COD}} \times (V_{sample})(\mathrm{mL})}{V_{methane}/V_{total}\ (\%)}$$

V_{gas}: estimated gas production (mL)

COD: COD values (g/L)

COD reduction: percentage of COD reduction (%)

V_{sample}: volume of the sample (manure:75 mL, inoculum:7.5 mL)

$V_{methane}/V_{total}$: the percentage of methane in total gas (%)

Here, the COD reduction rate is assumed to be 30%. The volume of methane produced per 1 g COD at 35 ˚C 1 atm, was assumed to be 0.395 L [51]. The percentage of methane in the total gas was assumed to be 60 %. Table 3-6 shows the estimated gas production.

The estimated gas production was also calculated from CHN values in appendix A by the following equation.

$$\mathrm{V_{gas}\ (mL)}$$

$$= \frac{solid\ weight\ (\mathrm{g}) \cdot \frac{weight\ percent\ of\ carbon\ (\%)}{100} \cdot 22{,}400\left(\frac{\mathrm{mL}}{\mathrm{mol}}\right) \cdot \frac{(273.15 + 35)\ \mathrm{K}}{273.15\ \mathrm{K}} \cdot \frac{biodegradation\ (\%)}{100}}{12(\mathrm{g/mol})}$$

Here, the solid weight of 75 mL manure was 3.75 g (5 wt%), and the solid weights of the 7.5 mL inoculums were obtained from table B-2 in appendix B. Carbon was assumed to produce either methane or carbon dioxide. The percentage of biodegradation of the manure and inoculums was assumed to be 30%. Table 3-6 shows the estimated gas production.

The differences in the estimated gas calculated from the COD values and CHN values are presumed to come from the relatively low COD values of the manure. The COD was measured with 2 ml of diluted liquid sample, for manure, it was diluted by 100 times. Therefore, lack of complete homogenization of the liquid manure may have caused the low COD value.

Table 3-6 Estimated gas production.

Estimated gas production (mL)	From COD values	From CHN values
Manure (5 wt% solid content) 75 mL	425.1	1029.0
Landfill leachate 7.5 mL	1.5	1.8
Wastewater treatment residue 7.5 mL	34.0	24.2
Anaerobic digester 7.5 mL	72.3	53.6
Manure 75 mL + landfill leachate 7.5 mL	426.6	1030.8
Manure 75 mL + wastewater treatment residue 7.5 mL	459.1	1053.2
Manure 75 mL + anaerobic digester 7.5 mL	497.4	1082.5

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Cumulative gas volume

By following the gas measurement method in chapter 3.4, gas evolution, which produces primarily CO_2 and CH_4, from each bioreactor, was measured and cumulative gas volume was calculated. Table 4-1 shows the average cumulative gas volume of three replicates of each test sample at 90 days. Original data for each replicate are shown in appendix E. Figures 4-1, 4-2, and 4-3 show a comparison of the cumulative gas between test samples in different inoculums, which are landfill leachate, wastewater treatment residue, and anaerobic digester. One of the blank samples with landfill leachate got broken accidentally after the measurement at 75 days, thus the average of two replicates was calculated for the blank of landfill leachate after 75 days.

Judging from table 4-1, and figures 4-1, 4-2, and 4-3, test samples with wastewater treatment residue and anaerobic digester show much higher gas production than landfill leachate. This is presumed due to the different microorganism activities in each inoculum. Cellulose samples produced higher gas production than blanks in every inoculum. However, PET with additive samples did not show higher gas production than blank and neat PET in any inoculum.

Compared to the estimated gas productions in chapter 3-6, actual gas productions of the blank samples in every inoculums were higher. It is presumed that the COD reduction rate and percentage of biodegradation of the manure and inoculums were higher in the experiment than the 30 % assumed in the calculation.

Table 4-1 Average cumulative gas volume at 90 days.

		Average cumulative gas volume (mL)
Landfill leachate	Cellulose	1189.3
	Neat PET	1017.0
	PET with 1% additive	1025.8
	PET with 5% additive	1071.2
	Blank	1018.8
Wastewater treatment residue	Cellulose	1448.0
	Neat PET	1161.0
	PET with 1% additive	1188.7
	PET with 5% additive	1170.0
	Blank	1176.3
Anaerobic digester	Cellulose	1418.8
	Neat PET	1273.2
	PET with 1% additive	1251.7
	PET with 5% additive	1227.2
	Blank	1226.7

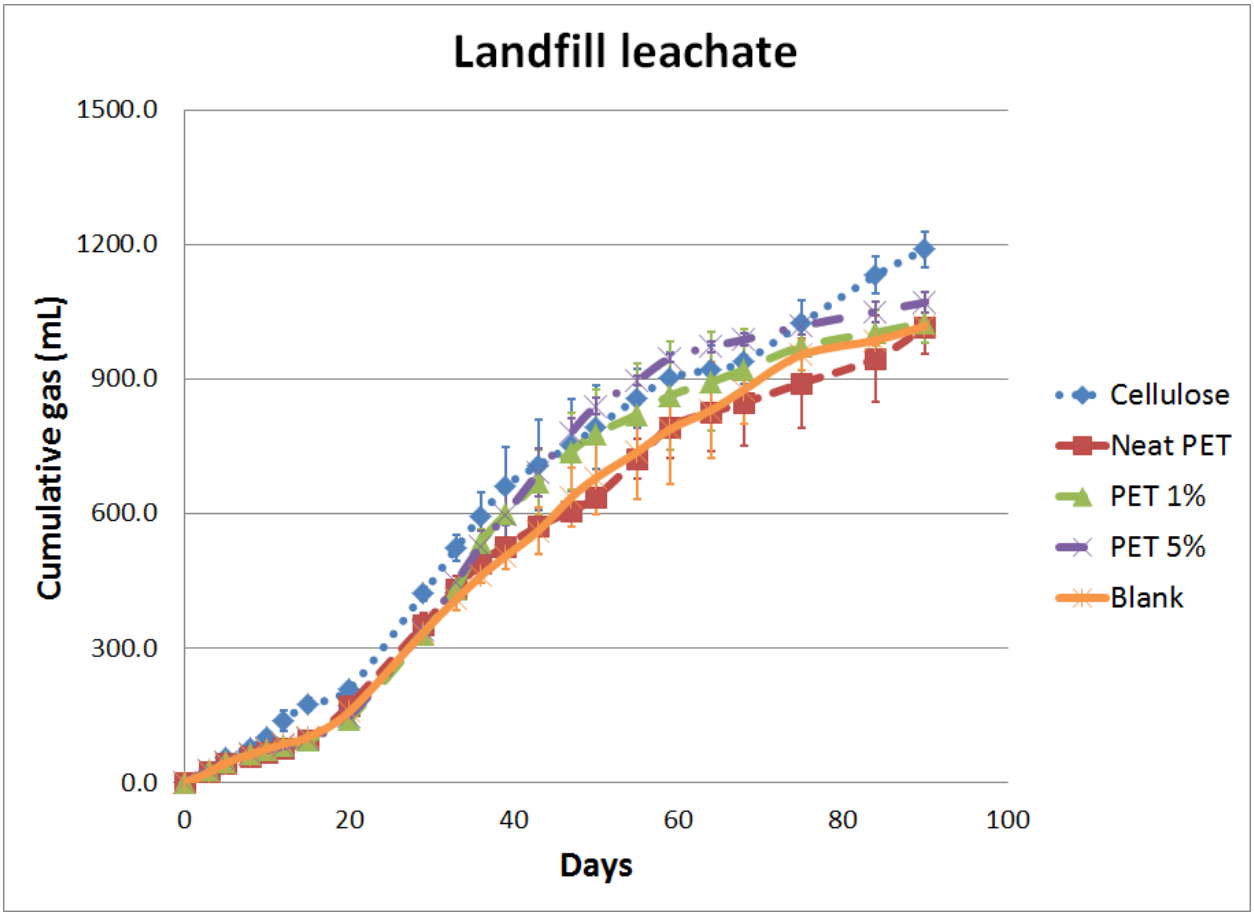

Figure 4-1 Cumulative gas of test samples with landfill leachate.

Note: error bars show standard deviations.

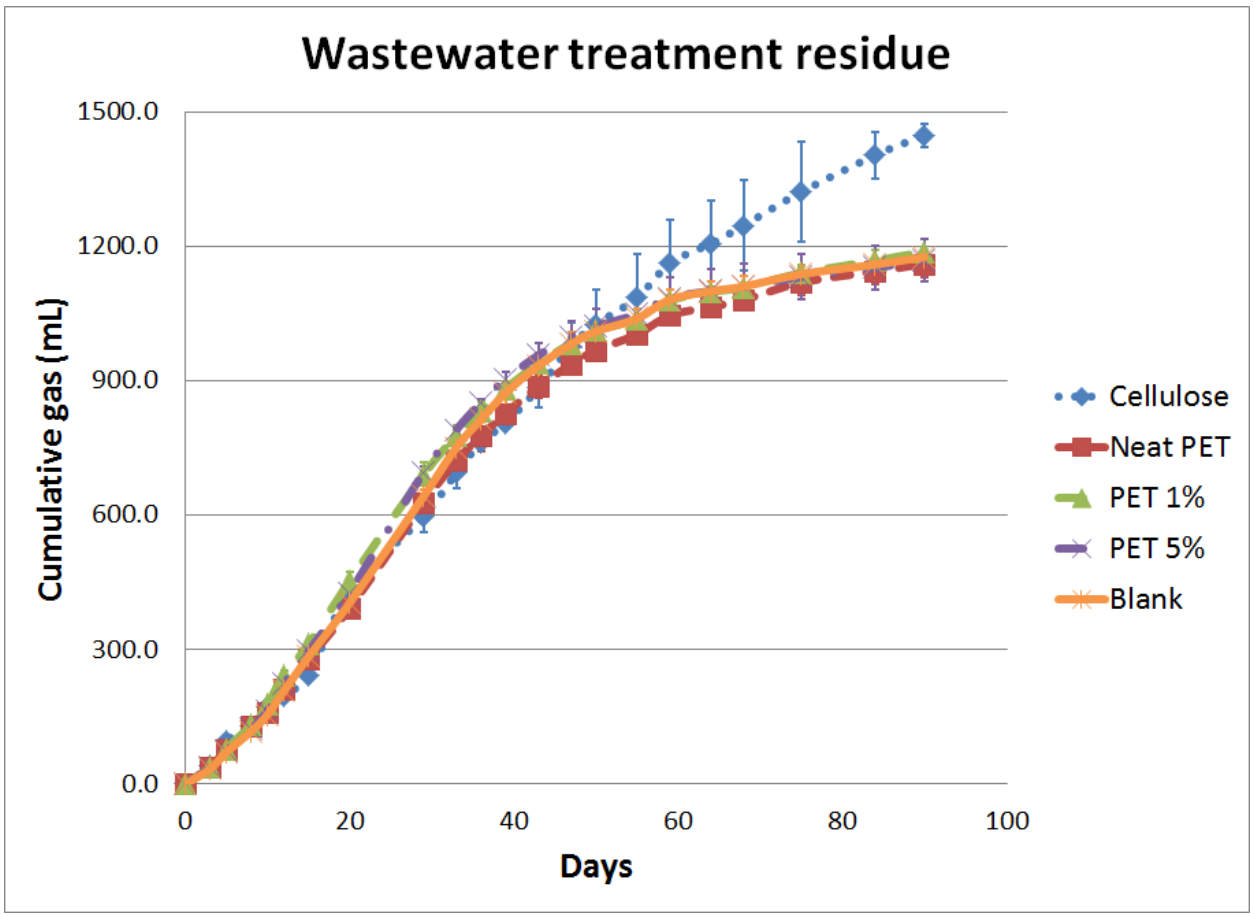

Figure 4-2 Cumulative gas of test samples with wastewater treatment residue.

Note: error bars show standard deviations.

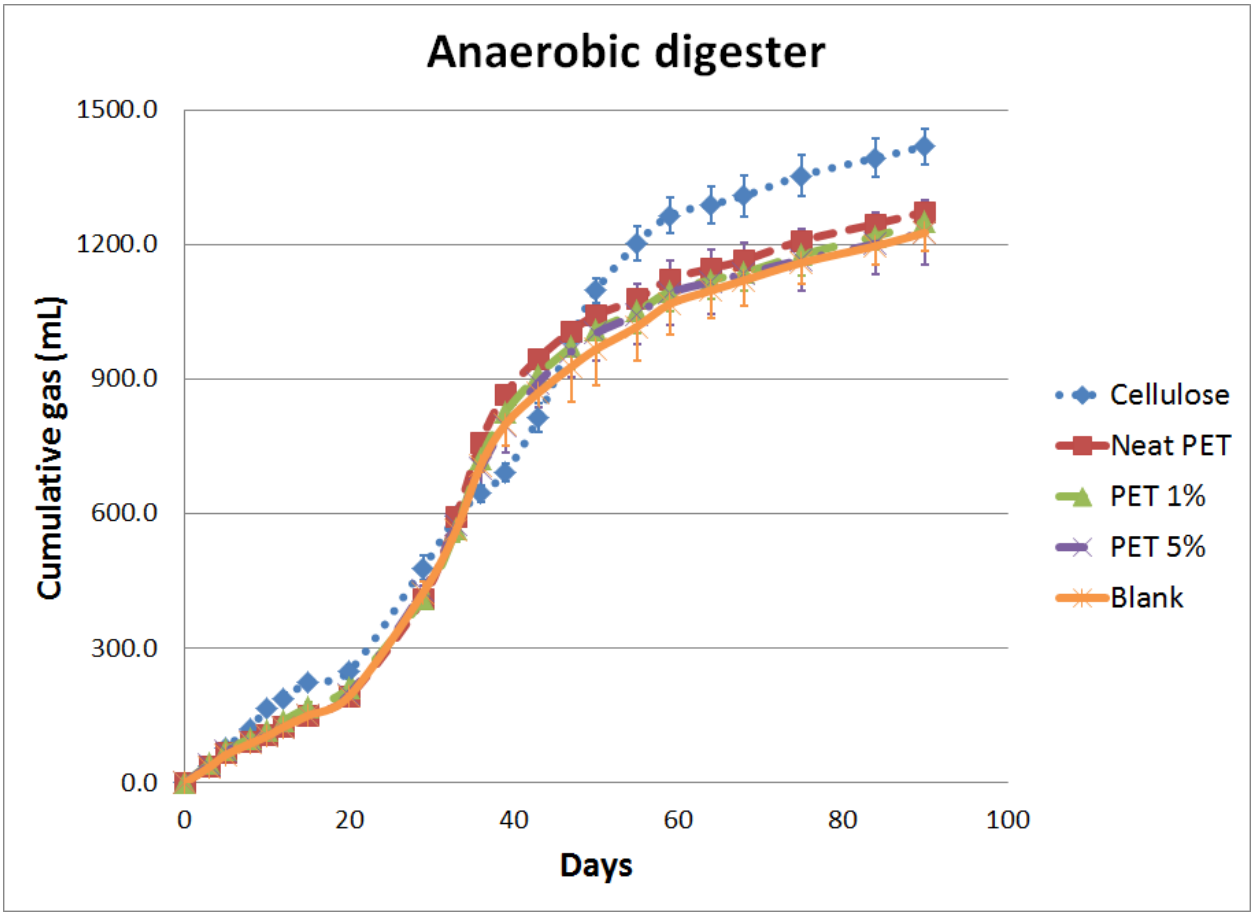

Figure 4-3 Cumulative gas of test samples with anaerobic digester.

Note: error bars show standard deviations.

4.2 Biodegradation extent

To normalize the difference in gas evolution between the test samples and blank, the biodegradation extent was calculated with the following equation.

$$\text{Biodegradation extent (\%)} = \frac{\text{average } C_g(\text{test sample}) - a\text{verage } C_g(\text{blank})}{C_i} \times 100$$

Here,

C_g: weight of produced gaseous carbon (g)

C_i: weight of carbon in test samples (g)

Assuming that the produced gas contains only CO_2 and CH_4, C_g is calculated with the following equation, with correction for standard temperature and pressure (STP) by the incubator temperature (35 ˚C) and atmospheric pressure at East Lansing (860ft, 0.96 atm).

$$Cg(\mathrm{g}) = \frac{C_v\ (\mathrm{mL})}{22{,}400(\mathrm{mL/mol}) \times \frac{(273.15+35)\ \mathrm{K}}{273.15\ \mathrm{K}} \times \frac{1\mathrm{atm}}{0.96\ \mathrm{atm}}} \times 12(\mathrm{g/mol})$$

Here,

C_v: volume of produced gas (mL)

From the data measured by CHN analyzer (Appendix A), C_i is calculated with the following equation,

$$C_i(\mathrm{g}) = \frac{sample\ weight\ (\mathrm{g}) \times weight\ percentage\ of\ carbon\ (\%)}{100}$$

For 0.55 g cellulose, C_i=0.240 g, for 3.00 g neat PET, C_i=1.878 g, for 3.00 g PET with 1% additive, C_i=1.884 g, and for 3.00 g PET with 5% additive, C_i=1.883 g, were obtained.

From the above equations, the average biodegradation extent of each sample at 90 days was obtained and shown in table 4-2. Original biodegradation extent data are shown in appendix F. Cellulose showed a high biodegradation extent in each inoculum. However, PET with additive did not show a high biodegradation in each inoculum.

Table 4-2 Biodegradation extent at 90 days.

		Difference to blank (mL)	Biodegradation extent (%)
Landfill leachate	Cellulose	170.6	32.4
	Neat PET	-1.7	0.0
	PET with 1% additive	7.1	0.2
	PET with 5% additive	52.4	1.3
Wastewater treatment residue	Cellulose	271.7	51.6
	Neat PET	-15.3	-0.4
	PET with 1% additive	12.3	0.3
	PET with 5% additive	-6.3	-0.2
Anaerobic digester	Cellulose	192.2	36.5
	Neat PET	46.5	1.1
	PET with 1% additive	25.0	0.6
	PET with 5% additive	0.5	0.0

4.3 Statistical analysis

For the statistical analysis, t-tests (α=0.05) were conducted for the cumulative gas volume at 90 days. Tables 4-3, 4-4, and 4-5 show the matrix of the results of the t-test for each inoculum.

Cellulose samples in every inoculum showed the t-test value less than 0.05 with other samples. In other words, the mean of cellulose samples' data was significantly different from other samples' data. However, there is no value of less than 0.05 between blank, neat PET, PET with 1 % additive and PET with 5% additive in every inoculum. Therefore, there was no significant difference between these samples' data.

Table 4-3 T-test results of cumulative gas volume with landfill leachate at 90 days.

	Neat PET	PET with 1% additive	PET with 5% additive	Blank
Cellulose	0.03	0.02	0.02	0.02
Neat PET		0.88	0.30	0.98
PET with 1% additive			0.28	0.88
PET with 5% additive				0.10

Note: T-tests values represent individual Student's T-test of pairs.

Table 4-4 T-test results of cumulative gas volume with wastewater treatment residue at 90 days.

	Neat PET	PET with 1% additive	PET with 5% additive	Blank
Cellulose	0.00	0.00	0.00	0.00
Neat PET		0.39	0.83	0.54
PET with 1% additive			0.66	0.62
PET with 5% additive				0.87

Note: T-tests values represent individual Student's T-test of pairs.

Table 4-5 T-test results of cumulative gas volume with anaerobic digester at 90 days.

	Neat PET	PET with 1% additive	PET with 5% additive	Blank
Cellulose	0.01	0.01	0.03	0.01
Neat PET		0.40	0.44	0.24
PET with 1% additive			0.67	0.51
PET with 5% additive				0.99

Note: T-tests values represent individual Student's T-test of pairs.

CHAPTER 5

CONCLUSIONS AND FUTURE WORK

5.1 Conclusions

In this study, the effect of a biodegradation promoting additive on PET in anaerobic digestion was investigated. The additive was kindly provided by ENSO Plastics, and three different PET sheets, which are neat PET, PET with 1 wt % additive, and 5 wt % additive, were made using the cast film extruder in the School of Packaging lab. As the anaerobic microorganism seeds, three different inoculums were obtained from landfill leachate, wastewater treatment residue, and an anaerobic digester. As bioreactors, 125 mL glass bottles with closures were prepared, and test samples, fresh cow manure, and inoculums were placed into the bioreactors. The bioreactors were kept in a 35 ˚C incubator. The gas produced was constituted of CH_4 and CO_2, and was measured for 90 days. Cellulose samples, prepared as positive controls, showed higher gas production than the other samples, and the cellulose biodegradation extent reached 32.4 %, 51.6 % and 36.5 % in each inoculum, respectively. PET with additive samples did not show higher gas production than either blank or neat PET in landfill leachate, wastewater treatment residue and anaerobic digester inoculums. Statistical analysis of the gas production data showed that only cellulose was significantly different than the other samples in landfill leachate, wastewater treatment and anaerobic digester inoculums (α=0.05). In conclusion, no significant difference was observed in PET with biodegradation promoting additive in this test environment. According to ENSO Plastics, the cloudiness of the PET sheets made for this experiment indicated insufficient dispersion of the additive: the company states that excellent dispersion is required to enhance biodegradation.

After the measurement at 90 days, the bioreactors were opened and test PET samples were collected and visually inspected with eyes and a microscope. Compared to the original samples, test PET samples still kept original shapes and no visual differences were observed.

Due to the short experimental period, each bioreactor was still producing gas at 90 days, although the gas evolution rates were much smaller than at the peak time. Therefore, there is a possibility that the result would change in an experiment with longer time. The gas production of the cellulose samples was not steady compared to the blank samples, and was less than the blank at some points. The reason may be because the initial pH drop of the cellulose samples was much higher than for the blank samples, and low pH could inhibit the microorganism growth, or even reduce the microorganism population. Thus, lower microorganism activity resulted in lower gas production.

One of the objectives of this research was to investigate the effect of different microorganism populations on biodegradation. Although gas production in the wastewater treatment residue and anaerobic digester inoculums was generally higher than in the landfill leachate inoculum, the differences for the PET samples were not statistically significant as for these samples the differences were relatively small.

5.2 Recommendations

This study, because of its limited nature, can provide only preliminary results and suggestions for additional research. First, this study was conducted in one condition, which was mesophilic temperature (35 ˚C) and relatively low solid content (5 wt % manure). Investigating different conditions, for instance thermophilic conditions (55 ˚C) or high solid content (over 30 %) may have different results. Second, in this study, the effect of biodegradation was judged by only gas production, but further analytical approaches, for instance, differential scanning

calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA), and scanning electron microscopy (SEM), may provide other insights to this study. Third, since low dispersion of the additive in the PET sheet may reduce the effect of the additive, running another experiment with PET sheets produced using a different type of extruder, which can disperse the additive more efficiently, for instance, a twin screw extruder, or using a commercial size extruder, may produce different results.

APPENDICES

APPENDIX A: CHN composition of samples

Table A-1 CHN data of samples.

Wt%	C	H	N
Cellulose	43.68	6.42	0.04
Neat PET	62.59	4.23	0.01
PET with 1% additive	62.80	4.24	0.02
PET with 5% additive	62.78	4.29	0.01
Manure	43.43	5.74	2.36
Landfill leachate	8.27	0.69	0.28
Wastewater treatment residue	36.49	5.33	5.18
Anaerobic digester	41.87	5.45	3.64

Note: Data shows the average of three measurements. All samples were dried by oven before measurements to prevent error caused by moisture.

APPENDIX B: Solid content and organic content of manure and inoculums

Table B-1 Solid content and organic content of manure.

Sample	1	2	3	Average
Cup (g)	12.3427	12.9071	12.9694	
Cup + wet manure (g)	21.0598	22.1419	20.3208	
Wet manure (g)	8.7171	9.2348	7.3514	
Cup + dried manure (g)	13.8810	14.4653	14.2466	
Dried manure (g)	1.5383	1.5582	1.2772	
Total solid content (%)	17.6	16.9	17.4	17.3
Cup + ash (g)	12.5397	13.1136	13.1375	
Ash (g)	0.1970	0.2065	0.1681	
Volatile solid content (%)	87.2	86.7	86.8	86.9

Note: To make 1L of 5 wt % solid manure, 289.3 g of manure and 710.7 g of water was mixed. In total, 4L of manure was made for the experiment.

Table B-2 Solid contents of inoculums.

	Solid content
Landfill leachate	0.47%
Wastewater treatment residue	1.40%
Anaerobic digester	2.70%

Note: Data was measured by moisture analyzer (AnD MX-50, A&D company ltd, Japan).

APPENDIX C: Temperature control capability of the oven

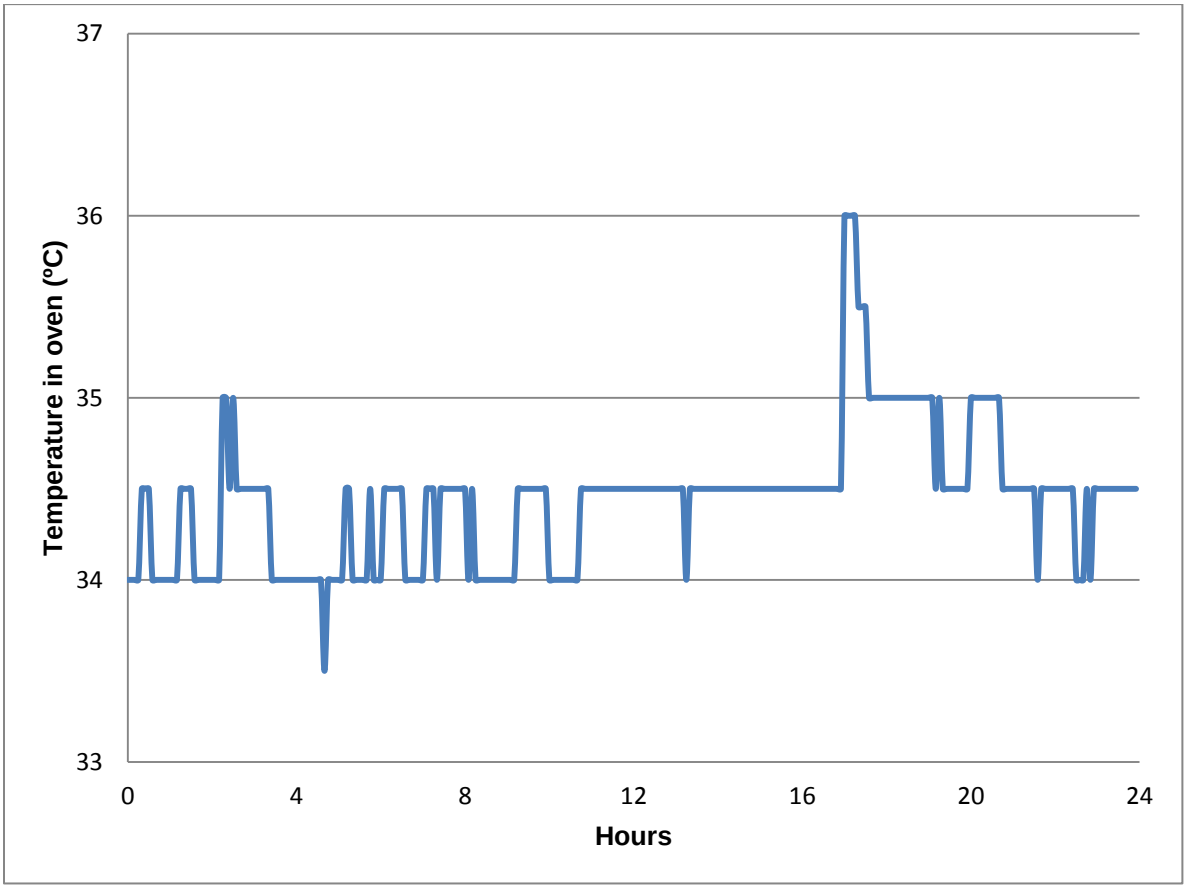

Figure C -1 Temperature record in oven.

Note: Sampling time of a temperature recorder was set up at every 5 minutes.

APPENDIX D: pH adjustment of the bioreactors

Table D-1 Amount of added NaOH (10 wt %).

Amount of added NaOH (mL)		Days		
Test samples	Inoculum	8	10	20
Blank	Landfill leachate	1.0		0.5
	Wastewater treatment residue	1.0		
	Anaerobic digester	1.0		0.5
Cellulose	Landfill leachate	1.0	1.0	0.5
	Wastewater treatment residue	1.0	1.0	
	Anaerobic digester	1.0	1.0	0.5
Neat PET	Landfill leachate	1.0		0.5
	Wastewater treatment residue	1.0		
	Anaerobic digester	1.0		0.5
PET with 1 % additive	Landfill leachate	1.0		0.5
	Wastewater treatment residue	1.0		
	Anaerobic digester	1.0		0.5
PET with 5 % additive	Landfill leachate	1.0		0.5
	Wastewater treatment residue	1.0		
	Anaerobic digester	1.0		0.5

APPENDIX E: Original gas evolution data of each bioreactor

Table E-1 Blank (only manure) with landfill leachate.

Note: Sample No.2 was accidentally broken after 75 days measurement.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	24.0	24.5	25.0	24.0	24.5	25.0	24.5	25.0	24.0	0.4
5	18.5	23.5	17.5	42.5	48.0	42.5	44.3	48.0	42.5	2.6
8	15.5	21.0	21.0	58.0	69.0	63.5	63.5	69.0	58.0	4.5
10	9.0	8.5	17.0	67.0	77.5	80.5	75.0	80.5	67.0	5.8
12	11.0	9.0	15.0	78.0	86.5	95.5	86.7	95.5	78.0	7.1
15	17.0	14.5	15.0	95.0	101.0	110.5	102.2	110.5	95.0	6.4
20	40.0	99.0	30.0	135.0	200.0	140.5	158.5	200.0	135.0	29.4
29	168.5	150.0	212.0	303.5	350.0	352.5	335.3	352.5	303.5	22.5
33	72.0	67.5	82.0	375.5	417.5	434.5	409.2	434.5	375.5	24.8
36	78.0	29.5	48.5	453.5	447.0	483.0	461.2	483.0	447.0	15.7
39	80.5	19.0	32.5	534.0	466.0	515.5	505.2	534.0	466.0	28.7
43	100.5	42.0	28.0	634.5	508.0	543.5	562.0	634.5	508.0	53.3
47	88.5	113.5	20.5	723.0	621.5	564.0	636.2	723.0	564.0	65.7
50	47.5	74.0	10.0	770.5	695.5	574.0	680.0	770.5	574.0	81.0
55	60.5	97.0	15.5	831.0	792.5	589.5	737.7	831.0	589.5	105.9
59	55.5	68.5	28.0	886.5	861.0	617.5	788.3	886.5	617.5	121.2
64	32.0	32.0	64.0	918.5	893.0	681.5	831.0	918.5	681.5	106.2
68	22.0	28.5	86.0	940.5	921.5	767.5	876.5	940.5	767.5	77.5
75	36.0	59.0	138.5	976.5	980.5	906.0	954.3	980.5	906.0	34.2
84	26.0	-	65.0	1002.5	-	971.0	986.8	1002.5	971.0	15.8
90	21.0	-	43.0	1023.5	-	1014.0	1018.8	1023.5	1014.0	4.8

Table E-2 Cellulose (0.55 g) with landfill leachate.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	24.0	23.5	25.5	24.0	23.5	25.5	24.3	25.5	23.5	0.8
5	31.0	30.5	29.5	55.0	54.0	55.0	54.7	55.0	54.0	0.5
8	23.5	24.5	22.0	78.5	78.5	77.0	78.0	78.5	77.0	0.7
10	20.0	28.0	21.0	98.5	106.5	98.0	101.0	106.5	98.0	3.9
12	64.0	0.0	47.5	162.5	106.5	145.5	138.2	162.5	106.5	23.4
15	31.0	53.0	26.0	193.5	159.5	171.5	174.8	193.5	159.5	14.1
20	24.0	50.0	30.5	217.5	209.5	202.0	209.7	217.5	202.0	6.3
29	218.0	220.0	198.0	435.5	429.5	400.0	421.7	435.5	400.0	15.5
33	127.0	65.0	113.0	562.5	494.5	513.0	523.3	562.5	494.5	28.7
36	87.5	25.0	100.0	650.0	519.5	613.0	594.2	650.0	519.5	54.9
39	98.5	22.5	78.5	748.5	542.0	691.5	660.7	748.5	542.0	87.1
43	66.5	31.0	46.5	815.0	573.0	738.0	708.7	815.0	573.0	100.9
47	53.5	46.0	31.5	868.5	619.0	769.5	752.3	868.5	619.0	102.6
50	42.0	64.5	13.5	910.5	683.5	783.0	792.3	910.5	683.5	92.9
55	39.0	129.0	26.0	949.5	812.5	809.0	857.0	949.5	809.0	65.4
59	32.0	57.0	48.5	981.5	869.5	857.5	902.8	981.5	857.5	55.8
64	12.0	28.5	16.0	993.5	898.0	873.5	921.7	993.5	873.5	51.8
68	9.5	33.5	9.0	1003.0	931.5	882.5	939.0	1003.0	882.5	49.5
75	28.5	153.0	76.0	1031.5	1084.5	958.5	1024.8	1084.5	958.5	51.7
84	42.0	73.0	205.0	1073.5	1157.5	1163.5	1131.5	1163.5	1073.5	41.1
90	62.5	46.0	65.0	1136.0	1203.5	1228.5	1189.3	1228.5	1136.0	39.1

Table E-3 Neat PET (3.00 g) with landfill leachate.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	25.0	26.5	29.0	25.0	26.5	29.0	26.8	29.0	25.0	1.6
5	18.0	17.0	19.0	43.0	43.5	48.0	44.8	48.0	43.0	2.2
8	15.0	15.5	14.0	58.0	59.0	62.0	59.7	62.0	58.0	1.7
10	8.0	10.0	7.0	66.0	69.0	69.0	68.0	69.0	66.0	1.4
12	7.5	9.0	12.5	73.5	78.0	81.5	77.7	81.5	73.5	3.3
15	17.0	13.0	21.0	90.5	91.0	102.5	94.7	102.5	90.5	5.5
20	49.0	44.0	141.5	139.5	135.0	244.0	172.8	244.0	135.0	50.4
29	189.5	201.0	145.0	329.0	336.0	389.0	351.3	389.0	329.0	26.8
33	75.0	89.5	82.0	404.0	425.5	471.0	433.5	471.0	404.0	27.9
36	67.0	71.0	33.5	471.0	496.5	504.5	490.7	504.5	471.0	14.3
39	38.0	42.5	30.5	509.0	539.0	535.0	527.7	539.0	509.0	13.3
43	53.5	47.5	33.0	562.5	586.5	568.0	572.3	586.5	562.5	10.3
47	40.0	33.0	31.5	602.5	619.5	599.5	607.2	619.5	599.5	8.8
50	37.0	26.5	26.5	639.5	646.0	626.0	637.2	646.0	626.0	8.3
55	106.0	115.0	35.0	745.5	761.0	661.0	722.5	761.0	661.0	43.9
59	96.0	85.0	33.0	841.5	846.0	694.0	793.8	846.0	694.0	70.6
64	62.5	25.0	10.5	904.0	871.0	704.5	826.5	904.0	704.5	87.3
68	40.5	14.0	9.5	944.5	885.0	714.0	847.8	944.5	714.0	97.7
75	50.5	34.5	45.0	995.0	919.5	759.0	891.2	995.0	759.0	98.4
84	37.0	77.0	51.5	1032.0	996.5	810.5	946.3	1032.0	810.5	97.1
90	26.5	62.5	123.0	1058.5	1059.0	933.5	1017.0	1059.0	933.5	59.0

Table E-4 PET with 1 % additive (3.00 g) with landfill leachate.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	27.0	28.0	28.5	27.0	28.0	28.5	27.8	28.5	27.0	0.6
5	19.0	18.0	20.0	46.0	46.0	48.5	46.8	48.5	46.0	1.2
8	15.0	14.5	21.0	61.0	60.5	69.5	63.7	69.5	60.5	4.1
10	11.0	7.0	12.0	72.0	67.5	81.5	73.7	81.5	67.5	5.8
12	10.0	8.5	10.0	82.0	76.0	91.5	83.2	91.5	76.0	6.4
15	14.0	14.0	10.5	96.0	90.0	102.0	96.0	102.0	90.0	4.9
20	46.0	54.5	38.5	142.0	144.5	140.5	142.3	144.5	140.5	1.6
29	179.5	186.0	202.0	321.5	330.5	342.5	331.5	342.5	321.5	8.6
33	112.0	115.0	77.0	433.5	445.5	419.5	432.8	445.5	419.5	10.6
36	103.0	127.5	76.5	536.5	573.0	496.0	535.2	573.0	496.0	31.4
39	45.5	99.0	48.0	582.0	672.0	544.0	599.3	672.0	544.0	53.7
43	36.0	100.5	80.0	618.0	772.5	624.0	671.5	772.5	618.0	71.5
47	23.0	78.0	102.0	641.0	850.5	726.0	739.2	850.5	641.0	86.0
50	13.5	43.0	61.5	654.5	893.5	787.5	778.5	893.5	654.5	97.8
55	14.0	44.5	70.0	668.5	938.0	857.5	821.3	938.0	668.5	113.0
59	29.5	42.0	55.0	698.0	980.0	912.5	863.5	980.0	698.0	120.2
64	45.5	22.5	24.0	743.5	1002.5	936.5	894.2	1002.5	743.5	109.9
68	54.0	15.0	12.0	797.5	1017.5	948.5	921.2	1017.5	797.5	91.9
75	98.0	34.0	23.0	895.5	1051.5	971.5	972.8	1051.5	895.5	63.7
84	55.0	22.0	17.0	950.5	1073.5	988.5	1004.2	1073.5	950.5	51.4
90	34.0	16.0	15.0	984.5	1089.5	1003.5	1025.8	1089.5	984.5	45.7

Table E-5 PET with 5 % additive (3.00 g) with landfill leachate.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	28.5	30.0	28.5	28.5	30.0	28.5	29.0	30.0	28.5	0.7
5	17.0	20.0	18.0	45.5	50.0	46.5	47.3	50.0	45.5	1.9
8	17.5	16.5	15.0	63.0	66.5	61.5	63.7	66.5	61.5	2.1
10	8.5	9.0	12.0	71.5	75.5	73.5	73.5	75.5	71.5	1.6
12	8.0	7.0	11.5	79.5	82.5	85.0	82.3	85.0	79.5	2.2
15	16.0	16.0	15.0	95.5	98.5	100.0	98.0	100.0	95.5	1.9
20	50.0	54.0	47.0	145.5	152.5	147.0	148.3	152.5	145.5	3.0
29	186.0	179.0	210.0	331.5	331.5	357.0	340.0	357.0	331.5	12.0
33	122.0	111.5	91.5	453.5	443.0	448.5	448.3	453.5	443.0	4.3
36	119.0	77.5	43.0	572.5	520.5	491.5	528.2	572.5	491.5	33.5
39	93.0	83.0	32.5	665.5	603.5	524.0	597.7	665.5	524.0	57.9
43	87.5	100.0	96.5	753.0	703.5	620.5	692.3	753.0	620.5	54.7
47	66.0	81.0	119.5	819.0	784.5	740.0	781.2	819.0	740.0	32.3
50	43.5	52.5	78.0	862.5	837.0	818.0	839.2	862.5	818.0	18.2
55	48.0	52.5	71.0	910.5	889.5	889.0	896.3	910.5	889.0	10.0
59	51.0	51.5	50.0	961.5	941.0	939.0	947.2	961.5	939.0	10.2
64	27.5	27.0	21.0	989.0	968.0	960.0	972.3	989.0	960.0	12.2
68	18.0	18.0	12.0	1007.0	986.0	972.0	988.3	1007.0	972.0	14.4
75	35.0	35.0	21.0	1042.0	1021.0	993.0	1018.7	1042.0	993.0	20.1
84	39.0	25.5	31.0	1081.0	1046.5	1024.0	1050.5	1081.0	1024.0	23.4
90	23.0	16.0	23.0	1104.0	1062.5	1047.0	1071.2	1104.0	1047.0	24.1

Table E-6 Blank (only manure) with wastewater treatment residue.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	33.5	36.5	30.5	33.5	36.5	30.5	33.5	36.5	30.5	2.4
5	39.0	34.0	39.5	72.5	70.5	70.0	71.0	72.5	70.0	1.1
8	48.0	45.0	47.0	120.5	115.5	117.0	117.7	120.5	115.5	2.1
10	41.5	37.0	33.0	162.0	152.5	150.0	154.8	162.0	150.0	5.2
12	59.5	57.5	41.0	221.5	210.0	191.0	207.5	221.5	191.0	12.6
15	79.0	70.0	80.0	300.5	280.0	271.0	283.8	300.5	271.0	12.3
20	114.5	129.0	118.5	415.0	409.0	389.5	404.5	415.0	389.5	10.9
29	209.0	250.0	253.0	624.0	659.0	642.5	641.8	659.0	624.0	14.3
33	95.0	111.5	122.5	719.0	770.5	765.0	751.5	770.5	719.0	23.1
36	59.0	60.5	66.5	778.0	831.0	831.5	813.5	831.5	778.0	25.1
39	58.0	58.5	62.5	836.0	889.5	894.0	873.2	894.0	836.0	26.3
43	58.0	62.0	62.5	894.0	951.5	956.5	934.0	956.5	894.0	28.4
47	51.5	45.5	47.5	945.5	997.0	1004.0	982.2	1004.0	945.5	26.1
50	29.5	28.0	27.0	975.0	1025.0	1031.0	1010.3	1031.0	975.0	25.1
55	34.0	30.5	22.0	1009.0	1055.5	1053.0	1039.2	1055.5	1009.0	21.4
59	40.0	39.5	47.0	1049.0	1095.0	1100.0	1081.3	1100.0	1049.0	23.0
64	20.0	17.5	18.0	1069.0	1112.5	1118.0	1099.8	1118.0	1069.0	21.9
68	11.0	11.0	10.5	1080.0	1123.5	1128.5	1110.7	1128.5	1080.0	21.8
75	34.5	23.5	24.5	1114.5	1147.0	1153.0	1138.2	1153.0	1114.5	16.9
84	24.5	20.0	20.5	1139.0	1167.0	1173.5	1159.8	1173.5	1139.0	15.0
90	17.0	17.0	15.5	1156.0	1184.0	1189.0	1176.3	1189.0	1156.0	14.5

Table E-7 Cellulose (0.55 g) with wastewater treatment residue.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	39.5	40.0	40.5	39.5	40.0	40.5	40.0	40.5	39.5	0.4
5	60.0	54.5	55.0	99.5	94.5	95.5	96.5	99.5	94.5	2.2
8	34.0	34.0	33.5	133.5	128.5	129.0	130.3	133.5	128.5	2.2
10	45.0	43.0	39.5	178.5	171.5	168.5	172.8	178.5	168.5	4.2
12	24.0	24.0	27.5	202.5	195.5	196.0	198.0	202.5	195.5	3.2
15	44.0	46.0	45.0	246.5	241.5	241.0	243.0	246.5	241.0	2.5
20	188.0	179.5	186.5	434.5	421.0	427.5	427.7	434.5	421.0	5.5
29	120.0	222.5	167.5	554.5	643.5	595.0	597.7	643.5	554.5	36.4
33	90.0	77.0	125.0	644.5	720.5	720.0	695.0	720.5	644.5	35.7
36	106.0	46.0	50.5	750.5	766.5	770.5	762.5	770.5	750.5	8.6
39	51.5	40.5	38.0	802.0	807.0	808.5	805.8	808.5	802.0	2.8
43	41.0	56.5	138.0	843.0	863.5	946.5	884.3	946.5	843.0	44.7
47	191.5	46.5	42.5	1034.5	910.0	989.0	977.8	1034.5	910.0	51.4
50	92.5	28.5	23.0	1127.0	938.5	1012.0	1025.8	1127.0	938.5	77.6
55	92.5	62.0	24.0	1219.5	1000.5	1036.0	1085.3	1219.5	1000.5	96.0
59	78.5	116.0	37.5	1298.0	1116.5	1073.5	1162.7	1298.0	1073.5	97.3
64	35.0	70.5	25.5	1333.0	1187.0	1099.0	1206.3	1333.0	1099.0	96.5
68	24.0	85.0	12.5	1357.0	1272.0	1111.5	1246.8	1357.0	1111.5	101.8
75	45.0	125.5	54.0	1402.0	1397.5	1165.5	1321.7	1402.0	1165.5	110.4
84	33.0	48.0	164.5	1435.0	1445.5	1330.0	1403.5	1445.5	1330.0	52.1
90	24.5	28.0	81.0	1459.5	1473.5	1411.0	1448.0	1473.5	1411.0	26.8

Table E-8 Neat PET (3.00 g) with wastewater treatment residue.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	35.5	38.5	39.0	35.5	38.5	39.0	37.7	39.0	35.5	1.5
5	38.5	36.5	43.0	74.0	75.0	82.0	77.0	82.0	74.0	3.6
8	46.0	53.0	53.5	120.0	128.0	135.5	127.8	135.5	120.0	6.3
10	24.0	36.0	35.0	144.0	164.0	170.5	159.5	170.5	144.0	11.3
12	46.0	60.0	53.0	190.0	224.0	223.5	212.5	224.0	190.0	15.9
15	76.5	75.0	47.0	266.5	299.0	270.5	278.7	299.0	266.5	14.5
20	122.0	100.0	120.5	388.5	399.0	391.0	392.8	399.0	388.5	4.5
29	216.0	229.0	256.5	604.5	628.0	647.5	626.7	647.5	604.5	17.6
33	75.0	111.5	99.0	679.5	739.5	746.5	721.8	746.5	679.5	30.1
36	48.0	60.5	55.5	727.5	800.0	802.0	776.5	802.0	727.5	34.7
39	53.5	53.0	47.5	781.0	853.0	849.5	827.8	853.0	781.0	33.1
43	72.0	55.5	52.0	853.0	908.5	901.5	887.7	908.5	853.0	24.7
47	68.5	38.5	40.0	921.5	947.0	941.5	936.7	947.0	921.5	11.0
50	43.5	26.0	25.5	965.0	973.0	967.0	968.3	973.0	965.0	3.4
55	50.5	27.5	28.0	1015.5	1000.5	995.0	1003.7	1015.5	995.0	8.7
59	53.5	38.0	36.0	1069.0	1038.5	1031.0	1046.2	1069.0	1031.0	16.4
64	26.0	15.0	15.0	1095.0	1053.5	1046.0	1064.8	1095.0	1046.0	21.5
68	21.0	13.5	14.0	1116.0	1067.0	1060.0	1081.0	1116.0	1060.0	24.9
75	41.5	27.5	46.0	1157.5	1094.5	1106.0	1119.3	1157.5	1094.5	27.4
84	27.0	20.0	27.5	1184.5	1114.5	1133.5	1144.2	1184.5	1114.5	29.6
90	16.5	18.0	16.0	1201.0	1132.5	1149.5	1161.0	1201.0	1132.5	29.1

Table E-9 PET with 1 % additive (3.00 g) with wastewater treatment residue.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	35.0	44.0	35.5	35.0	44.0	35.5	38.2	44.0	35.0	4.1
5	41.0	41.0	39.5	76.0	85.0	75.0	78.7	85.0	75.0	4.5
8	54.5	54.0	51.5	130.5	139.0	126.5	132.0	139.0	126.5	5.2
10	44.5	51.5	42.5	175.0	190.5	169.0	178.2	190.5	169.0	9.1
12	64.5	65.0	68.0	239.5	255.5	237.0	244.0	255.5	237.0	8.2
15	75.0	67.0	61.0	314.5	322.5	298.0	311.7	322.5	298.0	10.2
20	114.0	154.0	156.0	428.5	476.5	454.0	453.0	476.5	428.5	19.6
29	223.5	240.0	248.0	652.0	716.5	702.0	690.2	716.5	652.0	27.6
33	92.5	80.5	88.0	744.5	797.0	790.0	777.2	797.0	744.5	23.3
36	63.0	52.0	53.5	807.5	849.0	843.5	833.3	849.0	807.5	18.4
39	56.5	48.0	43.5	864.0	897.0	887.0	882.7	897.0	864.0	13.8
43	62.5	57.0	49.5	926.5	954.0	936.5	939.0	954.0	926.5	11.4
47	47.5	45.5	41.0	974.0	999.5	977.5	983.7	999.5	974.0	11.3
50	30.5	28.5	27.0	1004.5	1028.0	1004.5	1012.3	1028.0	1004.5	11.1
55	32.5	13.5	30.0	1037.0	1041.5	1034.5	1037.7	1041.5	1034.5	2.9
59	39.0	49.0	37.0	1076.0	1090.5	1071.5	1079.3	1090.5	1071.5	8.1
64	20.0	19.0	18.0	1096.0	1109.5	1089.5	1098.3	1109.5	1089.5	8.3
68	12.0	9.0	9.5	1108.0	1118.5	1099.0	1108.5	1118.5	1099.0	8.0
75	23.5	45.5	28.0	1131.5	1164.0	1127.0	1140.8	1164.0	1127.0	16.5
84	20.0	38.0	24.0	1151.5	1202.0	1151.0	1168.2	1202.0	1151.0	23.9
90	14.5	27.0	20.0	1166.0	1229.0	1171.0	1188.7	1229.0	1166.0	28.6

Table E-10 PET with 5 % additive (3.00 g) with wastewater treatment residue.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	36.5	38.0	36.5	36.5	38.0	36.5	37.0	38.0	36.5	0.7
5	37.0	38.5	38.0	73.5	76.5	74.5	74.8	76.5	73.5	1.2
8	49.5	43.0	50.5	123.0	119.5	125.0	122.5	125.0	119.5	2.3
10	43.5	40.0	39.5	166.5	159.5	164.5	163.5	166.5	159.5	2.9
12	61.0	60.0	61.0	227.5	219.5	225.5	224.2	227.5	219.5	3.4
15	70.0	75.0	71.0	297.5	294.5	296.5	296.2	297.5	294.5	1.2
20	141.0	120.5	132.0	438.5	415.0	428.5	427.3	438.5	415.0	9.6
29	275.0	265.0	263.0	713.5	680.0	691.5	695.0	713.5	680.0	13.9
33	82.0	111.0	95.5	795.5	791.0	787.0	791.2	795.5	787.0	3.5
36	48.0	71.0	60.5	843.5	862.0	847.5	851.0	862.0	843.5	7.9
39	40.0	62.5	55.5	883.5	924.5	903.0	903.7	924.5	883.5	16.7
43	39.0	66.0	56.5	922.5	990.5	959.5	957.5	990.5	922.5	27.8
47	30.0	48.5	44.5	952.5	1039.0	1004.0	998.5	1039.0	952.5	35.5
50	18.5	28.5	22.0	971.0	1067.5	1026.0	1021.5	1067.5	971.0	39.5
55	22.0	31.5	29.5	993.0	1099.0	1055.5	1049.2	1099.0	993.0	43.5
59	31.0	39.0	36.5	1024.0	1138.0	1092.0	1084.7	1138.0	1024.0	46.8
64	12.0	18.0	17.5	1036.0	1156.0	1109.5	1100.5	1156.0	1036.0	49.4
68	6.0	10.0	11.5	1042.0	1166.0	1121.0	1109.7	1166.0	1042.0	51.3
75	22.5	22.0	21.0	1064.5	1188.0	1142.0	1131.5	1188.0	1064.5	51.0
84	23.5	18.5	21.5	1088.0	1206.5	1163.5	1152.7	1206.5	1088.0	49.0
90	18.0	14.0	20.0	1106.0	1220.5	1183.5	1170.0	1220.5	1106.0	47.7

Table E-11 Blank (only manure) with anaerobic digester.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	35.0	36.5	34.0	35.0	36.5	34.0	35.2	36.5	34.0	1.0
5	28.0	26.5	27.0	63.0	63.0	61.0	62.3	63.0	61.0	0.9
8	23.0	29.5	25.0	86.0	92.5	86.0	88.2	92.5	86.0	3.1
10	18.5	15.0	14.0	104.5	107.5	100.0	104.0	107.5	100.0	3.1
12	20.0	21.0	20.0	124.5	128.5	120.0	124.3	128.5	120.0	3.5
15	24.0	27.5	26.0	148.5	156.0	146.0	150.2	156.0	146.0	4.2
20	50.0	39.0	40.5	198.5	195.0	186.5	193.3	198.5	186.5	5.0
29	219.5	261.5	211.5	418.0	456.5	398.0	424.2	456.5	398.0	24.3
33	141.5	124.5	152.5	559.5	581.0	550.5	563.7	581.0	550.5	12.8
36	131.5	124.0	178.5	691.0	705.0	729.0	708.3	729.0	691.0	15.7
39	57.0	85.0	131.5	748.0	790.0	860.5	799.5	860.5	748.0	46.4
43	53.5	56.0	101.5	801.5	846.0	962.0	869.8	962.0	801.5	67.7
47	42.0	62.0	68.5	843.5	908.0	1030.5	927.3	1030.5	843.5	77.6
50	30.5	50.5	37.0	874.0	958.5	1067.5	966.7	1067.5	874.0	79.2
55	52.0	58.5	41.5	926.0	1017.0	1109.0	1017.3	1109.0	926.0	74.7
59	59.0	51.0	42.0	985.0	1068.0	1151.0	1068.0	1151.0	985.0	67.8
64	37.0	29.0	23.0	1022.0	1097.0	1174.0	1097.7	1174.0	1022.0	62.1
68	30.0	22.5	16.0	1052.0	1119.5	1190.0	1120.5	1190.0	1052.0	56.3
75	50.0	40.5	27.0	1102.0	1160.0	1217.0	1159.7	1217.0	1102.0	46.9
84	44.5	33.5	34.5	1146.5	1193.5	1251.5	1197.2	1251.5	1146.5	42.9
90	32.0	26.5	30.0	1178.5	1220.0	1281.5	1226.7	1281.5	1178.5	42.3

Table E-12 Cellulose (0.55 g) with anaerobic digester.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	37.0	36.0	40.5	37.0	36.0	40.5	37.8	40.5	36.0	1.9
5	38.5	43.0	41.0	75.5	79.0	81.5	78.7	81.5	75.5	2.5
8	44.0	39.5	45.0	119.5	118.5	126.5	121.5	126.5	118.5	3.6
10	39.0	62.5	34.0	158.5	181.0	160.5	166.7	181.0	158.5	10.2
12	13.0	13.0	38.0	171.5	194.0	198.5	188.0	198.5	171.5	11.8
15	48.0	18.5	39.5	219.5	212.5	238.0	223.3	238.0	212.5	10.8
20	35.0	19.0	23.0	254.5	231.5	261.0	249.0	261.0	231.5	12.7
29	190.0	247.0	254.0	444.5	478.5	515.0	479.3	515.0	444.5	28.8
33	122.0	125.0	94.0	566.5	603.5	609.0	593.0	609.0	566.5	18.9
36	55.0	46.5	54.5	621.5	650.0	663.5	645.0	663.5	621.5	17.5
39	43.5	44.5	51.5	665.0	694.5	715.0	691.5	715.0	665.0	20.5
43	130.0	94.0	145.0	795.0	788.5	860.0	814.5	860.0	788.5	32.3
47	181.0	175.0	170.5	976.0	963.5	1030.5	990.0	1030.5	963.5	29.1
50	88.5	134.0	100.0	1064.5	1097.5	1130.5	1097.5	1130.5	1064.5	26.9
55	85.0	141.5	88.0	1149.5	1239.0	1218.5	1202.3	1239.0	1149.5	38.3
59	61.0	58.0	69.0	1210.5	1297.0	1287.5	1265.0	1297.0	1210.5	38.7
64	20.5	23.5	29.0	1231.0	1320.5	1316.5	1289.3	1320.5	1231.0	41.3
68	13.5	16.5	28.0	1244.5	1337.0	1344.5	1308.7	1344.5	1244.5	45.5
75	43.5	48.5	43.5	1288.0	1385.5	1388.0	1353.8	1388.0	1288.0	46.6
84	44.0	36.0	38.5	1332.0	1421.5	1426.5	1393.3	1426.5	1332.0	43.4
90	32.0	20.0	24.5	1364.0	1441.5	1451.0	1418.8	1451.0	1364.0	39.0

Table E-13 Neat PET (3.00 g) with anaerobic digester.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	37.0	37.5	35.5	37.0	37.5	35.5	36.7	37.5	35.5	0.8
5	30.0	33.0	31.5	67.0	70.5	67.0	68.2	70.5	67.0	1.6
8	25.0	23.0	25.0	92.0	93.5	92.0	92.5	93.5	92.0	0.7
10	16.0	15.0	13.0	108.0	108.5	105.0	107.2	108.5	105.0	1.5
12	18.5	21.5	17.5	126.5	130.0	122.5	126.3	130.0	122.5	3.1
15	22.0	25.5	25.0	148.5	155.5	147.5	150.5	155.5	147.5	3.6
20	38.5	45.0	45.5	187.0	200.5	193.0	193.5	200.5	187.0	5.5
29	220.5	216.0	219.0	407.5	416.5	412.0	412.0	416.5	407.5	3.7
33	188.0	166.0	188.0	595.5	582.5	600.0	592.7	600.0	582.5	7.4
36	173.0	167.0	160.5	768.5	749.5	760.5	759.5	768.5	749.5	7.8
39	103.0	131.0	90.5	871.5	880.5	851.0	867.7	880.5	851.0	12.3
43	69.5	83.5	84.0	941.0	964.0	935.0	946.7	964.0	935.0	12.5
47	54.5	61.0	66.5	995.5	1025.0	1001.5	1007.3	1025.0	995.5	12.7
50	30.5	36.5	38.5	1026.0	1061.5	1040.0	1042.5	1061.5	1026.0	14.6
55	32.5	38.0	41.0	1058.5	1099.5	1081.0	1079.7	1099.5	1058.5	16.8
59	38.5	42.0	49.0	1097.0	1141.5	1130.0	1122.8	1141.5	1097.0	18.9
64	22.5	24.5	26.0	1119.5	1166.0	1156.0	1147.2	1166.0	1119.5	20.0
68	21.0	17.0	16.5	1140.5	1183.0	1172.5	1165.3	1183.0	1140.5	18.1
75	70.0	26.0	34.0	1210.5	1209.0	1206.5	1208.7	1210.5	1206.5	1.6
84	58.0	23.5	31.5	1268.5	1232.5	1238.0	1246.3	1268.5	1232.5	15.8
90	36.0	22.5	22.0	1304.5	1255.0	1260.0	1273.2	1304.5	1255.0	22.2

Table E-14 PET with 1 % additive (3.00 g) with anaerobic digester.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	43.0	46.5	40.5	43.0	46.5	40.5	43.3	46.5	40.5	2.5
5	28.5	30.0	30.0	71.5	76.5	70.5	72.8	76.5	70.5	2.6
8	24.5	28.0	26.5	96.0	104.5	97.0	99.2	104.5	96.0	3.8
10	17.0	18.0	17.5	113.0	122.5	114.5	116.7	122.5	113.0	4.2
12	20.0	26.5	20.0	133.0	149.0	134.5	138.8	149.0	133.0	7.2
15	26.0	37.0	26.0	159.0	186.0	160.5	168.5	186.0	159.0	12.4
20	36.0	52.5	44.0	195.0	238.5	204.5	212.7	238.5	195.0	18.7
29	212.5	196.5	188.0	407.5	435.0	392.5	411.7	435.0	392.5	17.6
33	126.0	166.0	170.0	533.5	601.0	562.5	565.7	601.0	533.5	27.6
36	154.0	141.5	175.0	687.5	742.5	737.5	722.5	742.5	687.5	24.8
39	159.0	57.0	93.0	846.5	799.5	830.5	825.5	846.5	799.5	19.5
43	95.0	58.0	97.5	941.5	857.5	928.0	909.0	941.5	857.5	36.8
47	70.0	47.5	71.0	1011.5	905.0	999.0	971.8	1011.5	905.0	47.5
50	41.5	33.5	39.5	1053.0	938.5	1038.5	1010.0	1053.0	938.5	50.9
55	39.0	43.0	42.0	1092.0	981.5	1080.5	1051.3	1092.0	981.5	49.6
59	39.5	49.5	45.0	1131.5	1031.0	1125.5	1096.0	1131.5	1031.0	46.0
64	23.0	28.5	24.0	1154.5	1059.5	1149.5	1121.2	1154.5	1059.5	43.7
68	15.0	23.0	11.5	1169.5	1082.5	1161.0	1137.7	1169.5	1082.5	39.2
75	30.0	31.5	57.0	1199.5	1114.0	1218.0	1177.2	1218.0	1114.0	45.3
84	31.0	65.0	29.5	1230.5	1179.0	1247.5	1219.0	1247.5	1179.0	29.1
90	32.5	40.0	25.5	1263.0	1219.0	1273.0	1251.7	1273.0	1219.0	23.5

Table E-15 PET with 5 % additive (3.00 g) with anaerobic digester.

	Measured gas volume (mL)			Cumulative gas volume (mL)						
Day	1	2	3	1	2	3	Average	Max	Min	Standard deviation
3	35.5	39.5	46.5	35.5	39.5	46.5	40.5	46.5	35.5	4.5
5	30.0	29.5	32.0	65.5	69.0	78.5	71.0	78.5	65.5	5.5
8	21.0	24.0	21.0	86.5	93.0	99.5	93.0	99.5	86.5	5.3
10	12.0	12.0	11.0	98.5	105.0	110.5	104.7	110.5	98.5	4.9
12	21.0	19.0	19.0	119.5	124.0	129.5	124.3	129.5	119.5	4.1
15	27.0	23.0	26.0	146.5	147.0	155.5	149.7	155.5	146.5	4.1
20	44.0	49.5	51.5	190.5	196.5	207.0	198.0	207.0	190.5	6.8
29	225.5	232.5	235.5	416.0	429.0	442.5	429.2	442.5	416.0	10.8
33	134.5	129.0	173.5	550.5	558.0	616.0	574.8	616.0	550.5	29.3
36	141.5	82.5	158.0	692.0	640.5	774.0	702.2	774.0	640.5	55.0
39	134.0	73.0	77.0	826.0	713.5	851.0	796.8	851.0	713.5	59.8
43	101.0	99.5	85.5	927.0	813.0	936.5	892.2	936.5	813.0	56.1
47	79.5	67.0	61.5	1006.5	880.0	998.0	961.5	1006.5	880.0	57.7
50	47.0	37.5	39.0	1053.5	917.5	1037.0	1002.7	1053.5	917.5	60.6
55	50.0	32.0	45.5	1103.5	949.5	1082.5	1045.2	1103.5	949.5	68.2
59	49.5	41.0	50.5	1153.0	990.5	1133.0	1092.2	1153.0	990.5	72.4
64	26.0	25.5	23.0	1179.0	1016.0	1156.0	1117.0	1179.0	1016.0	72.0
68	18.0	20.5	13.5	1197.0	1036.5	1169.5	1134.3	1197.0	1036.5	70.1
75	35.0	34.0	27.5	1232.0	1070.5	1197.0	1166.5	1232.0	1070.5	69.4
84	35.0	36.0	35.0	1267.0	1106.5	1232.0	1201.8	1267.0	1106.5	68.9
90	29.0	20.0	27.0	1296.0	1126.5	1259.0	1227.2	1296.0	1126.5	72.8

APPENDIX F: Original biodegradation extent data of each bioreactor

Table F-1 Cellulose (0.55 g) with landfill leachate.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	-0.1	-0.2	0.2	0.0	0.2	-0.2	0.2
5	2.0	1.8	2.0	2.0	2.0	1.8	0.1
8	2.8	2.8	2.6	2.8	2.8	2.6	0.1
10	4.5	6.0	4.4	4.9	6.0	4.4	0.7
12	14.4	3.8	11.2	9.8	14.4	3.8	4.4
15	17.3	10.9	13.2	13.8	17.3	10.9	2.7
20	11.2	9.7	8.3	9.7	11.2	8.3	1.2
29	19.0	17.9	12.3	16.4	19.0	12.3	2.9
33	29.1	16.2	19.7	21.7	29.1	16.2	5.4
36	35.8	11.1	28.8	25.2	35.8	11.1	10.4
39	46.2	7.0	35.4	29.5	46.2	7.0	16.5
43	48.0	2.1	33.4	27.8	48.0	2.1	19.2
47	44.1	-3.3	25.3	22.0	44.1	-3.3	19.5
50	43.7	0.7	19.5	21.3	43.7	0.7	17.6
55	40.2	14.2	13.5	22.6	40.2	13.5	12.4
59	36.7	15.4	13.1	21.7	36.7	13.1	10.6
64	30.8	12.7	8.1	17.2	30.8	8.1	9.8
68	24.0	10.4	1.1	11.9	24.0	1.1	9.4
75	14.6	24.7	0.8	13.4	24.7	0.8	9.8
84	16.5	32.4	33.5	27.5	33.5	16.5	7.8
90	22.2	35.1	39.8	32.4	39.8	22.2	7.4

Table F-2 Neat PET (3.00 g) with landfill leachate.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.0	0.0	0.1	0.1	0.1	0.0	0.0
5	0.0	0.0	0.1	0.0	0.1	0.0	0.1
8	-0.1	-0.1	0.0	-0.1	0.0	-0.1	0.0
10	-0.2	-0.1	-0.1	-0.2	-0.1	-0.2	0.0
12	-0.3	-0.2	-0.1	-0.2	-0.1	-0.3	0.1
15	-0.3	-0.3	0.0	-0.2	0.0	-0.3	0.1
20	-0.5	-0.6	2.1	0.3	2.1	-0.6	1.2
29	-0.2	0.0	1.3	0.4	1.3	-0.2	0.7
33	-0.1	0.4	1.5	0.6	1.5	-0.1	0.7
36	0.2	0.9	1.1	0.7	1.1	0.2	0.3
39	0.1	0.8	0.7	0.5	0.8	0.1	0.3
43	0.0	0.6	0.1	0.3	0.6	0.0	0.2
47	-0.8	-0.4	-0.9	-0.7	-0.4	-0.9	0.2
50	-1.0	-0.8	-1.3	-1.0	-0.8	-1.3	0.2
55	0.2	0.6	-1.9	-0.4	0.6	-1.9	1.1
59	1.3	1.4	-2.3	0.1	1.4	-2.3	1.7
64	1.8	1.0	-3.1	-0.1	1.8	-3.1	2.1
68	1.7	0.2	-3.9	-0.7	1.7	-3.9	2.4
75	1.0	-0.8	-4.7	-1.5	1.0	-4.7	2.4
84	1.1	0.2	-4.3	-1.0	1.1	-4.3	2.4
90	1.0	1.0	-2.1	0.0	1.0	-2.1	1.4

Table F-3 PET with 1 % additive (3.00 g) with landfill leachate.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.1	0.1	0.1	0.1	0.1	0.1	0.0
5	0.0	0.0	0.1	0.1	0.1	0.0	0.0
8	-0.1	-0.1	0.1	0.0	0.1	-0.1	0.1
10	-0.1	-0.2	0.2	0.0	0.2	-0.2	0.1
12	-0.1	-0.3	0.1	-0.1	0.1	-0.3	0.2
15	-0.1	-0.3	0.0	-0.1	0.0	-0.3	0.1
20	-0.4	-0.3	-0.4	-0.4	-0.3	-0.4	0.0
29	-0.3	-0.1	0.2	-0.1	0.2	-0.3	0.2
33	0.6	0.9	0.3	0.6	0.9	0.3	0.3
36	1.8	2.7	0.8	1.8	2.7	0.8	0.8
39	1.9	4.0	0.9	2.3	4.0	0.9	1.3
43	1.4	5.1	1.5	2.6	5.1	1.4	1.7
47	0.1	5.2	2.2	2.5	5.2	0.1	2.1
50	-0.6	5.2	2.6	2.4	5.2	-0.6	2.4
55	-1.7	4.8	2.9	2.0	4.8	-1.7	2.7
59	-2.2	4.6	3.0	1.8	4.6	-2.2	2.9
64	-2.1	4.1	2.6	1.5	4.1	-2.1	2.7
68	-1.9	3.4	1.7	1.1	3.4	-1.9	2.2
75	-1.4	2.4	0.4	0.4	2.4	-1.4	1.5
84	-0.9	2.1	0.0	0.4	2.1	-0.9	1.2
90	-0.8	1.7	-0.4	0.2	1.7	-0.8	1.1

Table F-4 PET with 5 % additive (3.00 g) with landfill leachate.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.1	0.1	0.1	0.1	0.1	0.1	0.0
5	0.0	0.1	0.1	0.1	0.1	0.0	0.0
8	0.0	0.1	0.0	0.0	0.1	0.0	0.1
10	-0.1	0.0	0.0	0.0	0.0	-0.1	0.0
12	-0.2	-0.1	0.0	-0.1	0.0	-0.2	0.1
15	-0.2	-0.1	-0.1	-0.1	-0.1	-0.2	0.0
20	-0.3	-0.1	-0.3	-0.2	-0.1	-0.3	0.1
29	-0.1	-0.1	0.5	0.1	0.5	-0.1	0.3
33	1.1	0.8	1.0	0.9	1.1	0.8	0.1
36	2.7	1.4	0.7	1.6	2.7	0.7	0.8
39	3.9	2.4	0.5	2.2	3.9	0.5	1.4
43	4.6	3.4	1.4	3.2	4.6	1.4	1.3
47	4.4	3.6	2.5	3.5	4.4	2.5	0.8
50	4.4	3.8	3.3	3.9	4.4	3.3	0.4
55	4.2	3.7	3.7	3.8	4.2	3.7	0.2
59	4.2	3.7	3.6	3.8	4.2	3.6	0.2
64	3.8	3.3	3.1	3.4	3.8	3.1	0.3
68	3.2	2.7	2.3	2.7	3.2	2.3	0.3
75	2.1	1.6	0.9	1.6	2.1	0.9	0.5
84	2.3	1.4	0.9	1.5	2.3	0.9	0.6
90	2.1	1.1	0.7	1.3	2.1	0.7	0.6

Table F-5 Cellulose (0.55 g) with wastewater treatment residue.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	1.1	1.2	1.3	1.2	1.3	1.1	0.1
5	5.4	4.5	4.6	4.8	5.4	4.5	0.4
8	3.0	2.1	2.2	2.4	3.0	2.1	0.4
10	4.5	3.2	2.6	3.4	4.5	2.6	0.8
12	-0.9	-2.3	-2.2	-1.8	-0.9	-2.3	0.6
15	-7.1	-8.0	-8.1	-7.7	-7.1	-8.1	0.5
20	5.7	3.1	4.4	4.4	5.7	3.1	1.0
29	-16.6	0.3	-8.9	-8.4	0.3	-16.6	6.9
33	-20.3	-5.9	-6.0	-10.7	-5.9	-20.3	6.8
36	-12.0	-8.9	-8.2	-9.7	-8.2	-12.0	1.6
39	-13.5	-12.6	-12.3	-12.8	-12.3	-13.5	0.5
43	-17.3	-13.4	2.4	-9.4	2.4	-17.3	8.5
47	9.9	-13.7	1.3	-0.8	9.9	-13.7	9.8
50	22.1	-13.6	0.3	2.9	22.1	-13.6	14.7
55	34.2	-7.3	-0.6	8.8	34.2	-7.3	18.2
59	41.1	6.7	-1.5	15.4	41.1	-1.5	18.5
64	44.2	16.5	-0.2	20.2	44.2	-0.2	18.3
68	46.7	30.6	0.2	25.8	46.7	0.2	19.3
75	50.1	49.2	5.2	34.8	50.1	5.2	21.0
84	52.2	54.2	32.3	46.2	54.2	32.3	9.9
90	53.7	56.4	44.5	51.6	56.4	44.5	5.1

Table F-6 Neat PET (3.00 g) with wastewater treatment residue.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.0	0.1	0.1	0.1	0.1	0.0	0.0
5	0.1	0.1	0.3	0.1	0.3	0.1	0.1
8	0.1	0.3	0.4	0.2	0.4	0.1	0.2
10	-0.3	0.2	0.4	0.1	0.4	-0.3	0.3
12	-0.4	0.4	0.4	0.1	0.4	-0.4	0.4
15	-0.4	0.4	-0.3	-0.1	0.4	-0.4	0.4
20	-0.4	-0.1	-0.3	-0.3	-0.1	-0.4	0.1
29	-0.9	-0.3	0.1	-0.4	0.1	-0.9	0.4
33	-1.7	-0.3	-0.1	-0.7	-0.1	-1.7	0.7
36	-2.1	-0.3	-0.3	-0.9	-0.3	-2.1	0.8
39	-2.2	-0.5	-0.6	-1.1	-0.5	-2.2	0.8
43	-2.0	-0.6	-0.8	-1.1	-0.6	-2.0	0.6
47	-1.5	-0.9	-1.0	-1.1	-0.9	-1.5	0.3
50	-1.1	-0.9	-1.1	-1.0	-0.9	-1.1	0.1
55	-0.6	-0.9	-1.1	-0.9	-0.6	-1.1	0.2
59	-0.3	-1.0	-1.2	-0.9	-0.3	-1.2	0.4
64	-0.1	-1.1	-1.3	-0.8	-0.1	-1.3	0.5
68	0.1	-1.1	-1.2	-0.7	0.1	-1.2	0.6
75	0.5	-1.1	-0.8	-0.5	0.5	-1.1	0.7
84	0.6	-1.1	-0.6	-0.4	0.6	-1.1	0.7
90	0.6	-1.1	-0.7	-0.4	0.6	-1.1	0.7

Table F-7 PET with 1 % additive (3.00 g) with wastewater treatment residue.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.0	0.3	0.0	0.1	0.3	0.0	0.1
5	0.1	0.3	0.1	0.2	0.3	0.1	0.1
8	0.3	0.5	0.2	0.3	0.5	0.2	0.1
10	0.5	0.9	0.3	0.6	0.9	0.3	0.2
12	0.8	1.2	0.7	0.9	1.2	0.7	0.2
15	0.7	0.9	0.3	0.7	0.9	0.3	0.2
20	0.6	1.7	1.2	1.2	1.7	0.6	0.5
29	0.2	1.8	1.5	1.2	1.8	0.2	0.7
33	-0.2	1.1	0.9	0.6	1.1	-0.2	0.6
36	-0.1	0.9	0.7	0.5	0.9	-0.1	0.4
39	-0.2	0.6	0.3	0.2	0.6	-0.2	0.3
43	-0.2	0.5	0.1	0.1	0.5	-0.2	0.3
47	-0.2	0.4	-0.1	0.0	0.4	-0.2	0.3
50	-0.1	0.4	-0.1	0.0	0.4	-0.1	0.3
55	-0.1	0.1	-0.1	0.0	0.1	-0.1	0.1
59	-0.1	0.2	-0.2	0.0	0.2	-0.2	0.2
64	-0.1	0.2	-0.3	0.0	0.2	-0.3	0.2
68	-0.1	0.2	-0.3	-0.1	0.2	-0.3	0.2
75	-0.2	0.6	-0.3	0.1	0.6	-0.3	0.4
84	-0.2	1.0	-0.2	0.2	1.0	-0.2	0.6
90	-0.3	1.3	-0.1	0.3	1.3	-0.3	0.7

Table F-8 PET with 5 % additive (3.00 g) with wastewater treatment residue.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.1	0.1	0.1	0.1	0.1	0.1	0.0
5	0.1	0.1	0.1	0.1	0.1	0.1	0.0
8	0.1	0.0	0.2	0.1	0.2	0.0	0.1
10	0.3	0.1	0.2	0.2	0.3	0.1	0.1
12	0.5	0.3	0.4	0.4	0.5	0.3	0.1
15	0.3	0.3	0.3	0.3	0.3	0.3	0.0
20	0.8	0.3	0.6	0.6	0.8	0.3	0.2
29	1.7	0.9	1.2	1.3	1.7	0.9	0.3
33	1.1	1.0	0.9	1.0	1.1	0.9	0.1
36	0.7	1.2	0.8	0.9	1.2	0.7	0.2
39	0.3	1.2	0.7	0.7	1.2	0.3	0.4
43	-0.3	1.4	0.6	0.6	1.4	-0.3	0.7
47	-0.7	1.4	0.5	0.4	1.4	-0.7	0.9
50	-1.0	1.4	0.4	0.3	1.4	-1.0	1.0
55	-1.1	1.4	0.4	0.2	1.4	-1.1	1.1
59	-1.4	1.4	0.3	0.1	1.4	-1.4	1.1
64	-1.5	1.4	0.2	0.0	1.4	-1.5	1.2
68	-1.7	1.3	0.3	0.0	1.3	-1.7	1.2
75	-1.8	1.2	0.1	-0.2	1.2	-1.8	1.2
84	-1.7	1.1	0.1	-0.2	1.1	-1.7	1.2
90	-1.7	1.1	0.2	-0.2	1.1	-1.7	1.2

Table F-9 Cellulose (0.55 g) with anaerobic digester.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.3	0.2	1.0	0.5	1.0	0.2	0.4
5	2.5	3.2	3.6	3.1	3.6	2.5	0.5
8	5.9	5.8	7.3	6.3	7.3	5.8	0.7
10	10.3	14.6	10.7	11.9	14.6	10.3	1.9
12	9.0	13.2	14.1	12.1	14.1	9.0	2.2
15	13.2	11.8	16.7	13.9	16.7	11.8	2.0
20	11.6	7.2	12.8	10.6	12.8	7.2	2.4
29	3.9	10.3	17.2	10.5	17.2	3.9	5.5
33	0.5	7.6	8.6	5.6	8.6	0.5	3.6
36	-16.5	-11.1	-8.5	-12.0	-8.5	-16.5	3.3
39	-25.5	-19.9	-16.0	-20.5	-16.0	-25.5	3.9
43	-14.2	-15.4	-1.9	-10.5	-1.9	-15.4	6.1
47	9.2	6.9	19.6	11.9	19.6	6.9	5.5
50	18.6	24.8	31.1	24.8	31.1	18.6	5.1
55	25.1	42.1	38.2	35.1	42.1	25.1	7.3
59	27.0	43.5	41.7	37.4	43.5	27.0	7.3
64	25.3	42.3	41.5	36.4	42.3	25.3	7.8
68	23.5	41.1	42.5	35.7	42.5	23.5	8.6
75	24.4	42.9	43.3	36.8	43.3	24.4	8.8
84	25.6	42.6	43.5	37.2	43.5	25.6	8.2
90	26.1	40.8	42.6	36.5	42.6	26.1	7.4

Table F-10 Neat PET (3.00 g) with anaerobic digester.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.0	0.1	0.0	0.0	0.1	0.0	0.0
5	0.1	0.2	0.1	0.1	0.2	0.1	0.0
8	0.1	0.1	0.1	0.1	0.1	0.1	0.0
10	0.1	0.1	0.0	0.1	0.1	0.0	0.0
12	0.1	0.1	0.0	0.0	0.1	0.0	0.1
15	0.0	0.1	-0.1	0.0	0.1	-0.1	0.1
20	-0.2	0.2	0.0	0.0	0.2	-0.2	0.1
29	-0.4	-0.2	-0.3	-0.3	-0.2	-0.4	0.1
33	0.8	0.5	0.9	0.7	0.9	0.5	0.2
36	1.5	1.0	1.3	1.2	1.5	1.0	0.2
39	1.7	2.0	1.3	1.7	2.0	1.3	0.3
43	1.7	2.3	1.6	1.9	2.3	1.6	0.3
47	1.7	2.4	1.8	1.9	2.4	1.7	0.3
50	1.4	2.3	1.8	1.8	2.3	1.4	0.4
55	1.0	2.0	1.5	1.5	2.0	1.0	0.4
59	0.7	1.8	1.5	1.3	1.8	0.7	0.5
64	0.5	1.7	1.4	1.2	1.7	0.5	0.5
68	0.5	1.5	1.3	1.1	1.5	0.5	0.4
75	1.2	1.2	1.1	1.2	1.2	1.1	0.0
84	1.7	0.9	1.0	1.2	1.7	0.9	0.4
90	1.9	0.7	0.8	1.1	1.9	0.7	0.5

Table F-11 PET with 1 % additive (3.00 g) with anaerobic digester.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.2	0.3	0.1	0.2	0.3	0.1	0.1
5	0.2	0.3	0.2	0.3	0.3	0.2	0.1
8	0.2	0.4	0.2	0.3	0.4	0.2	0.1
10	0.2	0.4	0.3	0.3	0.4	0.2	0.1
12	0.2	0.6	0.2	0.4	0.6	0.2	0.2
15	0.2	0.9	0.3	0.4	0.9	0.2	0.3
20	0.0	1.1	0.3	0.5	1.1	0.0	0.5
29	-0.4	0.3	-0.8	-0.3	0.3	-0.8	0.4
33	-0.7	0.9	0.0	0.0	0.9	-0.7	0.7
36	-0.5	0.8	0.7	0.3	0.8	-0.5	0.6
39	1.1	0.0	0.8	0.6	1.1	0.0	0.5
43	1.7	-0.3	1.4	0.9	1.7	-0.3	0.9
47	2.0	-0.5	1.7	1.1	2.0	-0.5	1.2
50	2.1	-0.7	1.7	1.0	2.1	-0.7	1.2
55	1.8	-0.9	1.5	0.8	1.8	-0.9	1.2
59	1.5	-0.9	1.4	0.7	1.5	-0.9	1.1
64	1.4	-0.9	1.3	0.6	1.4	-0.9	1.1
68	1.2	-0.9	1.0	0.4	1.2	-0.9	0.9
75	1.0	-1.1	1.4	0.4	1.4	-1.1	1.1
84	0.8	-0.4	1.2	0.5	1.2	-0.4	0.7
90	0.9	-0.2	1.1	0.6	1.1	-0.2	0.6

Table F-12 PET with 5 % additive (3.00 g) with anaerobic digester.

	Biodegradation extent (%)						
Day	1	2	3	Average	Max	Min	Standard deviation
3	0.0	0.1	0.3	0.1	0.3	0.0	0.1
5	0.1	0.2	0.4	0.2	0.4	0.1	0.1
8	0.0	0.1	0.3	0.1	0.3	0.0	0.1
10	-0.1	0.0	0.2	0.0	0.2	-0.1	0.1
12	-0.1	0.0	0.1	0.0	0.1	-0.1	0.1
15	-0.1	-0.1	0.1	0.0	0.1	-0.1	0.1
20	-0.1	0.1	0.3	0.1	0.3	-0.1	0.2
29	-0.2	0.1	0.4	0.1	0.4	-0.2	0.3
33	-0.3	-0.1	1.3	0.3	1.3	-0.3	0.7
36	-0.4	-1.6	1.6	-0.1	1.6	-1.6	1.3
39	0.6	-2.1	1.2	-0.1	1.2	-2.1	1.4
43	1.4	-1.4	1.6	0.5	1.6	-1.4	1.4
47	1.9	-1.1	1.7	0.8	1.9	-1.1	1.4
50	2.1	-1.2	1.7	0.9	2.1	-1.2	1.5
55	2.1	-1.6	1.6	0.7	2.1	-1.6	1.7
59	2.1	-1.9	1.6	0.6	2.1	-1.9	1.8
64	2.0	-2.0	1.4	0.5	2.0	-2.0	1.7
68	1.9	-2.0	1.2	0.3	1.9	-2.0	1.7
75	1.8	-2.2	0.9	0.2	1.8	-2.2	1.7
84	1.7	-2.2	0.8	0.1	1.7	-2.2	1.7
90	1.7	-2.4	0.8	0.0	1.7	-2.4	1.8

BIBLIOGRAPHY

BIBLIOGRAPHY

[1] EPA, "Municipal Solid Waste Generation, Recycling, and Disposal in the United States Tables and Figures for 2012 U.S." Accessed Apr 22, 2015. http://www.epa.gov/solidwaste/nonhaz/municipal/msw99.htm

[2] Hoornweg, Daniel, and Perinaz Bhada-Tata, "What a waste: a global review of solid waste management." 2012. Accessed Apr 22, 2015. http://siteresources.worldbank.org/INTURBANDEVELOPMENT/Resources/336387-1334852610766/What_a_Waste2012_Final.pdf

[3] European Environment Agency (EEA), "Managing Municipal Solid Waste - a Review of Achievements in 32 European Countries." Accessed Apr 22, 2015. http://www.eea.europa.eu/publications/managing-municipal-solid-waste

[4] PlasticsEurope, "Plastics – the Facts 2012 An Analysis of European Plastics Production, Demand and Waste Data for 2011." Accessed Apr 22, 2015. http://www.plasticseurope.org/documents/document/20121120170458-final_plasticsthefacts_nov2012_en_web_resolution.pdf

[5] NAPCOR, "Postconsumer PET Container Recycling Activity in 2013." Accessed Apr 22, 2015. http://www.napcor.com/pdf/NAPCOR_2013RateReport-FINAL.pdf

[6] EPBP, "How to Keep a Sustainable PET Recycling Industry in Europe." Accessed Apr 22, 2015. http://www.epbp.org/

[7] The Council for PET Bottle Recycling, "Recycling Rate of PET Bottles." Accessed Apr 22, 2015. http://www.petbottle-rec.gr.jp/english/actual2.html

[8] Selke, Susan E. M., John D. Culter, and Ruben J. Hernandez, “Plastics Packaging: Properties, Processing, Applications, and Regulations.” 2004.

[9] Chemical & Engineering News, "Coca-Cola's Biobased Bottles." Accessed Apr 02, 2015. https://pubs.acs.org/cen/news/87/i21/8721notw9.html

[10] Chemistryworld, "Coca-Cola Collaborates on Bio-PET Project." Accessed Apr 02, 2015. http://www.rsc.org/chemistryworld/2012/06/coca-cola-collaborates-bio-pet-project

[11] Passport. “Global beverage packaging: refreshing packaging developments for sustained growth.” June 2014.

[12] ASTM D883 - 12, "Standard Terminology Relating to Plastics." *ASTM International*, 2012. www.astm.org.

[13] Leejarkpai, Thanawadee, Unchalee Suwanmanee, Yosita Rudeekit, and Thumrongrut Mungcharoen. "Biodegradable kinetics of plastics under controlled composting conditions." *Waste management* 31, no. 6 (2011): 1153-1161.

[14] Kijchavengkul, Thitisilp, and Rafael Auras. "Compostability of polymers."*Polymer international* 57, no. 6 (2008): 793-804.

[15] Themelis, Nickolas J., and Priscilla A. Ulloa. "Methane generation in landfills."*Renewable Energy* 32, no. 7 (2007): 1243-1257.

[16] Yagi, Hisaaki, Fumi Ninomiya, Masahiro Funabashi, and Masao Kunioka. "Mesophilic anaerobic biodegradation test and analysis of eubacteria and archaea involved in anaerobic biodegradation of four specified biodegradable polyesters." *Polymer Degradation and Stability* 110 (2014): 278-283.

[17] Yagi, Hisaaki, Fumi Ninomiya, Masahiro Funabashi, and Masao Kunioka. "Thermophilic anaerobic biodegradation test and analysis of eubacteria involved in anaerobic biodegradation of four specified biodegradable polyesters."*Polymer Degradation and Stability* 98, no. 6 (2013): 1182-1187.

[18] Yagi, Hisaaki, Fumi Ninomiya, Masahiro Funabashi, and Masao Kunioka. "Anaerobic biodegradation of poly (lactic acid) film in anaerobic sludge." *Journal of Polymers and the Environment* 20, no. 3 (2012): 673-680.

[19] Hubackova, Jitka, Marie Dvorackova, Petr Svoboda, Pavel Mokrejs, Jan Kupec, Iva Pozarova, Pavol Alexy, Peter Bugaj, Michal Machovsky, and Marek Koutny. "Influence of various starch types on PCL/starch blends anaerobic biodegradation." *Polymer Testing* 32, no. 6 (2013): 1011-1019.

[20] Hermanová, Soňa, Pavla Šmejkalová, Jan Merna, and Marie Zarevúcka. "Biodegradation of waste PET based copolyesters in thermophilic anaerobic sludge." *Polymer Degradation and Stability* 111 (2015): 176-184.

[21] Yam, Kit L., ed. "Landfills." *The Wiley encyclopedia of packaging technology*. John Wiley & Sons, 2009.

[22] Energy Information Administration, "Documentation for Emissions of Greenhouse Gases in the United States 2006." Accessed Apr 06, 2015. http://www.eia.gov/oiaf/1605/ggrpt/documentation/pdf/0638(2006).pdf

[23] Zimring, Carl A., and William L. Rathje, eds. "Landfills, Modern." *Encyclopedia of consumption and waste: the social science of garbage*. Vol. 1. Sage, 2012.

[24] Kumar, Sunil, Chart Chiemchaisri, and Ackmez Mudhoo. "Bioreactor landfill technology in municipal solid waste treatment: An overview." *Critical reviews in biotechnology* 31, no. 1 (2011): 77-97.

[25] Freudenrich, Ph.D. "How Landfills Work." *HowStuffWorks*. Accessed Apr 06, 2015. http://science.howstuffworks.com/environmental/green-science/landfill.htm

[26] Themelis, Nickolas J., and Young Hwan Kim. "Material and energy balances in a large-scale aerobic bioconversion cell." *Waste management & research* 20, no. 3 (2002): 234-242.

[27] Ebnesajjad, Sina, ed. "Biodegradable Polymers and Polymer Blends." *Handbook of biopolymers and biodegradable plastics: properties, processing and applications*. William Andrew, 2012.

[28] IHS Pressroom, "Consumer Pressure and Legislation Increasing Demand for Biodegradable Plastics by Nearly 15 Percent Annually During 2012 to 2017 in North America, Europe and Asia, Says IHS Study*."* Accessed Apr 06, 2015. http://press.ihs.com/press-release/bio-plastics/consumer-pressure-and-legislation-increasing-demand-biodegradable-plastic

[29] Ammala, Anne, Stuart Bateman, Katherine Dean, Eustathios Petinakis, Parveen Sangwan, Susan Wong, Qiang Yuan, Long Yu, Colin Patrick, and K. H. Leong. "An overview of degradable and biodegradable polyolefins." *Progress in Polymer Science* 36, no. 8 (2011): 1015-1049.

[30] EPI, "TDPA®: Totally Degradable Plastic Additive" Accessed Apr 14, 2015. http://www.epi-global.com/en/about-tdpa.php

[31] Wells Plastics Limited, "Why Reverte®?" Accessed Apr 14, 2015. http://www.reverteplastics.com/eng/reverte.php

[32] Add-X Biotech, "AddiFlex®" Accessed Apr 14, 2015. http://www.add-xbiotech.com/products.aspx

[33] Symphony Environmental, "*D_2w Oxo-biodegradable Plastic*" Accessed Apr 14, 2015. http://www.symphonyenvironmental.com/d2w

[34] P-Life Japan Inc, "P-Life" Accessed Apr 14, 2015. http://www012.upp.so-net.ne.jp/p-lifeasia/html/gijyutuen.htm

[35] Jakubowicz, Ignacy, Nazdaneh Yarahmadi, and Veronica Arthurson. "Kinetics of abiotic and biotic degradability of low-density polyethylene containing prodegradant additives and its effect on the growth of microbial communities."*Polymer Degradation and Stability* 96, no. 5 (2011): 919-928.

[36] Scoponi, Marco, Fiorella Pradella, and Vittorio Carassiti. "Photodegradable polyolefins. Photo-oxidation mechanisms of innovative polyolefin copolymers containing double bonds." *Coordination chemistry reviews* 125, no. 1 (1993): 219-230.

[37] EcoLogic, "HOW ECO-ONE™ WORKS." Accessed Apr 14, 2015. http://www.ecologic-llc.com/product/how-it-works

[38] *BioTec Environmental,* "About EcoPure®: An Organic Plastic Biodegradation Additive." Accessed Apr 14, 2015. http://www.goecopure.com/about-us

[39] TekPak Solutions, "Omnidegradable Packaging" Accessed Apr 14, 2015. http://www.tekpaksolutions.com/discovery.php

[40] ENSO Plastics, "Why ENSO Biodegradable Plastics." http://www.ensoplastics.com/Products/Products.html

[41] Lake, John Allen, and Samuel David Adams. “CHEMICAL ADDITIVES TO MAKE POLYMERIC MATERIALS BIODEGRADABLE.” BIO-TEC ENVIRONMENTAL LLC, assignee. Patent 2008/0103232. May 1 2008

[42] ASTM D5210 − 92, 2007, "Standard Test Method for Determining the Anaerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge." *ASTM International*, 2007. www.astm.org.

[43] ASTM D5511 – 12, 2012, "Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under High-Solids Anaerobic-Digestion Conditions." *ASTM International*, 2012. www.astm.org.

[44] ASTM D5526 – 12, 2012, "Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under Accelerated Landfill Conditions." *ASTM International*, 2012. www.astm.org.

[45] ASTM D7475 – 11, 2011, "Standard Test Method for Determining the Aerobic Degradation and Anaerobic Biodegradation of Plastic Materials under Accelerated Bioreactor Landfill Conditions." *ASTM International*, 2011. www.astm.org.

[46] ISO 13975:2012, "Plastics -- Determination of the ultimate anaerobic biodegradation of plastic materials in controlled slurry digestion systems -- Method by measurement of biogas production." *International Organization for Standardization*, 2012. www.iso.org/

[47] ISO 14853:2005, "Plastics -- Determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system -- Method by measurement of biogas production." *International Organization for Standardization*, 2005. www.iso.org/

[48] ISO 15985:2014, "Plastics -- Determination of the ultimate anaerobic biodegradation under high-solids anaerobic-digestion conditions -- Method by analysis of released biogas." *International Organization for Standardization*, 2014. www.iso.org/

[49] Fernández, J., M. Pérez, and L. I. Romero. "Kinetics of mesophilic anaerobic digestion of the organic fraction of municipal solid waste: influence of initial total solid concentration." *Bioresource technology* 101, no. 16 (2010): 6322-6328.

[50] Chen, Ye, Jay J. Cheng, and Kurt S. Creamer. "Inhibition of anaerobic digestion process: a review." *Bioresource technology* 99, no. 10 (2008): 4044-4064.

[51] Shin, Hang-Sik, Kyu-Seon Yoo, and Jae K. Park. "Removal of polychlorinated phenols in sequential anaerobic–aerobic biofilm reactors packed with tire chips." Water environment research 71, no. 3 (1999): 363-367.