

# **Contractor's Report to the Board**

## *Evaluation of the Performance of Rigid Plastic Packaging Containers, Bags, and Food Service Packaging in Full-Scale Commercial Composting*

***Produced under contract by:***

*California State University  
Chico Research Foundation*



*March 6, 2007*



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Publication #432-07-003

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*Prepared as part of contract IWM-C2061 (total contract amount: \$65,000, includes other services).*

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# Table of Contents

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List of Figures .....	ii
List of Tables .....	ii
Acknowledgements.....	i
Executive Summary .....	1
Introduction.....	3
Background.....	4
Types and Performance of Compostable RPPCs, Food Service Products, and Bags .....	6
Starch-based Polymers .....	9
Polyester-based Polymers.....	9
Cost of Compostable and Biodegradable Plastics .....	10
Degradation, Residuals, Toxicity of Compostable and Biodegradable plastics .....	10
Testing Standards for Compostable Plastics .....	14
Testing Plan .....	15
Experimental Work.....	16
Materials .....	16
Experimental Methods and Procedures .....	18
Laboratory Environment .....	20
Carbon Dioxide Concentration Results .....	20
Biodegradation Results.....	21
Phytotoxicity Testing .....	28
Heavy Metal Testing .....	28
Results .....	29
University Farm Compost Facility.....	31
Materials .....	31
Experimental Set-up .....	32
Procedure.....	33
University Farm Degradation Results .....	34
City of Chico Municipal Compost Facility.....	37
Materials .....	37
Experimental Set-up .....	38
Procedure.....	38
City of Chico Compost Facility Degradation Results .....	40
Conclusions.....	41
Recommendations.....	42
Appendices.....	43
Appendix A. Calculations .....	44
Appendix B. Pictures of Samples at the CSU, Chico Experimental Laboratory.....	46
Appendix C. Pictures of Samples at the CSU, Chico Farm .....	49
Appendix D. Pictures of Samples at the City of Chico Municipal Compost Facility .....	52

# List of Figures

---

Figure 1. Experimental set-up for laboratory environment.....	19
Figure 2. Sampling process schematic. ....	20
Figure 3. Carbon conversion percentage for compost control alone.....	23
Figure 4. Carbon conversion percentage for cellulose control. ....	24
Figure 5. Carbon conversion percentage for Kraft paper control. ....	24
Figure 6. Carbon conversion percentage for polyethylene negative control.....	25
Figure 7. Carbon conversion percentage for corn based BioBag trash bag. ....	25
Figure 8. Carbon conversion percentage for corn PLA clamshell container. ....	26
Figure 9. Carbon conversion percentage for corn PLA cup.....	26
Figure 10. Carbon conversion percentage for sugar cane plate. ....	27
Figure 11. CO <sub>2</sub> ppm concentration of BioBag trash bag after 21 days.....	28
Figure 12. Temperature of the air and compost during the duration of the university farm experiment. ....	33
Figure 13. Temperature of the air and compost at the City of Chico Compost Facility. ....	39

# List of Tables

---

Table 1. Commercially Available Biodegradable and Compostable Polymers .....	8
Table 2. Compostable product information for laboratory experiment. ....	17
Table 3. Designed-use analysis for compostable products. ....	18
Table 4. Heats of combustion, carbon content, and moisture % for compostable samples. ....	22
Table 5. Degradation rates for compostable samples.....	23
Table 6. Phytotoxicity of Compost Soil. ....	30
Table 7. Compostable Product Information for University Farm Experiment .....	32
Table 8. Carbon dioxide percentages from compostable materials at the university farm .....	35
Table 9. Disintegration results for compostable plastics at the university farm. ....	36
Table 10. Compostable product information for City of Chico compost experiment.....	37
Table 11. Carbon dioxide percentage of compostable materials at the Chico Municipal Compost Facility. ....	39
Table 12. Material Degradation Results for Compostable Samples at the Municipal Compost Facility.....	40

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# Acknowledgements

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This report is a culmination of work from many people who represent many organizations. The author would like to thank the following people and organizations who have helped develop this project; Mr. Edgar Rojas (CIWMB), Mr. Mike Leao (CIWMB), Dr. Cindy Daley (CSU, Chico), Dr. Ken Derucher (CSU, Chico), Mr. Tim Devine (CSU, Chico), Dr. Greg Kallio (CSU, Chico), Mr. Mitch Kofford (CSU, Chico), Dr. Randy Miller (CSU, Chico), Mr. Peter Natale (CSU, Chico), and Mr. Dale Wangberg (Waste Management Company, Chico Compost Facility).

Produced under a CIWMB contract with CSU, Chico Research Foundation. Contacts are Mr. Edgar Rojas (CIWMB. 916-341-6508) and Dr. Joseph Greene (CSU, Chico. 530-898-4977).

# Executive Summary

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The research in this report details the results of biodegradation testing of several compostable plastics that are commercially available in California. The manufacturers of these compostable plastic products claim to meet the ASTM D6400 standards for degradation, sustainable plant growth, and eco-toxicity in compost environments. The objectives of the research are to evaluate the compostability of these compostable plastic products and to test the compost residual soil for the presence of heavy metals and the ability to support plant life. The project includes a review of current research literature of compostable and biodegradable materials, as well as, degradation testing of several compostable plastics in three compost environments.

The first compost environment is a laboratory setting that follows the standards outlined in ASTM D6400. Pieces of five compostable plastic products, along with two positive controls of cellulose paper and Kraft paper and one negative control of polyethylene plastic wrap, were placed in a controlled warm and humid environments of 58°C for 45-days. The degradation was evaluated by measuring CO<sub>2</sub> gas, which evolves from the degrading compostable samples while in 2-Liter jars. The samples were tested in triplicate for each material.

The second compost environment is a commercial compost production facility at the university farm that is made from a mixture of cow manure and straw. The compostable samples were placed in perforated plastic bags with an appropriate amount of mature compost. The bags were buried in a compost mound, which is on a cement slab. The bags were buried approximately 1-meter below the surface. The mass of the compostable material was recorded over a 7-week degradation experiment along with the temperature of the air and of the compost mound.

The third compost environment is a commercial compost facility at the city of Chico municipal site that is produced from green, yard waste. As with the university farm experiment, the compostable samples were placed in perforated plastic bags with an appropriate amount of compost. The bags were placed in the compost mound that is in a large field. The bags were buried approximately 1-meter below the surface. The mass of the compostable material was recorded over a 20-week experiment along with temperature of the air and compost mound.

The biodegradation results in the laboratory environment demonstrate that the compostable materials degrade under compostable conditions, though the corn-starch based Biobag trash bag did not meet the degradation rate as defined in the ASTM D6400 standards. The cellulose positive control met the ASTM 70% degradation requirement. The degradation rates of the materials are listed according to highest rates as follows, cellulose control, sugar cane plate, Kraft paper control, PLA container, PLA cup, and corn-starch based Biobag trash bag. The sugar cane and PLA materials had degradation rates similar to the Kraft paper control and meet the compostability criterion of 60% degradation after 45-days. The polyethylene negative control and the compost inoculum soil demonstrated negligible degradation.

The trash bag degraded during the test but did not meet the compostability standards specified by ASTM. The trash bag poor results can be attributed to excess moisture in the test jars that was noted during several days in the experiment which limits the amount of oxygen available and can reduce biodegradation. The trash bag was retested with improved test methods at a later date and was found to have degradation similar to the Kraft paper control over a three-week time period. The results for the trash bag are inconclusive until a new full 45-day test, according to ASTM D-5338 standards, is completed.

All of the soil samples from the compostable materials had lead concentrations of 0.02 mg/kg, which is well below the maximum limit of 30 mg/kg for California. The cadmium concentrations

were also well below the maximum limit of 17 mg/kg. In fact, the amounts of lead and cadmium were less than 1% of the maximum allowable levels.

The PLA cup and container and the trash bag met the phytotoxicity requirements (poisonous to plants) and support growth of tomato seedlings after 10-days. The sugar cane plate, however, did not support growth during the test. The causes of the lack of plant growth can be attributed to inability of tomato seeds to adequately test for phytotoxicity. The Cress seed test is a more robust test. The phytotoxicity testing was repeated at a later time with lower amounts of samples and compost, though the ratio between the two was identical to the earlier tests. In the new test, 100 g of compost soil was blended with 16 g of sugar cane samples. The sugar cane demonstrated biodegradation in the new test after 30 days. The tomato seed test was repeated and seedlings grew in the presence of degraded sugar cane. The compost soil had ethanol and butanol in the compost due to the fermentation, though in not very high concentrations to affect phytotoxicity. The phytotoxic results then are inconclusive and should be repeated with concentrations specified by ASTM D-5338 standards.

The degradation and disintegration results at the university farm demonstrate that the compostable materials degrade under moist manure-based compost. All of the materials disintegrated after 72 days. The potato-starch based tray, corn-starch based trash bag, PLA plate, PLA straw, and PLA container degraded at similar rates as the cellulose control.

The degradation and disintegration results at the municipal compost facility demonstrate that the compostable materials degrade under moist green-waste compost. The PLA container, PLA cup, and PLA knife degraded at a similar rate as the Avicell cellulose control and were degraded completely in 7-weeks. The cornstarch-based trash bag and sugar cane plate degraded at a similar rate as the Kraft paper control. The three materials degraded between 80 and 90% after 20 weeks.

The three compost environments demonstrate similar results. In particular, PLA degrades very well in cow-manure and green waste compost. The trash bag experienced higher degradation in the moist cow manure compost than in the green waste compost. The cow manure compost is the most active and the best medium for degradation of the PLA and starch based compostable materials. The laboratory and municipal compost had similar degradation results, where the PLA materials degraded very quickly and the starch based plastic bag degraded more slowly. The trash bag had similar degradation rates after 45 days in the laboratory and in the municipal compost facility of around 30% degradation. The Kraft paper sample also had similar degradation in the laboratory environment (61%) as in municipal compost facility (52%). The sugar cane plate had the biggest difference in degradation rates between the two compost environments with higher degradation in the laboratory (63%) versus the municipal compost (19%) after 45 days. The moisture content was significantly higher in the laboratory experiment than at the municipal compost facility. The sugar cane plate is hydrophilic that can affect the degradation rate.

The research work can help increase the use of compostable plastic materials for selected applications. The compostable materials should be certified as compostable by BPI and included in procurement standards. The compostable plastic materials should perform well in simple applications, e.g., food service ware, lawn and leaf refuse bags that have dry contents, grocery bags, department store bags, and pet bag products. The compostable plastics would not most likely perform well in trash bag uses due to the likely exposure to moist debris. Thus, trash bag use is not recommended at this time. Compostable plastic materials could be very economical for organizations and institutions that service a controlled population, e.g., hospitals, correctional facilities, schools, and cruise ships.

# Introduction

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The California Integrated Waste Management Board (CIWMB) initiated a research project to study the degradation rates and performance of compostable plastics that can be used in rigid packaging plastic containers, trash bags, film liners, and food service products. The Department of Mechanical Engineering Mechatronic Engineering and Manufacturing Technology at California State University, Chico was hired to study the performance, degradation rates and byproducts of various compostable rigid packaging containers, other food service products, and bags using commercial composting methods. The research objectives in the research project are to evaluate several compostable plastic products that are sold in California and their respective quality.

The project is broken down into four areas, including a detailed work plan and budget, literature review, demonstration project, and evaluation report. The research can help manufacturers of compostable products, government agencies, and consumers better evaluate environmental claims of compostable plastic materials. The compostable plastics will be exposed to three different environmental environments that are common in commercial compost facilities.



# Background

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Plastics are seemingly ubiquitous in our world today. At the end of the service life, plastic products can be either collected for recycling or thrown away with the trash. Waste disposal companies usually collect the plastics with other recycled products. Plastics, metals, and glass are sorted from the refuse and sent to recyclers. The solid waste can be recycled or sent to an incinerator or landfill. As reported in a Statewide Waste Characterization Study, approximately 350,000 tons of rigid plastic packaging containers (RPPC) were disposed of in California during 2003 which represents approximately 1% by weight of the overall waste stream. Plastic trash bags comprised 1% and plastic film comprised 2.3% of the waste stream.<sup>[1]</sup> The commercial sector generated approximately 50% of the solid waste, the residential sector generated approximately 30% of the solid waste, and the self-hauled sector generated approximately 20% of the solid waste. In 2003, plastics contributed to 12% by weight of the waste stream for the commercial waste, 9.5% of the waste from residential waste, and 3.9% of the waste stream in self-hauled waste.<sup>[2]</sup> Food scrap composting can lead to significant diversion of waste products in landfills. The use of biodegradable and compostable plastics in California can reduce the amount of plastics in the landfills. Composting is a promising waste management option for degradable plastics because the composting process is designed to degrade wastes.

Several organizations, while small, are involved in setting standards for biodegradable and compostable plastics, including, US Composting Council (USCC), American Certification System of Biodegradable Products Institute (BPI), Environment & Plastics Industry Council, American Society for Testing and Materials (ASTM), European Committee for Standardization (CEN), Japan's GreenPla program, and British Plastics Federation. The standards from these organizations have helped the industry create biodegradable and compostable products that meet the increasing worldwide demand for more environmentally friendly plastics.<sup>[3]</sup> If a biodegradable polymer does not meet the requirements listed in ASTM D6400 or EN13433, then it is not considered compostable. It must degrade in a specified time frame without leaving any distinguishable residuals in the compost.<sup>[4]</sup>

Biodegradable polymers are those that are capable of undergoing decomposition into carbon dioxide, methane, water, inorganic compounds or biomass by the actions of microorganisms. The rate of decomposition, residuals, and by-products can be measured in standardized tests. Compostable polymers are those that are degradable under composting conditions, which includes actions of microorganisms, i.e., bacteria, fungi, and algae, under a mineralization rate that is compatible with the composting process. Polyethylene plastic bags that are produced with starch additives are not certified as compostable plastics since they do not meet the ASTM D6400 standards. The plastics do disintegrate but leave small plastic fragments in the compost, which violates the ASTM D 6400 standards. The ASTM D6400 standard differentiates between biodegradable and degradable plastics. Some synthetic polymers, e.g., Low Density Polyethylene (LDPE), can erode over time if blended with additives to facilitate degradation. These polymers break down into small fragments over time but are not considered biodegradable since they do not degrade at a rate specified in the ASTM D6400 standards. Oxo-degradable polymers, photodegradable polymers, and bioerodable polymers break down in environments different from the biodegradable and compostable polymers and as such are outside the scope of the research.

The Biodegradable Products Institute (BPI) provides important criteria for valid full-scale testing of compostable plastics.<sup>[5]</sup> The BPI Logo Program is designed to certify and identify plastic products that will biodegrade and compost satisfactorily in actively managed compost facilities.<sup>[6]</sup> The Biodegradable Products Institute and US Composting Council (USCC) use ASTM D6400 standards to approve products for their compostable logo. The ASTM standards are the result of eight years of

intensive work to identify plastic and paper products, which disintegrate and biodegrade completely and safely when composted in a municipal or commercial facility. The approved products with a compostable logo include compostable bags and film, food service items, and resins. Many of the compostable products studied in this research have the BPI compostable logo.

Composting is a waste treatment option for treating post-consumer organic materials. Many companies treat organic residue in a compost environment to provide compost as a commercial enterprise. Plastics can be used as nutrient for the compost operation if it is made from natural materials, e.g., corn, rice, potato, sugar cane etc. Compostable plastics are commercially available and are being used as replacements for synthetic plastic materials. Biodegradable plastics can be made into different commercial products, including, trash bags, food containers, packaging trays, plastic utensils, and packaging containers and bags. The use of biodegradable polymers is increasing at a rate of 30% per year in some markets worldwide.<sup>[7]</sup> There are, however, obstacles that cause concern for many communities to accept plastic bags for composting.<sup>[8]</sup> Degradable plastic bags that are effective in compost environments are found to retain moisture, have comparable mass as the composting feedstock, and begin to degrade after several days.<sup>[9]</sup>

The compostable plastics must not be a source of pollution or contamination to the commercial compost facilities and they must break down at rates similar to standard compost materials, e.g., yard waste, manure, paper, or cellulose. The purpose of the research study is to compare the degradation rates of several compostable plastics and to assess the ecotoxicity of the compost after exposure to the degraded materials. The compostable plastics used in the research are commercially available and claim to be compostable.

The plastics will be tested in three environments. First, the plastics are tested in a laboratory setting according the procedures outlined in ASTM D5338. The plastics are placed with mature compost in a 2-liter jar at a controlled temperature of 58°C and approximately 50% moisture. The CO<sub>2</sub> and O<sub>2</sub> gasses that evolve from the compostable plastics are monitored over a 45-day time period to quantify the degradation rate of the plastic. The second environment that the plastics are exposed to is a young compost material at the university farm. The compost is made from cow manure and straw. The plastics are placed in perforated plastic agricultural bags that are buried in the compost area. The third environment that the plastics are exposed to is a commercial compost operation in the City of Chico. The compost is young and active and made from yard waste. The second and third environments allow the study of the compostability of the plastics in compost environments that are similar to commercial compost facilities throughout the state of California.

# Types and Performance of Compostable RPPCs, Food Service Products, and Bags

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Many types of biodegradable polymers are available to degrade in a variety of environments, including, soil, air, or compost. A majority of biodegradable polymers are made up of wheat, corn, starch, cellulose, collagen, casein, soy protein polyesters, or triglycerides.<sup>[10]</sup> In the agriculture industry, biodegradable polymers can be used as pots for plants. They can be also used as ground coverings to assist the growth of plants and weed control. In packaging, compostable and biodegradable polymers are formed into trays for candies, bottles, cups, and clear clamshells for food service products. Trash bags, films, and sheet can be made from compostable plastics for household purposes. The bags, film, and sheet products can be thrown away along with any composting materials. The city of San Francisco and Norcal Waste Systems Company plan to use Biocorp compostable bags for the citywide composting program.<sup>[11]</sup>

The performance of compostable plastic products is highly dependent upon the physical properties of the materials. The mechanical properties of some compostable plastics are similar to polyethylene plastic. Tensile properties, dynamic mechanical properties, and impact properties of polylactic acid (PLA) were found to increase with the addition of polybutylene succinate (PBS).<sup>[12]</sup> The degradation rate also increased with the addition of PBS. Blends of thermoplastic starch (TPS) and hydrophobic biodegradable polymers can be made into compostable plastic film. The biodegradable plastic film has properties similar to polyethylene film. It can be made opaque to transparent, printable, sealable, shrinkable, and can be colorized. It can be made to be permeable to vapor and has good oxygen barrier properties.<sup>[13]</sup> Poly Lactic Acid (PLA) has properties similar to polyethylene terephthalate (PET). Compostable plastics can meet the hygienic requirements of the FDA regulations if the plastic is used in contact with food products. Mater-Bi Bio Bags™ can reduce the hygienic concerns associated with using biodegradable plastics in food waste containers. The compostable bags can provide weight reduction and hygienic benefits by using ventilated food waste bags. The Mater-Bi bags were found to have significantly lower odor and water leakage from organic solid waste as compared to non-ventilated bags.<sup>[14]</sup> Mater-Bi materials can provide a safe barrier for virus, bacteria and other potential pathogens that are found in packaging and surgical gloves.

Biodegradable polymers can be made into a rigid or flexible plastic products, which allows each polymer to fit particular market applications. Biodegradable polymers can be made into bags or sheet products like low-density polyethylene (LDPE). The biodegradable polymer is made from at least 90% starch from renewable resources, i.e., corn, potato, tapioca, wheat. The other 10% is water, plasticizers, or other degradable processing aids. Biodegradable polymers can be also made into more rigid packaging products to replace polyethylene terephthalate (PET). The polyesters are produced from hydrocarbons and degrade within a couple of weeks in compost soil for polyhydroxyalkanoates versus decades for typical thermoplastic aromatic polyesters, such as PET. Several packaging materials produced from biodegradable aliphatic polyester polymers successfully meet U.S., European, Australian, and Japanese standards by degrading in 12-weeks under aerobic conditions in a compost environment and by breaking down to CO<sub>2</sub> and H<sub>2</sub>O.<sup>[15]</sup>

Polylactic acid (PLA), which is manufactured and supplied by Cargill Dow is a very important biodegradable polymer. It is a very common biodegradable polymer that has high clarity for packaging applications. It can be used for thermoformed cups, candy wraps, optically enhanced films, and shrink labels. PLA, unfortunately, has limited use due to its high cost.<sup>[16]</sup> Table 1 lists commercially available biodegradable and compostable polymers.<sup>[17]</sup> The biodegradable products degrade to carbon dioxide and water in the presence of oxygen. Biomass can also be formed with

some plastics that are not produced from organic sources. The PLA plastics are produced in hundreds of millions of pounds per year and are available to customers around the world. In 1999, Dow Chemical and Cargill created a joint venture, named, Cargill-Dow to become the largest biodegradable polylactic acid (PLA) producer with in the world with annual capacity of 140,000 metric tons per year of Natureworks<sup>TM</sup> PLA.<sup>[18]</sup> The plastic is targeted for rigid packaging and fiber applications as an alternative to PET.<sup>[19]</sup>

Researchers at Argonne National Laboratory, in Argonne, Illinois are developing PLA biodegradable plastic from potato starch, which should lead to commercialization with several industrial partners, including General Electric and Henkel Corporation.<sup>[20]</sup>

In Europe, compostable plastic bags are currently available for supermarket carrier bags, “knot” bags for fruit and vegetable in supermarkets, kitchen waste bags, and garden waste sacks.<sup>[21]</sup> Eastman Chemical opened an Eastar Bio plant in the U.K. in 2002 with a production capacity of 33 million pounds per year.<sup>[22]</sup> The total European Union polymer consumption for plastic bags and sacks is on the order of 2 to 2.5 M tones per year in 1999. The total consumption of all biodegradable polymer products in European Union was estimated to be 25,000 to 20,000 tonnes per year.<sup>[23]</sup> Approximately, 8,000 tonnes per year of Novamont’s Master-Bi corn starch plastic bags are used.<sup>[24]</sup>

In Australia, biodegradable polymers applications are being used in grocery, retail, and compost industries as bags for fruit, bait, bread, and ice.<sup>[25]</sup> Australia uses the European standards for compostable and biodegradable plastics certification. Biodegradable plastic bags are available in the local bottle-shop liquor stores. The environmentally friendly bags are made from Matter-Bi<sup>TM</sup> biodegradable plastic.

**Table 1. Commercially Available Biodegradable and Compostable Polymers\***

Material	Type	Supplier/ Distributor	Products	Degradation Products	Extent of Degradation	Standard Met
Biomax™	aliphatic copoly- esters, modified PET	Dupont/ www.allcompost.co m	Coating and film for food packaging, sandwich bags, utensils, fibers.	Carbon dioxide, water, biomass.	2 to 4 months in compost depending upon temperature	ASTM D6400
Biopol™	PHB/V polybuty- rate and valeric acid	Metabolix Inc/ Biocorp	Consumer disposables, Containers, trash bags, packaging	Carbon dioxide, water.	20 days in sludge, to 1 month in compost	ASTM D6400, EN13432
Eastar Bio™	Biodegrad- able copolyester	Eastman Chemical Company/ Farnell Packaging Biodegradable Products	Trash bags, film, liners	Carbon dioxide, water, biomass.	2 to 4 months in compost depending upon temperature	ASTM D6400, EN13432
Ecoflex™	Aliphatic- aromatic Polyester	BASF/ www.allcompost.co m	Compost bags, trash bags, carrier bags, fruit and vegetable bags.	Carbon dioxide, water, biomass.	2 to 6 months in compost depending upon temperature	ASTM D6400, EN13432
Mater- Bi™	60% starch and 40% polyvinyl alcohol	Novamont/ BioBag Corporation	Trash bags, lawn and garden bags	Carbon dioxide, water, biomass.	3 to 6 months in compost depending upon temperature	ASTM D6400, EN13432, BPI
Nature- Works™	Polylactic acid (PLA)	Cargill Dow/ Biodegradable Food Service, Eco- Products, Inc.	Clear cups, clamshells, salad bowls	Carbon dioxide, water	1 to 3 months in compost depending upon temperature	ASTM D6400, EN13432
Plantic™	Starch- PVOH	Plantic Technologies of Australia/ same	Rigid containers, trays	Carbon dioxide, water.	1 to 2 months in compost depending upon temperature	EN 13432

\*Note: The polymers are available in bag, Gaylord, or truckload quantities.

## **Starch-based Polymers**

Starch-based polymers can be produced from potato, corn, wheat, or tapioca. These polymers can be processed on thermoplastic forming operations, e.g., extrusion, injection molding, rotational molding, blow molding, etc. Starch can be the basis of a biodegradable plastic since it is produced from renewable resources. A biodegradable polymer may include additives to improve the properties. Three such polymers are Mater-Bi™<sup>[26]</sup> made from 60% starch with vinyl alcohol, ammonium hydroxide, and urea, NOVON™ made from 90-95% starch and 5% additives, and AMIPOL™ made from 100% starch.<sup>[27]</sup> Novon International produces NOVON™, a starch-based polymer that contains performance enhancing additives, such as synthetic linear polymers, plasticizers, and components that enhances degradability. Water is the most common dispersant agent for starch-based plastics. The starch is not typically modified with acid treatments, chemicals, or enzymes.

A common biodegradable polymer made from a combination of starch and polyester is Ecoflex™ manufactured by BASF. Ecoflex™ is known for its blown film applications such as packaging films, agricultural films, hygienic films, and trash bags. It has similar properties to a commodity polymer, LDPE. Ecoflex™ provides a compostable plastic material to produce trash bags. It is made from aliphatic-aromatic copolyester blended with equal amounts of starch. Ecoflex™ meets the requirements for biodegradable polymer classification from the European,<sup>[28]</sup> U.S., and Japanese standards because Ecoflex™ can be degraded by micro-organisms.<sup>[29]</sup> Ecoflex™ is a compostable material. The Biodegradable Products Institute (BPI) granted its "Compostable Logo" to BASF's Ecoflex™ resins for use in biodegradable films and coatings.<sup>[30]</sup>

In Australia and Europe, Cadbury Chocolates of Australia have selected Plantic™, a biodegradable polymer from Plantic Technologies of Australia, for thermoformed trays that holds individual chocolates in their box of chocolates. The compostable plastic material is made from starch. It meets the European standard DIN EN 13432 for biodegradation.

## **Polyester-based Polymers**

The majority of compostable plastics belong to the polyester family, including poly-lactic acid (PLA), poly-caprolactone (PCL), poly-butyrate adipate terephthalate (PBAT), aliphatic copolyesters, modified PET (Biomax™), and polyhydroxybutyrate blended with poly-3-hydroxy-butyrate-valerate (PHB/V).<sup>[31]</sup> The biopolymer of PHB and PCL can be used for plastic bags. Plastic bags made from these materials decompose completely to carbon dioxide and water by microorganisms.<sup>[32]</sup> Polylactic acid (PLA) is a synthetic and renewable aliphatic polyester that has a potential for use in compostable and biodegradable plastic bags. The biopolymer PLA bags from Cargill Dow are being used in Taiwan for commercial packaging products. The bags are referred to as Nature Green™. PLA is a bio-based plastic made from corn. Cargill Dow claims that the material performs as well as traditional plastics and fits all current disposal systems, including in industrial compost facilities.<sup>[33]</sup> NEC Corporation in Tokyo reports that natural-fiber reinforcements derived from the Kenaf plant can increase PLA's rigidity and heat resistance by 70% to 80%. NEC reports that PLA reinforced with 20% (by weight) Kenaf fibers has a heat-distortion temperature of 248°F and is expected to find commercial use in computer housings.<sup>[34]</sup>

PLA though is not well suited for flexible film production other than for biaxially oriented sheet substitution. If starch is blended with the PLA, the flexibility can be increased. However, it is necessary to add low molecular weight plasticizers to reduce the brittleness of starch. In the Netherlands, grocery shoppers use clear, flexible, compostable bags made from Cargill Dow's corn-based NatureWorks PLA, rather than traditional petroleum-based plastic film.<sup>[35]</sup> The starch blend also increases biodegradability and reduces the cost. The brittleness can be reduced with the

addition of plasticizers.<sup>[36]</sup> Polyester-based compostable plastics can be used for other rigid packaging containers, including, trays, cups, and containers.

## ***Cost of Compostable and Biodegradable Plastics***

The compostable plastic products are more expensive than conventional plastics due in part to its low-scale production. If more products are purchased and the production rate rises to full-scale production the price can be reduced. Biodegradable plastic products currently on the market are from 2 to 10 times more expensive than traditional plastics. The cost for biodegradable polyesters varies from \$1.50 to \$2.00 per pound. The specific gravity can vary between 1.22 and 1.35.<sup>[37]</sup> Some environmental organizations argue that the cheaper price of traditional plastics does not reflect their true cost when you consider the costs of disposal and environmental impact.

The high cost is a disadvantage to the compostable plastic when compared to paper, LDPE, PP, PS, and PET. An Australian company is trying to produce affordable biodegradable plastics by incorporating low cost materials and processing methods.<sup>[38]</sup> Metabolix Incorporated recently introduced PHBV with a new fermentation process that can provide the compostable plastic in large-scale production volumes at around \$1 per pound.

Nat-UR Cutler Food Service provides biodegradable spoons, knives, and forks for a price of \$15.50 for 240 pieces. Nat-UR also sells compostable trash bags for San Francisco residents. The cost of 40 bags of 20-gallon size is \$19. They also offer a plates and trash bags at a cost of \$24 for 100 plates and \$24 for 40 bags of 40-gallon size, respectively. Plastic cups are available as well at a cost of \$26 for 100 10-oz cups. All of the products claim to meet ASTM D6400-99 standards.

Several companies provide compostable RPPCs, cutlery, and bags.<sup>[39]</sup> NatureWorks PLA is made into many different types of containers, including cups, lids, plates, and storage containers. The costs of 1000 pieces can range from \$25 to \$145.<sup>[40]</sup>

Other environmentally friendly and biodegradable bags and cups are available at Biobag USA Corporation.<sup>[41]</sup> The bags are produced from Mater-Bi materials, which are supplied by Novamont, an Italian research company. All of the Biobag products meet the ASTM D6400 standard for degradation and safe residues and are certified by the US Composting council and meet the California state law regarding biodegradation. Biobag products are available for bags and liners, shopping bags, pet products, composting system, Agro Film, and toilet systems. Retail Biobags are available for kitchen bags, garden film, toilet system, and Nature Waste Bags. Biodegradable Plastic Cups are also available. The costs range from \$0.08 to \$0.20 per bag and \$0.07 to \$0.14 per cup. The costs of biodegradable plastic bags are expensive when compared to cost of typical polyethylene bags of \$0.01 to \$0.02 per bag.

## ***Degradation, Residuals, Toxicity of Compostable and Biodegradable plastics***

Compostable polymer products undergo degradation that leads to the conversion of the polymer into carbon dioxide in aerobic conditions, carbon dioxide/methane in anaerobic conditions and water. Degradation can only occur when the polymer is exposed to microorganisms found naturally in soil, sewage, river bottoms, and other similar environments. The breakdown of degradable plastics has been categorized into disintegration and mineralization.<sup>[42]</sup> Disintegration occurs when the plastic materials disintegrate and are no longer visible, but the polymer still maintains a finite chain length. Microorganisms can degrade the polymers when the polymer chain is broken down to very small molecular units. Mineralization occurs when the polymer chains are metabolized by microorganisms after the initial oxidation process to carbon dioxide, water, and biomass.

Four mechanisms are often involved in the degradation of plastics, namely, oxidation of polymers, microbiological digestions of natural ingredient, such as starch or cellulose, microbiological digestion of the biodegradable polymer, such as aliphatic polyesters, and the microbial digestion of polymer fragments.<sup>[43]</sup> The compostability of biodegradable plastics are also dependent upon the microorganisms present in the compost soil. The growth rate of the organisms depends upon the temperature, moisture, pH, and carbon to nitrogen ratio, also referred to as compost maturity index. The plastics will degrade more quickly at higher temperatures and higher moisture content. The rate of degradation of plastic bags made from degradable plastic is very dependent upon the polymer type, material thickness, moisture level, temperature, and amount of bacteria present.<sup>[44]</sup> Microorganisms isolated from soil samples were screened for their ability to degrade several polyester-based plastics. One reactive strain could degrade polylactic acid, poly butylenes succinate, polycaprolactone, and polyethylene succinate, but not polyhydroxybutyrate-co-valerate.

The compost activity of poly- $\beta$ -hydroxybutyrate (P $\beta$ HB) and a copolymer of 20% - $\beta$ -hydroxyvalerate (P $\beta$ HB-P $\beta$ HV) were measured in a simulated municipal solid waste compost environment at 55°C and a constant moisture content of 54%.<sup>[45]</sup> Polymer disintegration was measured through weight loss. The PVHV degraded faster than the PHB and met the degradation standards for ASTM D6400. Poly-caprolactone (PCL) degraded in a compost environment at a controlled temperature of 50°C and 45% to 55% moisture levels.<sup>[46]</sup> The compost was taken from a municipal solid waste facility. CO<sub>2</sub> was measured by passing the gas through a conical flask containing H<sub>2</sub>SO<sub>4</sub> solution to absorb the NH<sub>3</sub> and then to an infrared analyzer to measure the CO<sub>2</sub>. After 11 days 59% of the PCL degraded in the compost. The degradation rate can be increased to 85% in 11 days if the compost is mixed with dog food. Mature compost has a lower temperature and lower moisture content than fresh compost which results in a lower degradation rate.

Moisture content and temperature of the environment can affect the degradation rate. The degradation behavior of starch-based polymers was found to be highly dependent upon on the presence of moisture and temperature.<sup>[47]</sup> Higher moisture content and higher temperatures lead to increased biodegradation rates. However, moisture content greater than 70% can retard degradation due to reduced amount of oxygen exposure. The degradation behavior of three commercial biodegradable polymers, i.e., poly 3-hydroxybutyrate (PHB), Sky-Green™ (SG), aliphatic polyester made from succinic acid, adipic acid, butenediol, and ethylene glycol and Mater-Bi™, a composite composed of starch based biodegradable polymers were incubated in forest soil, sandy soil, in activated sludge soil, and in farm soil at three temperatures.<sup>[48]</sup> Seven PHB degrading fungi, five SG degrading fungi, and six MB degrading fungi were isolated by analyzing the microbiological characteristics of the fungi. Biodegradation of all three polymers was the most active in the activated sludge soil. The incubation temperature affected biodegradability of isolated fungi. The PHB degraded more than SG, which degraded more than MB.<sup>[49]</sup>

The degradation of compostable plastics can be monitored in compost environments. Eight kinds of biodegradable plastics were studied for their degradability in controlled laboratory composting environments.<sup>[50]</sup> The degradability of the biodegradable plastic was found to be dependent strongly on the type of polymer. The degradability of the eight kinds of plastics tested ranged from a small percentage to 65% over an 8-day period while composting at 50°C. In another study the biodegradability of five different biodegradable garbage bags was analyzed according to the DIN-standard.<sup>[51]</sup> The tests proved that a biodegradable polymer can be degraded under controlled composting conditions. The bags were made from cornstarch, polycaprolactone and Kraft paper. The results demonstrated that all five plastic products decomposed to the standards of 60% within six months. The bags were considered fully compostable since they degraded by breaking down into carbon dioxide and water, and left no toxic residue in the soil.



Mater-Bi™ compostable plastic is a wholly compostable polymer based on a blend of at least 50% starch with the remaining synthetic hydrophilic degradable polyester. The polymer was evaluated for suitability in disposal by composting.<sup>[52], [53], [54]</sup> The results indicate that Mater-Bi is readily degradable in standard laboratory biodegradation tests, including semi-continuous activated sludge (SCAS) test for simulating breakdown in municipal waste-water treatment plants and pilot composting systems. The degradation rate of Mater-Bi™ bags depends on the exact formulation used and physical properties of the product. Toxicity tests undertaken with the Mater-Bi™ bags and composted products have shown that they are non-toxic in the standard animal and plant tests.

The compostability and biodegradability of polymers can be tested in three different stages, lab-scale, pilot-scale, and full-scale operations. A pilot plant scale composter using simulated solid waste was found to undergo similar physical and chemical changes as a full scale composting system.<sup>[55]</sup> Screening levels and testing methods were developed to address the biodegradability and compostability of synthetic polymers. Degradation of the individual polymer materials were found to occur at different rates. Rates of biodegradation should be tested under realistic test conditions.<sup>[56]</sup> Information about the biodegradability of polymeric materials and products is required to understand the fate of polymers in the environment. Studies using Nuclear Magnetic Resonance (NMR) to spectroscopy analyze the degradation of individual components of polymeric materials. The biodegradation of heterogeneous components of polymers will occur at different rates and are not always representative of the material as a whole. Biodegradable and compostable packaging materials were tested in a commercial full-scale compost facility. The results demonstrated that the biodegradable polymers resulted in no negative effects on plant yield or soil.<sup>[57]</sup>

The compostable materials must also not leave any toxic residues or chemicals that negatively affect the compost soil quality. The quality of the compost can be evaluated for analytical and biological criteria, including soil density, total dry solids, salt content, inorganic nutrients content, and eco-toxicological behavior.<sup>[58]</sup> The inorganic nutrients evaluated in the compost are total nitrogen, phosphorous, potassium, magnesium or calcium, and ammonium-nitrogen. The eco-toxicological tests can include determination of growth inhibition with tomato and radish plants. The toxic effects on plants are referred to as “Phytotoxicity”. Plant phytotoxicity testing on the finished compost that contains degraded polymers can determine if the buildup of inorganic materials from the plastics is harmful to plants and crops and if they slow down soil productivity.<sup>[59]</sup> ASTM 6002 recommends OECD Guideline 208 for phytotoxicity testing. The testing procedure determines phytotoxicity by blending the compost containing the compostable plastic material with compost soil. The plant emergence survival and growth are evaluated. Three plant species are generally tested. The results from compost containing material are compared to compost without material and a soil control.<sup>[60]</sup> The plant species can be tomato, cucumber, radish, rye, barley, or grass. Plant biomass tests can reveal quality differences between composts and can indicate potential plant stress induced by the compost at the given level used in the test.<sup>[61]</sup> The test with tomato seeds is sensitive to maturity factors, nitrogen levels, and phosphorus. The tests are sensitive to cold environments.

Polymer residue from degraded plastics must not be harmful to plants growing in the soil. Chinese cabbage was shown to grow at the same yield in compost with 1% compostable plastic (PLA) as compost without the compostable plastic.<sup>[62]</sup> Biocompost from kitchen wastes produced improvements in the soil characteristics by enhancing the soil pH, organic matter, and reducing the nitrogen/carbon ratio in the soil.<sup>[63]</sup> A polymer based upon a blend of starch and Bionelle™ was found to completely mineralize to carbon dioxide in 45 days in a compost environment.<sup>[64]</sup> No indication of any pathology was found in earthworms exposed to the polymer or the residuals. The polymer is considered safe for the species of earthworms.

Some results suggest that the small polymeric fragments may provide useful properties as a soil additive. Grass growing studies using municipal waste derived compost in combination with chopped plastic fibers demonstrated improved growing rates and improved root structure development which can accelerate sod production.

Many additives used in plastic bags and rigid packaging containers, such as, plasticizers, color pigments, stabilizers, and degradation promoters, can contain toxic heavy metals. The heavy metals can make the compost unusable. Five biodegradable garbage bags that degraded in a compost environment according to the DIN standard were found to contain trace amounts of heavy metals. The bags were made from cornstarch, polycaprolactone, and Kraft paper. All five materials disintegrated in the compost at a mineralization rate of 60% within 5 months.<sup>[65]</sup>

# Testing Standards for Compostable Plastics

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In the U.S., ASTM D6400 is the acceptable standard for evaluating compostable plastics. The ASTM D6400 standard specifies procedures to certify that compostable plastics will degrade in municipal and industrial aerobic composting facilities.<sup>[66]</sup> The standard establishes the requirements for labeling of materials and products, including packaging made from plastics. The standard determines if plastics and products made from plastics will compost satisfactorily, including biodegrading at a rate comparable to known compostable materials. The standards assure that the degradation of the materials will not contaminate the compost site nor diminish the quality of the compost in the commercial facility resulting from the composting process. ASTM D6400 refers the ASTM D6002 as a guide for assessing the compostability of environmentally degradable plastics in conjunction with ASTM D5338 to determine aerobic biodegradation under controlled composting conditions. ASTM 6400 specifies that a satisfactory rate of biodegradation is the conversion of 60% of the organic carbon in the plastic into carbon dioxide over a time period not greater than 45 days. ASTM D5338 will be used in the research to test the compost-ability of several rigid packaging containers, bags, and cutlery that claim to be made from biodegradable plastics.

Compostable plastics are being used in the United States with the help of a certification program and the establishment of ASTM D6400 standards. BPI and the US Composting Council (USCC) established the Compostable Logo program in the United States.<sup>[67]</sup> The BPI certification demonstrates that biodegradable plastic materials meet the specifications in ASTM D6400 and will biodegrade swiftly and safely during municipal and commercial composting. Several degradable plastics, which are available for composting, are listed for 2002.<sup>[68]</sup> The compostable logo is helpful for consumers to identify which products meet the ASTM D6400 standards.<sup>[69]</sup> Verification of the ASTM standard is accomplished through an independent third-party consultant who is selected by the manufacturer.

In Europe, compostable plastics are being used in several applications. Compostable plastics, must comply with the European Norm EN13432, which is the criteria for compostability. EN13432 requires a compostable plastic material to break down to the extent of at least 90% to H<sub>2</sub>O and CO<sub>2</sub> and biomass within a period of 6 months. ISO14855 standard specifies a testing method to evaluate the ultimate aerobic biodegradability of plastics, based on organic compounds, under controlled composting conditions by measurement of the amount of carbon dioxide evolved and the degree of plastic at the end of test.

The Australian standard for degradable plastics includes test methods that enable validation of biodegradation of degradable plastics. It is a system for certification of degradable polymers that conform to the standard, e.g., EN 13432.<sup>[70]</sup> The standard provides coverage to the range of potential application areas and disposal environments in Australia. The standard is not so severe as to exclude Kraft paper as do some European standards. Kraft paper is excluded as a positive control due to the potential presence of sulfonated pollutants. A more effective positive control can be either cellulose filter paper or microcellulose AVICEL PH101. The standard was developed with reference to the existing international standards. The standard differentiates between biodegradable and other degradable plastics, as does ASTM D6400, and clearly distinguishes between biodegradation and abiotic disintegration even though both degradation systems demonstrate that sufficient disintegration of the plastic has been achieved within the specified testing time. The standard addresses environmental fate and toxicity issues, as does ASTM D5152. Lastly, the Australian standard states that total mineralization is required, where all of the plastic is converted to carbon dioxide, water, inorganic compounds and biomass under aerobic conditions, rather than disintegration into finely indistinguishable fragments and partial mineralization.<sup>[71]</sup>

The heavy metal limits in the European standard are more stringent than those listed in the US standards. Heavy metal concentrations in the EN13432 standard allow a limited amount of metal, i.e., lead (30 mg/kg), cadmium (0.3 mg/kg), chrome (30 mg/kg), copper (22.5 mg/kg), nickel 15 mg/kg, zinc (100 mg/kg), and mercury (0.3 mg/kg). The US standard allows the following amounts: lead (150 mg/kg), cadmium (17 mg/kg), chrome (Not Specified), copper (750 mg/kg), nickel 210 mg/kg, zinc (1400 mg/kg), and mercury (8.5 mg/kg).<sup>[72]</sup> Acceptable levels of heavy metals in sewer sludge are provided per US EPA Subpart 503-13. Testing of five biodegradable garbage bags found the heavy metal content lower than allowable standards. Pigments with green and blue colors cause the amount of copper to increase in soil.<sup>[73]</sup> Pigments of heavy yellow can cause the amount of lead to increase in soil.

## **Testing Plan**

The testing plan in this research is based upon research work in Australia. Dr. Greg Lonergan developed very thorough testing methods at his Swinburn University research facility in Melbourne, Australia. Dr. Lonergan has a state-of-the-art facility that specializes in compost testing of biodegradable materials including biodegradable thermoplastics. Dr. Lonergan has performed extensive biodegradation research in the laboratory and pilot-scale experiments. The laboratory-based method consists of utilizing approximately 24 ice-tea type glass jars that serve as composting vessels with appropriate amounts of compost in the jar. Rubber tubes on the top of each vessel collect the emitting gasses, which are sampled by computer control and measured with a gas sensor that is based upon smoke alarm sensors. Micro-cellulose AVICEL PH101 is used as a positive control. The sensor is calibrated at 10% CO<sub>2</sub> with a gas chromatograph. The temperature, moisture, and soil conditions are closely monitored. The lab scale enables very precise measurements and control of the degradation process. The pilot-scale environment is larger operation than in the lab where the compost is an outside environment. The pilot-scale research is based upon compost bins that are instrumented to measure the temperature and moisture of the compost.

# Experimental Work

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The testing in this research occurs in a laboratory setting, at a pilot scale facility at CSU, Chico and at a commercial compost facility in Chico. The laboratory operation is similar to the one used in Dr. Lonergan's laboratory in Swinburn University, Australia that measures the degradation rate of the compostable plastics according to EN13432 standards. The compost facility at the CSU, Chico University Farm simulates a pilot scale operation. The laboratory and pilot-scale methods provide evaluation of the inherent biodegradation of plastic products in compost environments. The lab data can provide indications of how the polymers will degrade in full-scale operations. The third method to test for degradation is at a full-scale, commercial compost facility, namely, the City of Chico municipal compost facility. The full-scale test can confirm the compostability of the biodegradable plastic materials in a large-scale operation.

The first testing environment is under controlled laboratory settings. The closely monitored environment allows measurement of the degradation rate of the compostable materials as well as control of important laboratory conditions, such as, compost temperature, moisture, and pH. The purpose of the laboratory experiment is to compare the degradation rates of several compostable materials with known compostable standard materials, as well as to assess toxicity of the degradation products from the compostable plastics. The experiment will use ASTM D6400 laboratory protocols, though, the successful materials will not be certified to meet the ASTM D6400 standards since the laboratory is not ASTM certified. The laboratory may be ASTM certification in the future if sufficient funding is acquired.

Biodegradation can be measured at a chemical level by monitoring the conversion of starch in the plastics to carbon dioxide. The compostable plastic materials are exposed to mature compost at a constant temperature and moisture level over a 45-day period. Mature compost of 18-months is used to insure that the degradation is due to the conversion of the compostable plastic and not from degradation of organics in the soil. The inoculum soil, defined as compost material that is comprised of soil and green yard waste, were screened with a sieve of less than 10 mm to remove the large pieces. The test is an optimized simulation of intensive aerobic composting where the biodegradability of the samples is determined under moist conditions.

## **Materials**

The materials are all commercially available plastics that are made from corn, polylactic acid (PLA), or sugar cane. The compostable materials that were added to compost in the laboratory experiment were representative samples of a plate made from sugar cane, a trash bag made from corn, and a clear clamshell container and a cup made from NatureWorks polylactic acid (PLA). The compostable materials are described more fully in Table 2.

**Table 2. Compostable product information for laboratory experiment.**

Compostable Product	Plastic Material Source	Company	Cost	Description
Trash bag: 49.2 L (13-gallon)	Corn starch	Bio-Bag, Eco-Products Inc.	\$4.95 per 12 bags	Eco-Products carries a full line of BioBags that are certified compostable and biodegradable by ASTM standards. Being made from corn and other renewable resources, these bags can completely biodegrade in home compost bins. <sup>[74]</sup>
Cup: 300 ml (10-oz)	Natureworks PLA	Eco-Products Inc.	\$65.60 per 1000 cups	Compostable biodegradable PLA based plastic cups.
Clamshell container	Natureworks PLA	Biodegradable Food Service, LLC	\$56 per 250 count	Clamshell Containers that meet ASTM compost ability standards and are biodegradable and FDA approved for food contact. <sup>[75]</sup>
Plate: 25.4 cm (10 in)	Sugar cane	Stalk Market (China)	\$69.90 per 500 plates	Made from 100% bio-degradable, compostable sugar cane fiber (Bagasse) that is a by product of the sugar refining process. Product is both microwavable and freezer safe. After use, product can be 100% catabolized as compost. <sup>[76]</sup>

The compostable plastics can be used in every day food usage. The garbage bag can be used to capture kitchen waste. The cup and plate can be used during meals to hold liquids and food. The storage container can be used to store foods in the refrigerator.

The compostable products can be tested for end-use performance by exposing them to common household environments, fluids and foods. All of the materials performed very well in the tests. The garbage bag did not leak after holding 200 ml of water for 2 days. The cup performed nearly as well as a polystyrene cup from Solo party pack. The clamshell container performed well at room and cold temperatures, but warped at boiling water temperature and while in the microwave. The PLA clamshell was not designed to withstand microwave temperatures. Other PLA products are available that claim to be appropriate for microwave use. The plate performed very well at cold, room, and even boiling water temperatures. The results are listed in Table 3.

**Table 3. Designed-use analysis for compostable products.**

Object	Company	Thickness, mm	Test	Performance
Trash bag: 49.2 L (13-gallon)	Bio-Bag, Eco-Products Inc.	0.05	Moisture and weights	The trash bag held food and paper waste without leaking for 2 days. The bag held 200 ml of water for 12 hours without leaking. After 20 hours the bag leaked approximately 20 ml. After 30 hours exposure the bag held 10 pound weight without breaking The bag broke with a weight of 15 pounds.
Cup: 300 ml (10-oz)	Eco-Products Inc.	0.18	Fluid testing	The cup held water, apple juice, orange juice and milk without leaking but deformed when boiling water was put in it. It did not leak.
Clamshell container	Biodegradable Food Service, LLC	0.20	Food storage	The food container held water meat, rice, and vegetables with out leaking but deformed when put in the microwave for 55 seconds on high power.
Plate: 25.4 cm (10 in)	Stalk Market (China)	0.53	Food use	The plate held hot pizza without leaking and held meat and vegetables while being heated in a microwave for 55 seconds on high power without leaking. The plate was unaffected by freezer temperatures, but warped slightly after exposure to boiling water.

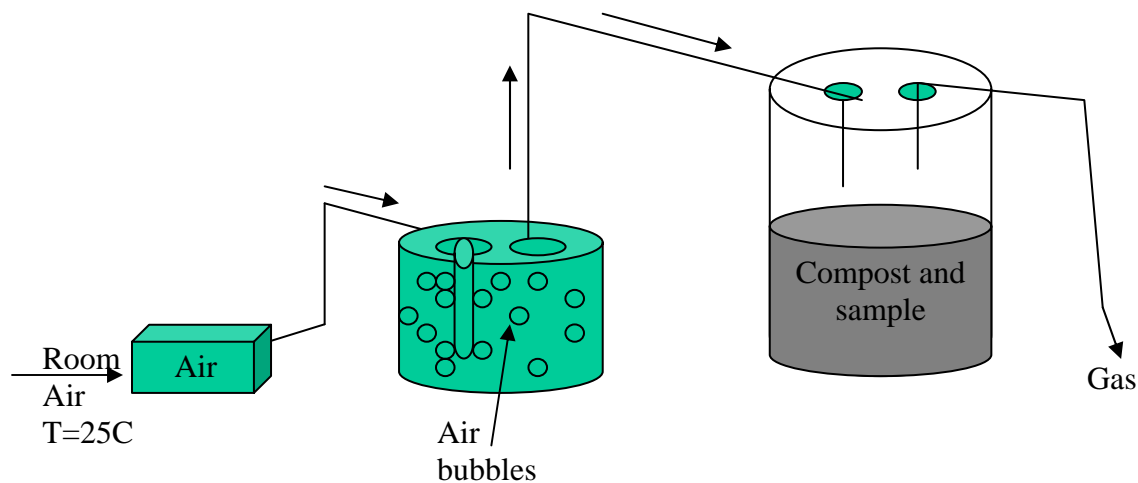
## ***Experimental Methods and Procedures***

The biodegradation of the compostable materials was tested in a controlled experimental environment. The experimental set up for the laboratory experiment is based upon procedures outlined in ASTM D5338. The procedures to measure the gases were done with detectors as allowed in the ASTM standards. Also, moist air was introduced to the top of the container rather than at the bottom. Each of the compostable materials was added to compost soil in a 2-liter glass-canning jar and placed in an oven maintained at 58°C. The room temperature was between 23°C and 25°C during the course of the experiment. The jar containers have a rubber seal on the top. The lid of the jars was modified to add two rubber stoppers with 5 mm tubes for moist air supply and gas withdrawal.

The experimental set-up is described in Figure 1. Moist air was added from a 5-L per minute air-supply pump with 3-mm tubing passing through a 1-L water tank. The moist air passed to a manifold that had 24 tubes that delivered moist air to the top of the jars.

The moisture content of the compost is maintained between 45% and 55%. At regular intervals, 45-ml of gas is withdrawn from the top of the jar with the use of a syringe and placed in measuring container for the carbon dioxide sensor. The sampling tube was 5-mm in diameter and approximately 200-mm long. The sampling schematic is shown in Figure 2. Carbon dioxide is measured at daily intervals. Oxygen was measured as needed to ensure that the content was greater than 6% in the containers. Three replicates of each sample were used in the experiment.

**Figure 1. Experimental set-up for laboratory environment.**



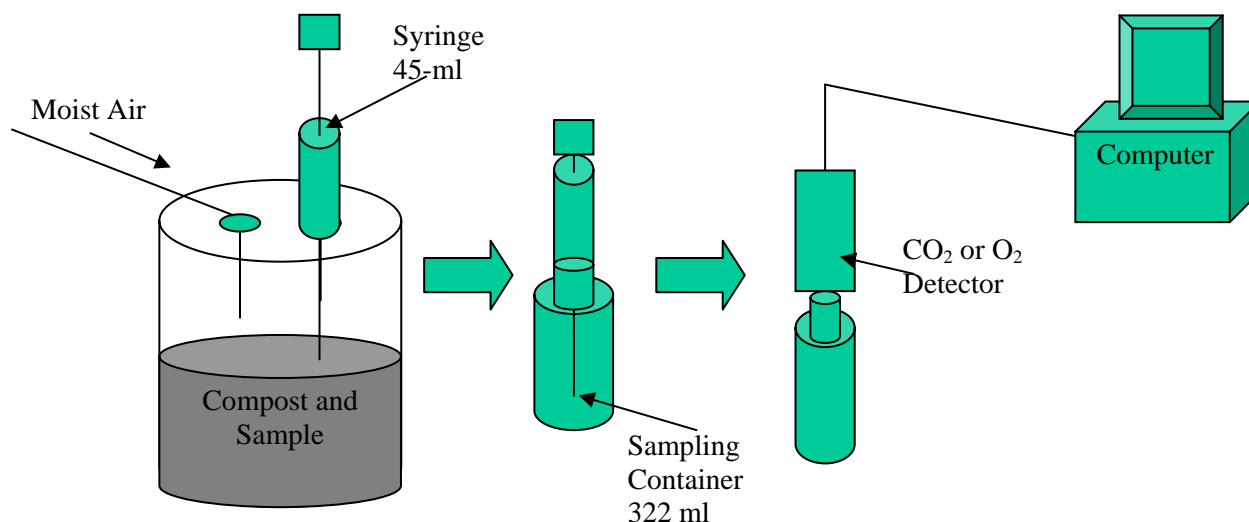
The samples were prepared with mature compost (18-months old) with a pH of 8.7, ash content of 35%, Carbon/Nitrogen (C/N) ratio of 10. The C/N ratio was calculated based upon carbon dioxide and ammonia measurements taken with the Solvita instrument on the compost at the beginning of the test. Solvita is an easy-to-use test that measures both carbon-dioxide ( $\text{CO}_2$ ) and ammonia ( $\text{NH}_3$ ) levels in the soil and also indicates a Maturity Index value. The index is useful for maturity level of the compost soil.<sup>[77]</sup> The inoculum soil was screened with a sieve of less than 10 mm. The dry solids content was 95% and the volatile solids was 63%. The volatile solids percentage is calculated by the ratio of the difference between the dry weight and the ash content divided by the dry weight.

Cellulose filter paper (Cellupure filter) from FilterQueen™ and Kraft paper were used as positive control materials. Polyethylene plastic sheet, called Clingwrap, from Glad was used as a negative control as required in the ASTM standard. The test materials were cut up into approximately 25 mm by 25 mm pieces. Table 3 lists the thickness of the sample materials. The materials are added to a 2-liter vessel and the vessel is weighed at the beginning of the test and then several times throughout the experiment. The 2-liter vessel was filled with 600 grams of compost and 100 grams of compostable sample. The sample materials occupied 1.5 liters of the vessel and left 0.5 liters of open volume for the gas to occupy. ASTM D5338 specifies that a maximum of 75% of the container can be filled with the compostable sample and compost.

The moisture content of the samples is regularly monitored with a digital Sartorius moisture analyzer. Distilled water was added, as needed, to achieve an overall moisture content of 50%. The moisture content is found by drying the sample with infrared heat until the mass is unchanged. The composting vessels were placed in an oven with temperature of  $58^\circ\text{C}$  ( $\pm 2^\circ\text{C}$ ) for 45 days. The temperature of the air in the laboratory was between  $23^\circ\text{C}$  and  $25^\circ\text{C}$  throughout the 45-days.  $\text{CO}_2$  and  $\text{O}_2$  gases were measured with PASCO detectors by removing 40 ml of gases from each vessel and inserting the gases in a known volume container for the detector. The vessels were rotated and shaken weekly to maintain uniformity. The contents were mixed with a plastic utensil if necessary. Moisture content was measured regularly and distilled water was added if needed. Excess liquid was noted on the daily log and removed by adding air. The mass of the sample jars and oxygen was measured at regular intervals. Oxygen levels ranged between 17% and 21% during the experiment, which met the ASTM requirements of greater than 6% in the containers.



**Figure 2. Sampling process schematic.**



## **Laboratory Environment**

Carbon dioxide and oxygen were measured with sensors from Pasco company. The gas sensors measure carbon dioxide or oxygen concentrations in an enclosed 320-ml measurement bottle. The gas sensors use infrared detection to measure the energy absorbed by carbon dioxide or oxygen molecules and then display the appropriate concentration. The carbon dioxide concentration is expressed in parts-per-million (ppm). The CO<sub>2</sub> gas sensor has a range between 0 ppm and 300,000 ppm with accuracy of 100 ppm or 10% of value for range of 0 to 10,000 ppm, whichever is greater. It has 20% of value accuracy for range between 10,000 and 50,000, and qualitative only for values between 50,000 and 300,000. The CO<sub>2</sub> sensor is calibrated with sampling outside air at 400 ppm. The operating temperature range is 20°C to 30°C.

The oxygen sensor measures the percentage of oxygen that is present in the container. The detection error of the sensor is  $\pm 1\%$ . The highest concentration of gas is in the composting jar in the oven. The concentration in the composting jar is out of the range for the detector. The gas from the composting container is withdrawn with the 40-ml sampling syringe and diluted with room-air CO<sub>2</sub> concentrations in the 320-ml measurement bottle. The gas concentration readings then must be converted back to the appropriate concentration from the compost container. Also, ppm concentrations in the composting vessel must be converted into g of CO<sub>2</sub> and then to g of carbon as described in Appendix A.

## **Carbon Dioxide Concentration Results**

During degradation of the compostable plastics CO<sub>2</sub> is produced. The compostable plastic, with an initial 100-gram amount degrades throughout the test. The initial compostable sample, though, has moisture and other elements besides carbon. For instance, cellulose has a chemical structure of C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>, which can result in a maximum of 42% C in the original dry sample. The chemical structures of Kraft paper, corn starch, PLA, and sugar cane are more complex. Kraft paper is made from Kraft pulp, which is 44% cellulose. Corn starch's primary carbon source is native amylase corn starch (C<sub>5</sub>H<sub>8</sub>O<sub>3</sub>)<sub>n</sub>, where *n* is the degree of polymerization. The chemical structure of PLA is (C<sub>3</sub>H<sub>4</sub>O<sub>5</sub>)<sub>n</sub>. Sugar cane's primary carbon source is from sucrose (C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>)<sub>n</sub>. The percentage of carbon in each based solely on the chemical formulas is as follow: Kraft paper is 44% Carbon; starch is 55%; PLA is 30%; sugar cane is 42% Carbon. The amount of carbon can be less than the

theoretical values depending upon the amount of other materials added to the compostable material to enable them to be processed into plastic parts or bags.

The amount of carbon can be directly determined experimentally with calorimetry. A bomb calorimeter is a constant-volume calorimeter made from stainless steel that measures the change in temperature of a known volume of distilled water as a combustible material is ignited. The bomb calorimeter is capable of withstanding the large pressure and force of explosive reactions. A calorimetry bomb (Parr Series 1300 Calorimeter with model 1101 stainless steel oxygen bomb) was used to measure the carbon content of the samples by igniting the sample and measuring the amount of carbon dioxide that is produced with the Pasco detector. The carbon content was calculated based on converting the ppm measurement to mg/m<sup>3</sup> in the sample container with Equation 2 in Appendix A.

The CO<sub>2</sub> gas was vented through the exhaust port at the end of the test and gathered in the 320ml sampling tube. The ppm of CO<sub>2</sub> was measured with the PASCO CO<sub>2</sub> gas detector. The volume of the calorimeter was 0.340 liter. The pressure was 25 atmospheres. The heats of combustion for the materials were also calculated. The plastic samples were also measured for moisture content. The results are provided in Table 4. The trash bag and PLA containers had higher heats of combustion than the cellulose material. The Kraft paper and sugar cane plate had lower heats of combustion than the cellulose material. The cellulose, Kraft paper, and sugar cane samples had approximately 7% moisture content, whereas, the trash bag and PLA samples had 1% or less moisture content. The moisture content is an average of 3 measurements.

## ***Biodegradation Results***

The biodegradation percentage can be determined from the amount of CO<sub>2</sub> measured during the 45-day experiment and the amount of initial carbon present in the sample with the use of Equation 4 in Appendix A. Pictures of the degradation experiment are provided in Appendix B. The CO<sub>2</sub> was measured according to the procedure outlined previously. Different techniques were used to obtain consistent results. The jars were monitored daily for moisture content and compactness of samples. The jars were periodically stirred to mix the contents to reduce the settling effect of soil on the bottom of the jar and compost sample on the top. The most consistent CO<sub>2</sub> gas readings were obtained when the jars were kept closed and not mixed. However, some of the jar contents displayed moisture content less than 45%. Water was added as needed. The measured ppm readings were tested for open jar mixing method versus closed jar method. The open jar mixing method experienced less concentrations of CO<sub>2</sub> than the closed jar method but had better moisture control. Future work can develop a new procedure that is based upon combinations of the two methods. The two methods were calibrated for the different types of compost samples and the results were modified to account for the measurement method. Also, the measured CO<sub>2</sub> ppm readings were less than expected from a control experiment where a known volume (10 ml) of CO<sub>2</sub> gas was added to two jars filled with 1 liter of compost. The average ppm readings were off by a factor of 3. The ppm concentrations were adjusted to account for the measurement error. The results are still valid since the same technique was used for all of the samples.

**Table 4. Heats of combustion, carbon content, and moisture % for compostable samples.**

Material	Heat of Combustion KJ/g	Bomb Calorimetry % Carbon Content	Moisture %
Cellulose	-14.42	16.35	6.09
Kraft paper	-12.62	16.53	7.19
Corn-based BioBag trash bag	-20.25	21.94	1.03
PLA container	-16.31	18.65	0.56
PLA cup	-17.10	17.01	0.37
Sugar cane plate	-13.22	15.11	6.74

The CO<sub>2</sub> concentrations are measured for 4 control materials and 4 compostable plastic samples. The control samples include the compost itself, cellulose, Kraft paper, and polyethylene as a negative control. Two of the compostable samples are made from PLA. The other two plastic samples are made from corn starch and from sugar cane. The amount of CO<sub>2</sub> was measured daily over a 45-day period. The amount of carbon resulted from the CO<sub>2</sub> concentrations is calculated for each day. After 45 days the total amount of biodegradation conversion can be found by adding individual daily results. The total biodegradation results for the 8 samples are listed in Table 5. The compost alone and polyethylene (negative control) produced very little CO<sub>2</sub> which resulted in less than 1% conversion of the polyethylene into carbon, which can be accounted for by experimental error. The degradation rate of the compost and polyethylene samples were approximately 0.1 mg/day. The cellulose and Kraft paper represented positive controls for the experiment. The cellulose degraded 74% over the 45-day experiment and Kraft paper degraded 61%. ASTM D6400 requires at least 70% degradation of cellulose or the test is considered invalid for D-6400 compostability certification. The Kraft paper samples had comparable degradation conversion and degradation rates as the PLA and sugar cane samples. The corn-based trash bag had lower biodegradation conversion and low degradation rates than the cellulose or Kraft paper positive control materials.

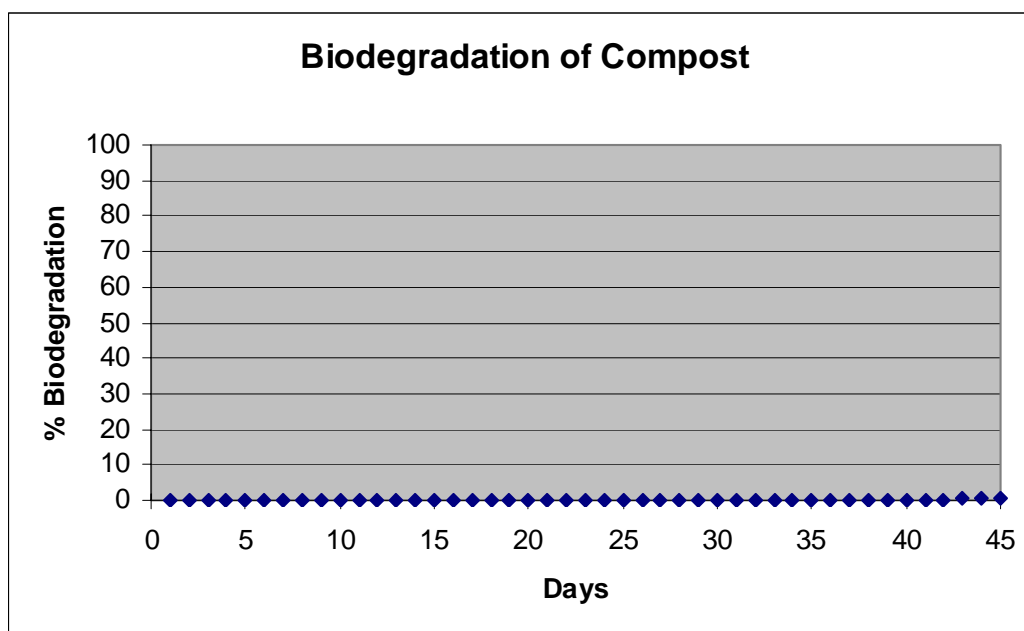
The conversion of the organic materials in each of the eight materials into CO<sub>2</sub> can be represented by graphing the total conversion percentage on a daily basis as depicted in Figures 3 through 10. The results represent an average of 3 samples per material. Figure 3 illustrates the degradation of the compost material alone. This is well within the measurement error in the experiment and is negligible. Figure 4 describes the degradation of the cellulose material. The curve demonstrates degradation throughout the 45-day trial. Figure 5 describes the degradation of Kraft paper. Figure 6 describes the degradation of polyethylene plastic. Figure 7 describes the degradation of compostable trash bag. Figure 8 describes the degradation of the corn PLA container. Figure 9 describes the degradation of the corn PLA cup. Figure 10 describes the degradation of the sugar cane plate. The experiment was interrupted for 5 days during the middle of the test when the PASCO sensor broke. A Vernier carbon dioxide sensor, which operates on the same infrared

detection principle, was used as a replacement for the PASCO sensor until a new one was delivered. The data was interpolated during the 5 lost days and on weekend days.

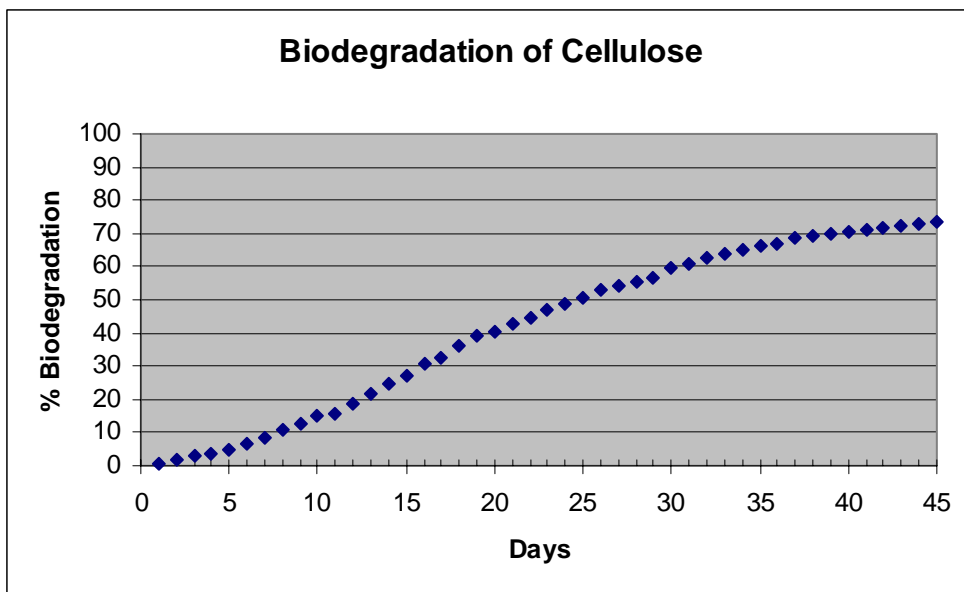
**Table 5. Degradation rates for compostable samples.**

Material	Biodegradation Conversion %	Degradation rate mg/day
Cellulose positive control	73.66	16.4
Sugar cane plate	63.48	14.1
Kraft paper positive control	61.28	13.6
PLA container	62.77	13.9
PLA cup	61.01	13.6
Corn-based Biobag trash bag	60.10	13.4
Polyethylene negative control	0.58	0.1
Compost	0.33	0.1

**Figure 3. Carbon conversion percentage for compost control alone.**



**Figure 4. Carbon conversion percentage for cellulose control.**



**Figure 5. Carbon conversion percentage for Kraft paper control.**

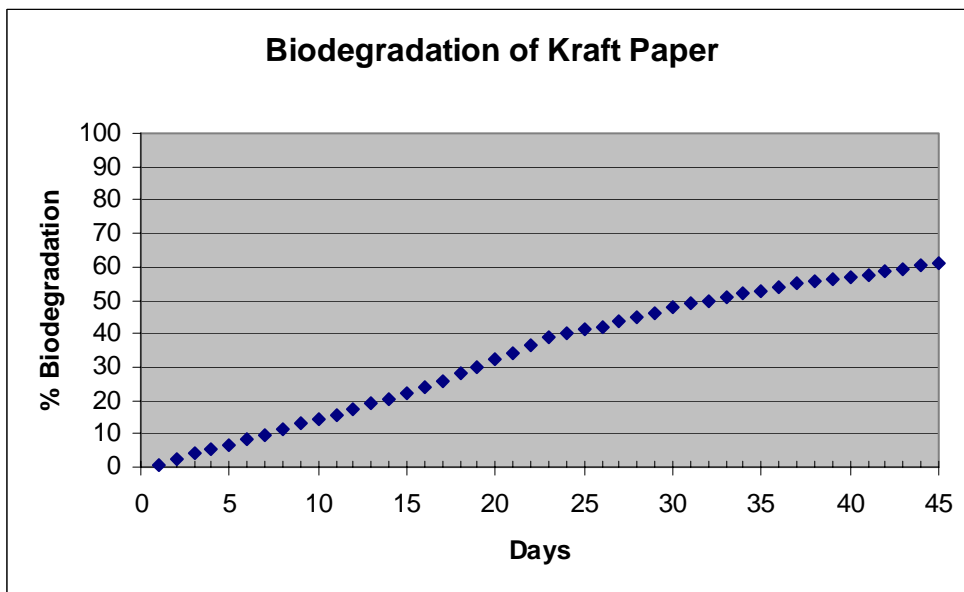


Figure 6. Carbon conversion percentage for polyethylene negative control.

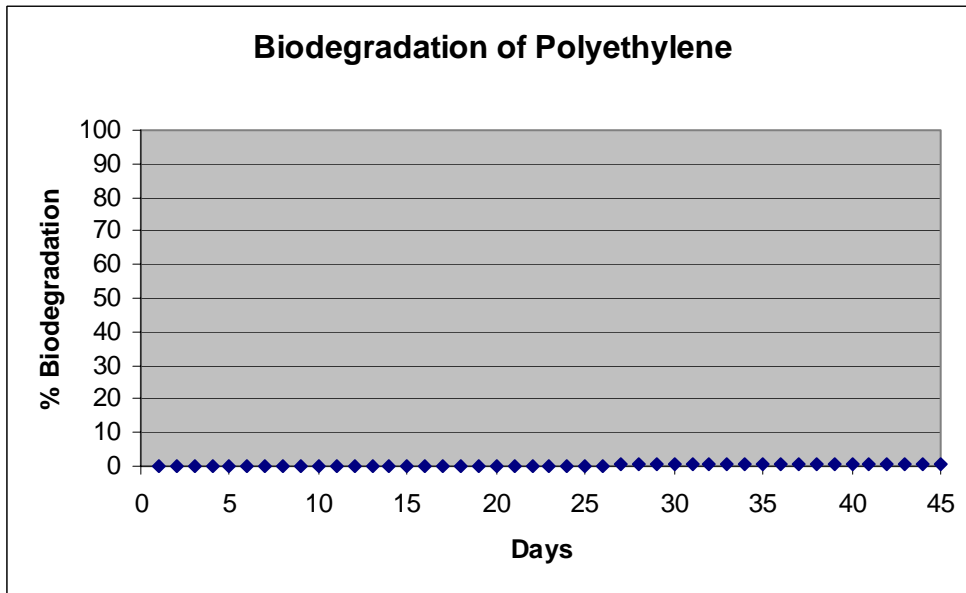


Figure 7. Carbon conversion percentage for corn based BioBag trash bag.

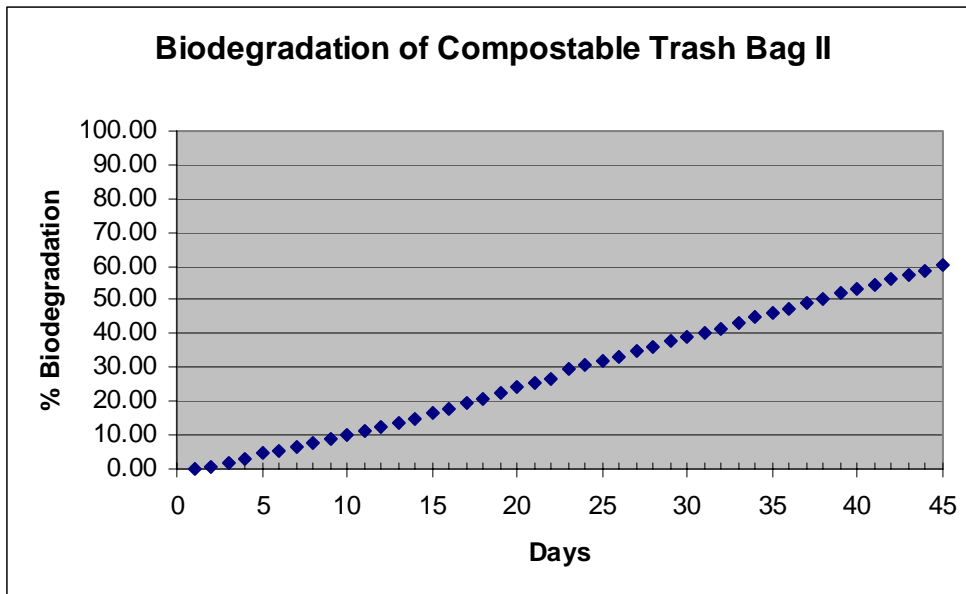


Figure 8. Carbon conversion percentage for corn PLA clamshell container.

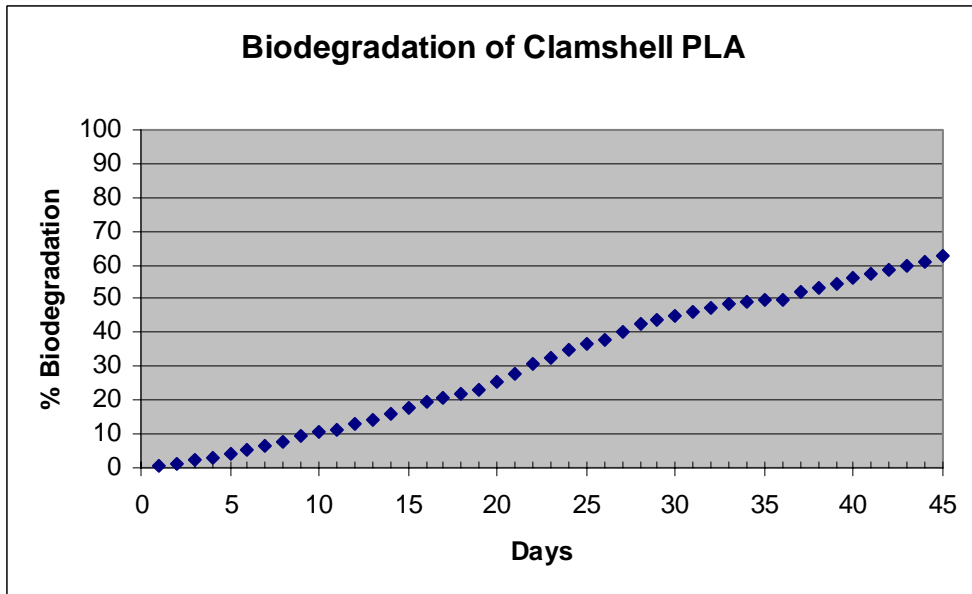
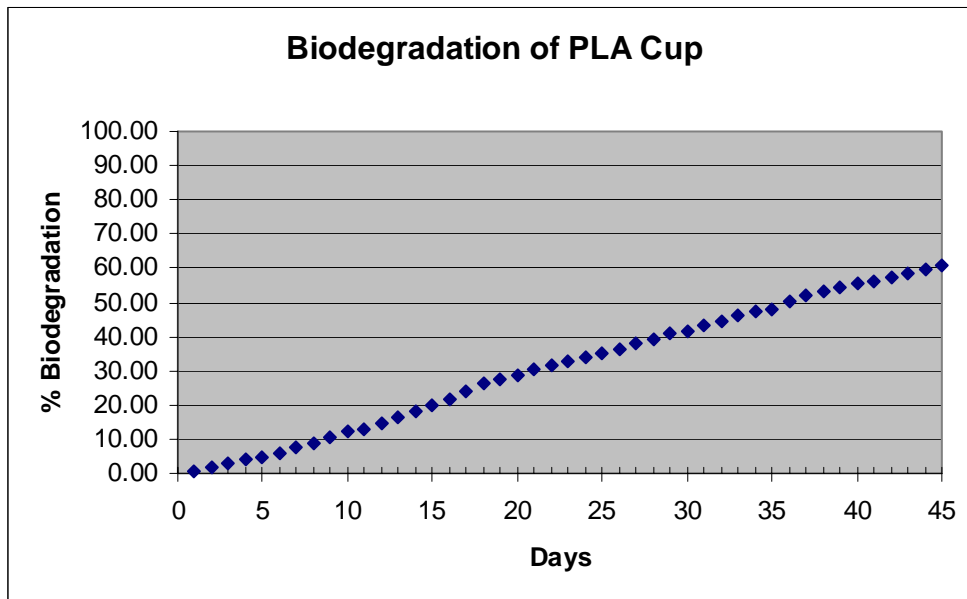
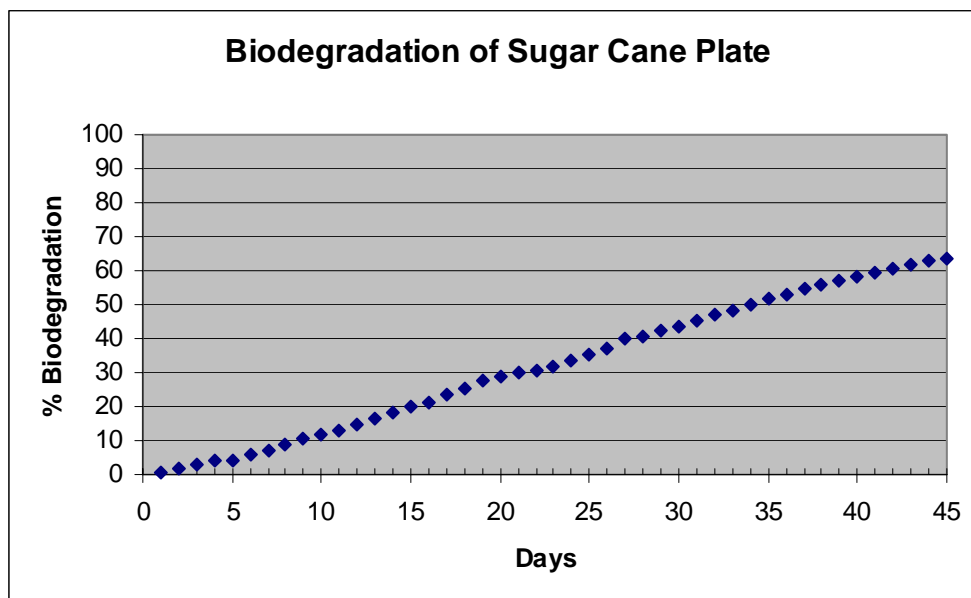


Figure 9. Carbon conversion percentage for corn PLA cup.



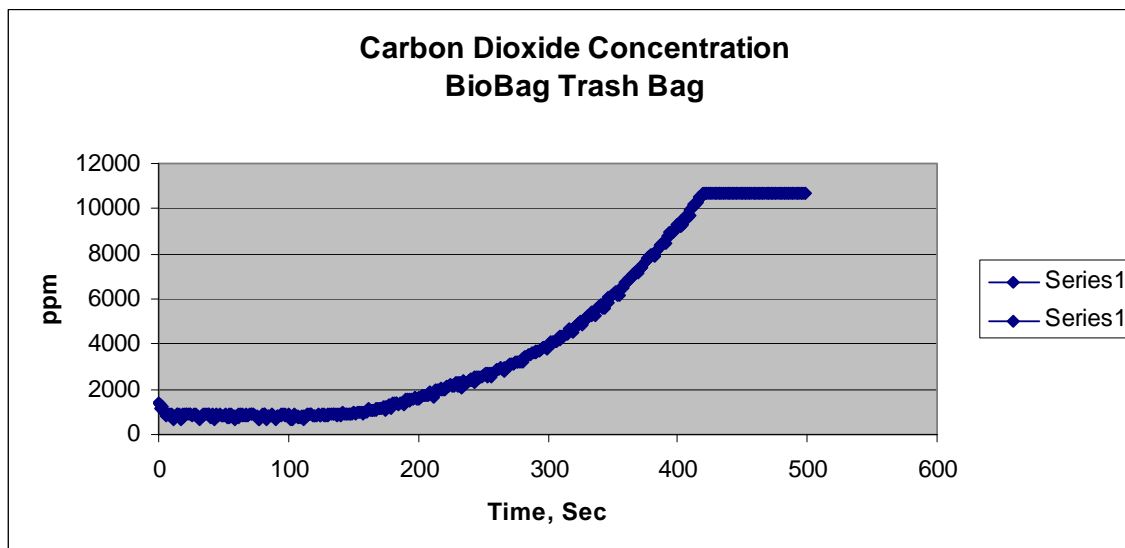
**Figure 10. Carbon conversion percentage for sugar cane plate.**



The trash bag degraded during the test and met the compostability standards specified by ASTM. The trash bag was retested with improved test methods at a later date and was found to have degradation similar to the Kraft paper control over the 45-day test period. The new test procedures have better moisture control and automated CO<sub>2</sub> measurement. In the new procedures, 100 g of trash bag samples were added to 600 grams of mature soil compost in a 3.7 L glass jar. As in the previous test, the moisture content is 50% and the temperature is held at 50°C for 45-days. The test method results for 33 test samples will be described in more detail in future reports after the research project is completed in 2007. Preliminary results are provided in this report to establish the biodegradation capability of the biodegradable plastic bag. In the new experiment, the jars are fed with moist air as the biogas is withdrawn with the aid of a vacuum pump. The test apparatus can test 42 jars in series and is computer controlled with LabView data acquisition system. The CO<sub>2</sub> is measured with Pasco IR detectors, as previously described, and the CO<sub>2</sub> concentration output is saved in a computer file for each sample jar. The Biobag biodegradable trash bag was tested with Kraft paper control and blank compost control. The materials were tested in triplicate. Figure 11 depicts the CO<sub>2</sub> concentration versus time for one biodegradable trash bag sample after 3 weeks. The figure illustrates a delay period when the biogas is being pulled from the sample jar followed by a steady increase of CO<sub>2</sub> concentration as the biogas is pulled through the detector. The slope of the ppm-time curve is the rate of carbon dioxide added to the detection jar during the experiment. The rate also indicates the concentration of carbon dioxide in the sample jar as well as the biodegradation of the test samples. Table 6 lists the CO<sub>2</sub> rate for Kraft paper and biodegradable trash bag over the first 21 days of the experiment. The tables shows that the biodegradable trash bag exhibits on average 85% of the concentration of CO<sub>2</sub> as Kraft paper. Thus, the biodegradation rate of the biodegradable trash bag is similar to the biodegradation of Kraft paper for the first 21 days. Biodegradation results of the biodegradable trash bag are shown in Figure 7 to meet the ASTM D-6400 standards of 60% biodegradation.



Figure 11. CO<sub>2</sub> ppm concentration of BioBag trash bag after 21 days.



### ***Phytotoxicity Testing***

The compostable materials must not release toxic materials into the compost soil after degrading. The compost soil can be tested to assess phytotoxicity, which indicates poisonous environment to plants. The germination of tomato seedlings in the compost soil was evaluated after a 10-day duration. The phytotoxicity test was based upon the ISO 11269 standard. The tomato seeds are a “Tiny Tim” variety from Vaughans Seed Company. The tomato variety is one that is used in the Biology classes on campus and is known to grow quickly and is robust. The tomato seed is of a 1994 variety. 10 to 12 seeds were planted in small beverage cups (280 ml) that were filled with approximately 50 grams of compost from each of the 24-samples.

The sample containers were watered frequently while in a greenhouse. The green house was warm and moist with a temperature of 25°C and relative humidity of 80%. After 10-days in the green house with ambient light, the number and length of shoots were recorded for each sample. The lack of emerging seedlings would indicate phytotoxicity. The percentage of seeds that germinated and the average length of the seedlings are listed in Table 7. Ten seeds were placed in each container. A germination index is determined by taking the product of percent germination and the average length and dividing by 100.

All of the samples had seedlings grow. The sugar cane materials were tested a second time several months after the first test and exhibited more consistent seedling growth. The sugar cane was tested after the 45-day biodegradation test prescribed in ASTM D-6400. The degraded sugar cane and compost were evaluated with cucumber seeds at 25°C, 80% relative humidity, and atmospheric pressure in the greenhouse. The seedlings exhibited growth after a few days and the results are listed from the 4-days time period.

### ***Heavy Metal Testing***

The degraded materials should not leave any heavy metals in the compost soil after degradation. The compost soil was tested for lead and cadmium. The acceptable limit is 30 mg/kg for lead and 0.3 mg/kg for cadmium. The compost soil for each sample was put into solution and the heavy

metal in the compost soil was measured with Fisherbrand<sup>[78]</sup> hollow cathode single-element 2 inch diameter lamps with elements for lead and cadmium. The results for cadmium were delayed because of a 7-week back-order on the lamp.

Lead and cadmium were measured by flame atomic absorption spectrometry using a Jarrell-Ash Model. Lead and cadmium absorption was measured at 283.3 nm and 228.8 nm respectively. The background correction was measured at 281.2 nm for Lead and at 226.5 nm for cadmium. The detection limits are 0.02 ppm lead and 0.005 ppm cadmium in the analytical solution. For a 1-g sample the detection limits are 0.2 ppm Pb and 0.05 ppm Cd.

The soil samples that were used during the phytotoxicity testing were also used to measure the lead and cadmium levels. Approximately 10 g of compost soil from each sample was dried for 24 hours at 105 °C. The average moisture loss was about 30%. About 3 g of each sample was weighed into a 150 mL beaker to which 50 mL of 8 M HNO<sub>3</sub> was added. The samples were digested for 4 hours at 85 °C with occasional stirring. After 4 hours, 50 mL of deionized water was added to each sample followed by vacuum filtration through a Whatman GF/A glass filter with 1% (v/v) HNO<sub>3</sub>. The filtrate was quantitatively transferred to a 250-mL volumetric flask and filled to the mark with 1% (v/v) HNO<sub>3</sub>. The resulting samples all had a relatively intense orange-red appearance.

Sample preparation included adding a 0.8239 g sample of Pb(NO<sub>3</sub>)<sub>2</sub> to a 500-mL volumetric flask, dissolved and diluted to the mark with 1% (v/v) HNO<sub>3</sub> yielding a 1099.5 ppm Pb<sup>2+</sup> solution. Various standard solutions in the range of 0.220 to 1.10 ppm Pb<sup>2+</sup> in 1% (v/v) HNO<sub>3</sub> were prepared along with a 1 M HNO<sub>3</sub> solution.

Standard solutions were prepared by dissolving 0.2460g Cd in approximately 3mL of 6M HCl and approximately 2 mL of 8M HNO<sub>3</sub> in a 250 mL volumetric flask and diluted to the mark with 1% HCl (v/v) yield on 984 ppm Cd solution. Various standard solutions including a blank from mature compost alone were prepared from 0.0984ppm to 9.840 ppm Cd in 1% HCl.

## Results

The standard solutions and eight sample solutions were analyzed using a ThermoElectron S Series Flame Atomic Absorption Spectrophotometer using an air-acetylene flame and equipped with a Pb hollow-cathode lamp detecting at 283.3 nm and a Cd hollow-cathode lamp. The sample solutions gave absorbances at or very near the lowest standard employed which was just above the detection limit of the instrument. Using 0.220 ppm Pb<sup>2+</sup> as the detection limit leads to an upper limit of 20 ppm Pb<sup>2+</sup> in the original soil samples. The 20 ppm value equates to 0.02 mg/kg for Pb. The Cd concentrations were lower than 1ppm which equates to 0.001 mg/kg Cd. All of the soil samples from the compostable materials had lead concentrations much lower than the limit of 30 mg/kg Pb and Cd concentrations lower than the limit of 17 mg/kg Cd. In fact, the measured values for Pb and Cd were at the lower detection limits of the Pb and Cd detectors.

**Table 6. Phytotoxicity of Compost Soil.**

<b>Sample</b>	<b>Material</b>	<b>Percentage of Germination from Seeds</b>	<b>Average Length, mm after 10-days</b>	<b>Germination Index</b>	<b>pH</b>	<b>Average, pH</b>
1	Compost	70	25	17.5	8.7	8.73
2	Compost	50	6	3	8.5	
3	Compost	30	6	1.8	9	
4	Cellulose	70	6	4.2	8.7	8.70
5	Cellulose	20	12	2.4	8.7	
6	Cellulose	50	6	3	8.7	
7	Kraft Paper	40	12	4.8	9.2	8.93
8	Kraft Paper	10	6	0.6	8.9	
9	Kraft Paper	40	9	3.6	8.7	
10	Polyethylene	40	12	4.8	8.7	8.60
11	Polyethylene	10	12	1.2	8.5	
12	Polyethylene	20	12	2.4	8.6	
13	Trash Bag	40	12	4.8	8.7	8.93
14	Trash Bag	10	12	1.2	9	
15	Trash Bag	60	12	7.2	9.1	
16	PLA Container	30	12	3.6	8.9	8.80
17	PLA Container	30	6	1.8	8.8	
18	PLA Container	20	6	1.2	8.7	
19	Sugar Cane**	30	25	7.5	8.7	8.7
20	Sugar Cane**	40	25	10	8.8	
21	Sugar Cane**	60	25	15	8.6	
22	PLA Cup	10	3	0.3	9.1	8.97
23	PLA Cup	40	12	4.8	8.9	
24	PLA Cup	30	6	1.8	8.9	

\*\* Retested after 45-day degradation testing in March 2007.

# University Farm Compost Facility

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The second environment for the compostable materials is similar to a commercial production operation. The university farm uses cow manure and straw to create a compost material that is sold commercially. The university farm environment represents a commercial compost facility with very active manure-based compost that should provide a high degree of enzyme activity and nutrients for the compostable materials to degrade.

## **Materials**

Several compostable products were buried in compost at the CSU, University Farm. They include a tray made from potato starch, a trash bag made from corn starch, and a straw, fork, cup, plate and clear clamshell container made from PLA. Several materials were used as positive controls at the university farm. The materials are described more fully in Table 8.

The materials are all commercially available plastic products that are made from potato, corn and polylactic acid (PLA). The compostable materials that were added to compost in the laboratory experiment were representative samples of a plate made from sugar cane, a trash bag made from corn, and a clear clamshell container and a cup made from NatureWorks polylactic acid (PLA). The positive controls include cellulose filter paper from Filter Queen Vacuum and Avicell pH101 Fluka microcellulose powder, which is the European and Australian control standard. No ASTM standards exist for compost testing at commercial facilities. The compost soil is typically fresh and has a lot of background carbon dioxide in the soil from degrading organic materials. Unfortunately, the background carbon dioxide can mask the degradation of the sample materials. However, degradation can be indicated by disintegration of the compostable material over the duration of the experiment. A negative control, e.g., a polyethylene bag, was not used in the university compost experiment. It is well known that polyethylene bags are unaffected by soil and does not degrade in the soil over a three-month time frame. Polyethylene bags are thought to degrade after 100 years of soil exposure.

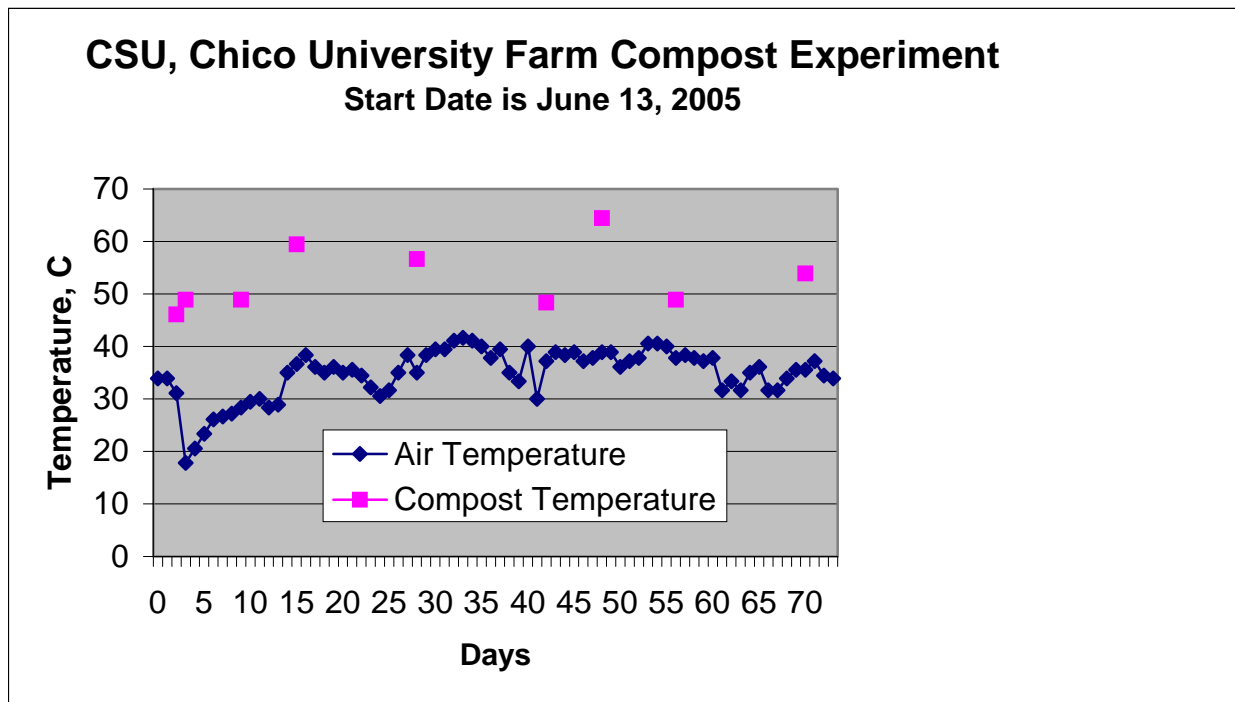
**Table 7. Compostable Product Information for University Farm Experiment**

Compostable Sample	Organic Source	Company
Avicell pH101 control	Microcellulose	Fluka
Filter paper control	Cellulose	Filter Queen Vacuum cleaner
Tray	Potato-based starch	Plantic Australia
Trash bag: 49.2 L (13-gallon)	Corn-starch based	Biobag, Eco-Products Inc.
Cup: 300 ml (10-oz)	Natureworks PLA	Eco-Products Inc.
Straw	Natureworks PLA	Eco-Products Inc.
Fork	Natureworks PLA	Eco-Products Inc.
Plate: 25.4 cm (10 in)	PLA from China	Wei Mon Industry, Co. Chinese Company
Clamshell container	Natureworks PLA	Biodegradable Food Service, LLC

### ***Experimental Set-up***

The finished plastic products and compost were placed in a perforated plastic agricultural bag and placed in the compost mound. The temperature and moisture of the compost in the bag were measured and the ambient temperature and weather conditions were recorded. The compost mounds were turned several times a week to mix the compost. The plastic sample bags were removed from the compost before the turning operation and then were placed back in the compost after the turning. Samples of the compost were tested at regular intervals as well as the temperature and compost maturity index at the compost sites. The compost maturity index can be defined as compost that is resistant to further decomposition and free of compounds, such as ammonia and organic acids, that can be poisonous to plant growth. Initially, CO<sub>2</sub> was measured with Dräger tubes. After several weeks, though, the Dräger tubes were replaced with the Solvita test kit. However, measurement of CO<sub>2</sub> did not prove to be a reliable indicator of degradation for the compostable samples since the level of CO<sub>2</sub> in the compostable samples appeared very similar to the levels of CO<sub>2</sub> in the background compost with no samples. Ultimately, the level of degradation of the plastic samples was measured directly by the disintegration of the plastic over time. The mass of the plastic samples was recorded at regular intervals.

**Figure 12. Temperature of the air and compost during the duration of the university farm experiment.**



## Procedure

The compost pile was located on a concrete pad. The procedure for testing the compost at the university farm involved placing the compostable sample in a perforated polyethylene agricultural bag with approximately 1-kg of compost soil. The contents were mixed well. Water was added to the compost mound at regular intervals to keep the compost moist. The compost mound was rotated and mixed every two or three days. The bags were placed in holes in the compost mound approximately 500 mm below the surface and mark location with flags. Moisture content, temperature, and CO<sub>2</sub> and ammonia of compost in bag were measured at regular intervals. The bags were removed from compost mound before a mechanical turning machine mixed the compost.

The University Farm at California State University Chico produces 250-tons of compost from dairy manure and rice straw annually using conventional windrow methods. The nutrient composition, or NPK, is 1.2 parts Nitrogen to 0.5 parts Phosphorous to 1.5 parts Potassium. The organic matter content is approximately 30% and the pH is 8. The fecal coli forms is 0 counts, the E. coli is 0 counts, and Salmonella is 0 counts. The heavy metals content of the compost was negative for Arsenic, Lead and Mercury. <sup>[79]</sup>

The compostable plastics were buried on June 13, 2005. All of the materials were fully degraded after 7 weeks or by August 22, 2005. The moisture content of the compost was approximately 55% over the duration of the experiment. The temperature of the outside air ranged from 18°C to 42°C. The temperature of the compost ranged from 48°C to 64°C during the duration of the experiment. The temperature graphs are provided in Figure 12. Pictures of the compostable samples during the experiment at the university farm are provided in Appendix C.

## **University Farm Degradation Results**

Material degradation, which can be measured by weight loss, is defined as the breakdown of the material into smaller fragments. Biodegradation is chemical decomposition by biological agents, especially bacteria, of the organic nature of the material into carbon dioxide, water and biomass. Degradation is decomposition of a compound by stages, exhibiting well-defined intermediate products. Disintegration, which can be measured by sieving, occurs when materials become reduced to components, fragments, or particles. Mineralization develops or hastens mineral formation. The breakdown of degradable plastics can be categorized into disintegration and mineralization.<sup>[80]</sup>

As the compostable plastic degrades the carbon that is present in the organic material is converted by microorganisms in the compost soil into CO<sub>2</sub> gas. The degradation of the compostable plastic can be quantified by measuring the evolved carbon dioxide from the sample bags. Carbon dioxide can be measured with a Dräger tube, which is commonly used by industrial hygienists to measure carbon dioxide. A Dräger tube was inserted in the bag to measure the carbon dioxide. Dräger tubes can be used to detect carbon dioxide in the soil. The principle of operation of Dräger tubes is based on a chemical reagent system that is housed in a closed glass tube and reacts by changing color when brought into contact with a gas or vapor. The concentration of the substance is characterized by the length of discoloration. The concentration can be read off directly from a scale printed on the glass tube.<sup>[81]</sup> The Dräger tubes were used to measure the carbon dioxide for the first 29 days.

The concentrations of the carbon dioxide was very low with the tubes and the procedure was replaced with a more appropriate system from Solvita. Solvita is based on a novel, patented gel-colorimetry technology in which respiration gases from composts are captured and indicated in a color-coded system. The color-coded indicators are compared to table values for carbon dioxide and ammonia levels.<sup>[82]</sup> The Solvita method was used from the 29<sup>th</sup> day to the 72<sup>nd</sup> day at the end of the experiment.

The sample materials were actual compostable materials that were buried in the original shape and condition as purchased. The samples were placed with an appropriate amount of compost in the bag and then buried in the compost mound. On selected days, the Dräger CO<sub>2</sub> detector tube was placed in the bag after it was removed from the compost mound and allowed to detect the CO<sub>2</sub> gas for approximately 30 seconds. The carbon dioxide measurements of the samples over the experiment time frame is provided in Table 9. All of the compostable materials degraded over time and all were consumed in 72 days. The CO<sub>2</sub> measurement results were mixed and provided very little quantitative information. The background amount of carbon dioxide from the compost was in many cases the same level of carbon dioxide as the degrading compostable plastic.

**Table 8. Carbon dioxide percentages from compostable materials at the university farm**

Days	Hole 1	Hole 2	Hole 3	Hole 4	Hole 5	Hole 6	Hole 7	Hole 8	Hole 9	Hole 10
	Compost	Cellulose Control	Tray	Plate PLA	Straw PLA	Fork PLA	Cup PLA	BioBag Trash	Container PLA	Avicel Control
	Initial 500 g	Initial 1.1 g (1mm)	Initial 5.6 g (3mm)	Initial 19.3 g (0.53mm)	Initial 0.9 g (1mm)	Initial 4.2 g (1mm)	Initial 14 g (0.18mm)	Initial 19.2 g (0.05mm)	Initial 23.7 g (0.20mm)	Initial 250 g (powder)
1 Drager	0.5	1	0.5	0.5	1.5	0.5	0.5	0.5		
7	0.5	1	1	0.5	0.5	0.5	0.5	0.5		
15	0.5	0	1	1	0.5	0.5	1	0.5		
28	0.5	0	0	0	0	0	0.5	0		
29 Solvita	0	1.5	1.5	1.5	1.5	1.5	2	1.5	Start	Start
42	0	0	0	0	0	0	0	0	0.5	0
56	0	0	0	0	0	0	0	0	0	0
70	0	0	0	0	0	0	0	0	0	0

Disintegration occurs when the plastic materials fall apart, but the polymer still maintains a finite chain length. Microorganisms can degrade the polymers when the polymer chain is broken down to very small molecular units. Mineralization occurs when the polymer chains are metabolized by micro-organisms after the initial oxidation process to carbon dioxide, water, and biomass. The degradation of the samples can also be found by measuring the mass of the sample over time as it degrades. The mass measurement over time is listed in Table 9.

Removing the bag from the compost mound and then separating the compost from the sample materials with the use of a 2-mm screen if necessary, shaking off the compost, and then weighing the sample measured the disintegration of the materials. Use of the sieve is necessary to assess the quality of compost. The Avicel cellulose control disintegrated quickly in 25 days. The PLA container disintegrated after 38 days. The cellulose control material was fully disintegrated after 59 days as was the potato starch tray, the corn starch trash bag, and the PLA plate and straw. The PLA cup and fork disintegrated after 72 days.



**Table 9. Disintegration results for compostable plastics at the university farm.**

		Initial				
		14-Jun Start	12-Aug	59 days	25-Aug	72 days
Item	Hole No	Mass, g	Mass, g	% Disintegration	Mass, g	% Disintegration
Cellulose Control	2	1.1	0	100	0	100
Tray-starch	3	5.6	0	100	0	100
Plate-PLA	4	19.3	0	100	0	100
Straw-PLA	5	0.9	0	100	0	100
Biobag-starch	8	19.2	0	100	0	100
Avicel* Control	10	250	0	100**	0	100***
Container*-PLA	9	23.7	15.4	35**	0	100***
Cup-PLA	7	14	7.5	46	0	100
Fork-PLA	6	4.2	1.4	67	0	100
*Start date = July 18						
** 25 days of disintegration						
*** 38 days of disintegration						

# City of Chico Municipal Compost Facility

The third environment for the compostable materials is a commercial production composting operation. The city of Chico municipal compost facility is located on a 10-acre site that produces 500,000 cubic yards of compost each year via aerobic windrow compost. The compost is mixed with a large machine called a windrow turner. The turning machine straddles a windrow of approximately eight feet high by 13 feet across. Turners drive through the windrow at a slow rate of forward movement. A steel drum with paddles turns the compost rapidly. As the turner moves through the windrow, fresh air (oxygen) is injected into the compost by the drum/paddle assembly and waste gases produced by harmful bacteria are removed. The oxygen feeds the beneficial composting bacteria and thus speeds the eventual composting process. This process is then extended by windrow dynamics.<sup>[83]</sup> The facility accepts green yard waste, which includes lawn clippings, leaves, wood, sticks, weeds, and pruning. Testing in commercial compost facilities allows the compostable plastics to be exposed to active compost that should have a high degree of enzyme activity and high temperatures that mimic typical composting conditions in a traditional compost facility.

## Materials

The commercially available compostable materials that were buried in the compost facility were a plate made from sugar cane, a trash bag made from corn starch, as well as, a knife, cup, and a clear clamshell container made from PLA. As in the university farm experiment, no negative control was used. The materials are described more fully in Table 10.

**Table 10. Compostable product information for City of Chico compost experiment.**

Compostable Sample	Organic Source	Company
Avicell pH101 control	Microcellulose	Fluka
Kraft paper control	Cellulose	Office Max Company
Trash bag: 49.2 L (13-gallon)	Corn-starch based	Biobag, Eco-Products Inc.
Plate: 25.4 cm (10 in)	Sugar cane	Stalk Market (China)
Cup: 300 ml (10-oz)	Natureworks™ PLA	Eco-Products Inc.
Knife	Natureworks™ PLA	Eco-Products Inc.
Clamshell container	Natureworks™ PLA	Biodegradable Food Service, LLC

## ***Experimental Set-up***

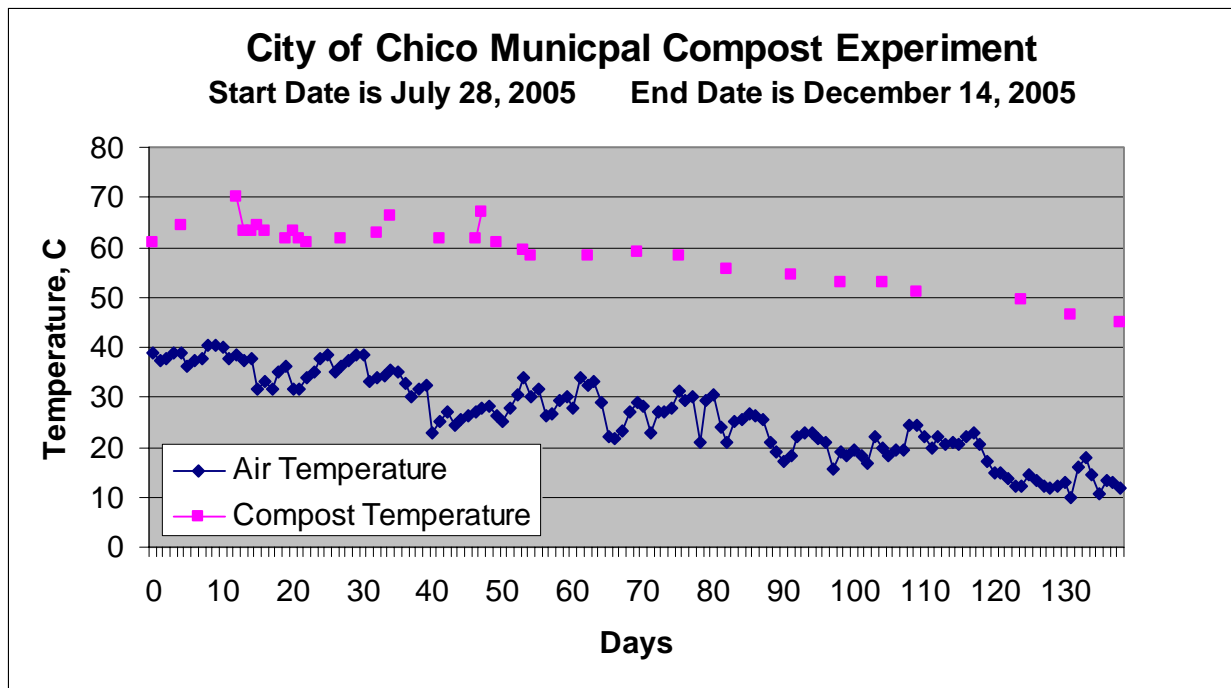
As in the university farm experiment, the finished plastic products and compost were placed in a perforated plastic agricultural bag and buried in the compost mound. The temperature and moisture of the compost in the bag were measured and the ambient temperature and weather conditions were recorded. The compost mounds were turned several times a week to mix the compost. The plastic sample bags were removed from the compost before the turning operation and then were placed back in the compost after the turning. Samples of the compost were tested at regular intervals as well as the temperature and compost maturity index at the compost sites with the Solvita test kit. As in the university farm experiment, the measurement of CO<sub>2</sub> did not prove to be a reliable indicator of degradation for the compostable samples since the level of CO<sub>2</sub> in the compostable samples appeared very similar to the levels of CO<sub>2</sub> in the background compost with no samples. Ultimately, the level of degradation of the plastic samples were measured directly by the disintegration of the plastic over time. The mass of the plastic samples was recorded at regular intervals.

## ***Procedure***

The compost pile was located in a dirt field. The procedure for testing the compost at the university farm involved placing the compostable sample in a perforated polyethylene agricultural bag with approximately 1 Kg of compost soil. The contents were mixed well. Water was added to the compost mound at regular intervals to keep the compost moist. The compost facility had excellent drainage which resulted in drier compost conditions compared to the university farm and laboratory experiment. The compost mound was rotated and mixed regularly. The days that the compost was watered and turned were recorded. The bags were placed in holes in the compost mound approximately 500 mm below the surface and mark location with flags. Moisture content, temperature, and CO<sub>2</sub> and ammonia of compost in bag were measured at regular intervals. The bags were removed from compost mound before a mechanical turning machine rotated the mound.

The compostable samples were added to a fresh compost mound that was created 1 week before the experiment started. The compostable plastics were buried on July 28, 2005. The experiment ended on December 14, 2005. The moisture content of the compost varied between 16% and 40% over the duration of the experiment. The temperature of the outside air ranged from 10°C to 42°C. The temperature of the compost ranged from 45°C to 71°C during the duration of the experiment. The temperature graphs are provided in Figure 13. Pictures of the compostable samples during the experiment at the university farm are provided in Appendix D.

**Figure 13. Temperature of the air and compost at the City of Chico Compost Facility.**



The carbon dioxide was measured with the Solvita method as described before with the use of tabled values. The results are listed in Table 12. As in the university farm experiment, the results are mixed with little qualitative information. The method was discontinued after 48 days.

**Table 11. Carbon dioxide percentage of compostable materials at the Chico Municipal Compost Facility.**

Day	Hole 1	Hole 2	Hole 3	Hole 4	Hole 5	Hole 6	Hole 7
Number	Avicel	Bag	Knife	Container	Plate	Cup	Kraft Paper
12	0.6	0.6	0.6	0.6	1.5	0.6	0.6
33	0.5	0	0	0	0	0	0.5
48	0	0.1	0.1	0.1	0.1	0.6	0.1

## City of Chico Compost Facility Degradation Results

Disintegration can be measured by measuring the mass of the sample over time as it degrades. As with the university farm experiment, the bags were removed from the compost mound and the contents were screened with a 2-mm sieve to separate the compostable sample from the compost. The samples were shaken to removed the dirt and then collected and weighed. The disintegration results indicate degradation, not mineralization, of the compostable materials. The results are listed in Table 12.

The degradation of the compostable samples varied between compostable materials. Some of the materials were fully degraded in 7 weeks, including the Avicel microcellulose control, and the PLA knife, PLA cup, and PLA clamshell container. Thus, the PLA materials had disintegration rates comparable to the cellulose control material. The Kraft paper control had similar disintegration rates as the corn-starch based trash bag and the sugar cane plate. The three materials degraded 88%, 84%, and 78%, respectfully, after 20-weeks.

**Table 12. Material Degradation Results for Compostable Samples at the Municipal Compost Facility.**

		Initial 28-Jul	2 weeks	7 weeks	12 weeks	14 weeks	20 weeks
Item	Hole No	Mass, g	% Degradation	% Degradation	% Degradation	% Degradation	% Degradation
Avicel cellulose control	1	28.3	29	100	100	100	100
Cup- PLA	6	13.983	28	100	100	100	100
Knife- PLA	3	3.876	48	100	100	100	100
Container- PLA	4	22.642	12	100	100	100	100
Kraft Paper Control	7	20.9	28	52	69	73.40	88
Trash bag- corn starch	2	18.863	20	31	65	70.79	84
Plate- Sugar Cane	5	23.418	15	19	37	41.88	78

# Conclusions

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The biodegradation results in the laboratory environment demonstrate that all of the compostable materials degrade under compostable conditions as defined in the ASTM D6400 standards. The cellulose positive control met requirement of 70% degradation after 45-days. ASTM specifies that the test results are valid if the cellulose control degrades 70% or more. The degradation rates of the materials are listed according to highest rates as follows, cellulose control, sugar cane plate, Kraft paper control, PLA container, PLA cup, and corn-starch based trash bag. The sugar cane and PLA materials had degradation rates similar to the Kraft paper control and meet the compostability criterion of 60% degradation after 45-days. The polyethylene negative control and the compost inoculum soil demonstrated negligible degradation.

The PLA cup and container and the trash bag met the phytotoxicity requirements (poisonous to plants) and support growth of tomato seedlings after 10 days. The sugar cane plate met the phytotoxicity requirements and supported growth of cucumber seedlings after 4 days.

All of the soil samples from the compostable materials had lead and cadmium concentrations well below the limits of 30 mg/kg Pb and 17 mg/kg Cd. The measured amounts of cadmium and lead were less than 1% of the maximum allowable levels.

The degradation and disintegration results at the university farm demonstrate that the compostable materials degrade under moist manure-based compost. All of the materials disintegrated after 72 days. The potato-starch based tray, corn-starch based trash bag, PLA plate, PLA straw, and PLA container degraded at a similar rate as the cellulose control.

The degradation and disintegration results at the municipal compost facility demonstrate that the compostable materials degrade under moist green-waste compost. The PLA container, PLA cup, and PLA knife degraded at a similar rate as the Avicel cellulose control and were degraded completely in 7-weeks. The corn starch-based Biobag trash bag and sugar cane plate degraded at a similar rate as the Kraft paper control. The three materials degraded between 80 and 90% after 20 weeks.

The three compost environments demonstrated similar results. In particular, PLA degraded very well in cow-manure and green waste compost. The trash bag experienced higher degradation in the moist cow manure compost than in the green waste compost. The cow manure compost is the most active and the best medium for degradation of the PLA and starch based compostable materials. The laboratory and municipal compost had similar degradation results, where the PLA materials degraded very quickly and the starch based plastic bag degraded more slowly. The trash bag had similar degradation rates after 45 days in the laboratory and in the municipal compost facility of around 30% degradation. The Kraft paper sample also had similar degradation in the laboratory environment (61%) as in municipal compost facility (52%).

The sugar cane plate had the biggest difference in degradation rates between the two compost environments with higher degradation in the laboratory (63%) versus the municipal compost (19%). The moisture content was significantly higher in the laboratory experiment than at the municipal compost facility. The sugar cane plate is hydrophilic that can affect the degradation rate.

# Recommendations

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The research work can help increase the use of compostable plastic materials for selected applications. The compostable materials should be certified as compostable by BPI and included in procurement standards. A procurement officer or recycling coordinator can use the BPI certification as a minimum requirement for purchased compostable products. The compostable plastic materials should perform well in simple applications, e.g., food service ware, lawn and leaf refuse bags that have dry contents, grocery bags, department store bags, and pet bag products. The compostable plastics would not most likely perform well in trash bag uses due to the likely exposure to moist debris. Thus, trash bag use is not recommended at this time. Also, lawn and leaf bags might not be suitable for compostable plastics in wet environments.

Compostable plastic materials could be very economical for organizations and institutions that service a controlled population, e.g., hospitals, correctional facilities, schools, and cruise lines. The cost of disposal of waste at these locations can be offset by the use of compostable plastics, which have a compost nutrient value. Compostable plastics can be a benefit to compost operators by having an organic nutrient source that does not have the bacteria problems of food waste.

Compostable plastics can reduce the amount of plastic in the landfills. In 2003 for California, plastics accounted for roughly, 10% by weight of the materials in the waste stream. Compostable plastics make the most sense as replacements for clamshell and other rigid containers, which account for 24,627 tons and 22,081 tons respectively. If compostable plastics were implemented at several large institutions, the amount of plastic waste that can be diverted from the waste stream could approach 5,000 to 10,000 tons.

Appropriate labeling of compostable plastics is essential for effective use. The Society of Plastics Institute's (SPI) resin identification code for rigid plastics containers should not be used to indicate compostable plastics. Rather, a colored (e.g., green) label that is similar to the BPI logo would be appropriate with a circle and a "C" in the middle. The symbol should not be on the same location as the recycling symbol, but should be on the side of the container. In fact, a recycling symbol with a red line through it indicating that the plastic is not recycled would be helpful. Collection practices would have to be modified to include the use of compostable plastics. The compostable plastics should not be collected with recycled materials, but rather included with the yard waste compostable materials.

# Appendices

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Appendix A. Calculations

Appendix B. Pictures of Samples at the CSU, Chico Experimental Laboratory

Appendix C. Pictures of Samples at the CSU, Chico University Farm

Appendix D. Pictures of Samples at the City of Chico Municipal Compost Facility



## Appendix A. Calculations

The concentration of CO<sub>2</sub> in the compost container is found by converting the ppm concentration that is measured in the 320-ml measurement bottle to a ppm concentration in the 40-ml sampling tube, which has the same concentration as the compost container. First, the amount of g-mols of CO<sub>2</sub> present in the 320-ml measurement bottle is determined from the ppm concentration difference between the 320-ml bottle with 40-ml gas from the compost containers and the background ppm concentration of CO<sub>2</sub> in the room. The difference represents the amount of g-mols that was added from the 40-ml gas sample.

Secondly, the concentration, in g-mols/ml, that is the concentration of CO<sub>2</sub> in the compost container can be converted to ppm concentration of CO<sub>2</sub> with the use of the Ideal Gas Law relationship as described in Equation 1.<sup>[84]</sup> The gram-molecular weight for CO<sub>2</sub> is 44 g/mol.

$$ppm = \frac{RT}{P \times MW} \times mg / m^3 \quad \text{Equation 1}$$

where, P is the pressure in the vessel in mm Hg,

R is the universal gas constant, 62.4 (L- mmHg)/(°K -mol),

T is the temperature in Kelvin, and

MW is the gram molecular weight, g/mol.

Thirdly, the concentration of CO<sub>2</sub> in ppm can be converted to mg/m<sup>3</sup> by multiplying the ppm measurement by the gram molecular weight of CO<sub>2</sub> and then dividing by 24.45. This is valid when measurements are taken at 25°C and atmospheric pressure of 760 torr (760 mm Hg). For temperatures and pressures different than this, the concentration of carbon dioxide can be converted from ppm to mg/m<sup>3</sup> as described in Equation 2. The total amount of carbon is the concentration of carbon in grams per liter times the volume of the gas in the chamber of 1 liter as described in Equation 3.

$$mg / m^3 = \frac{P}{(RT)} \times MW \times ppm \quad \text{Equation 2}$$

where, P is the pressure in the vessel in mm Hg,

R is the universal gas constant, 62.4 (L- mmHg)/(°K -mol)

T is the temperature in Kelvin

MW is the gram molecular weight, g/mol

Fourthly, the grams of CO<sub>2</sub> can be converted to grams of Carbon by multiplying by the atomic mass of Carbon (12g) and then dividing by the molecular weight of CO<sub>2</sub> (44g), as described in Equation 4.

$$g_c = g_{CO_2} \times \frac{12}{44} \quad \text{Equation 4}$$

Lastly, the percentage of biodegradation of the materials, Equation 5, is calculated by dividing the average net gaseous carbon production of the test compound by the original average amount of carbon in the compostable sample and multiplying by 100.

$$\% \text{ biodegradation} = \frac{\text{mean}C_{g, \text{test}} - \text{mean}C_{g, \text{blank}}}{C_i} \times 100 \quad \text{Equation 5}$$

where,  $C_{g, \text{test}}$  is the amount of gaseous-carbon produced in sample, g,

$C_{g, \text{blank}}$  is the amount of gaseous-carbon produced in inoculum soil alone, g, and

$C_i$  is the amount of carbon in test compound added, g.

An alternative method to calculate the amount of carbon that is present in the ppm concentration involves a simpler calculation that relates the density of CO<sub>2</sub> and the density of air in the different volumes of gas. The calculation addresses the volume percent of CO<sub>2</sub> in the initial measurement container compared to the volume percent after adding 40-ml of the compost gas. First, the gas ppm concentration in the 320-ml measurement container is converted to volume percent CO<sub>2</sub> with Equation 6. Note, that ppm is mass of substance divided by 1 million times the mass of solution. Thus, 400 ppm of CO<sub>2</sub> represents 0.004% CO<sub>2</sub>.

$$\text{vol}\%_{\text{CO}_2} = \text{ppm}_{\text{CO}_2} \frac{\rho_{\text{air}}}{\rho_{\text{CO}_2}} \times 100 \quad \text{Equation 6}$$

where,  $\rho_{\text{air}}$  is the density of air, 1.2928 g/cc at 25 °C and 1 atmosphere pressure, and

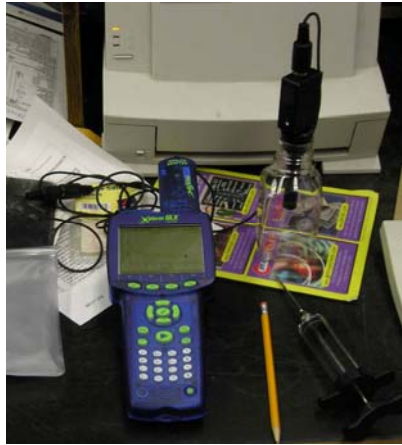
$\rho_{\text{CO}_2}$  is the density of CO<sub>2</sub>, 1.9768 g/cc at 25 °C and 1 atmosphere pressure.

Secondly, the volume fraction of CO<sub>2</sub> present in the initial concentration is multiplied by the 320ml volume to yield the volume of CO<sub>2</sub>, which is converted to mass of CO<sub>2</sub>. Similarly, the ppm concentration after the 40-ml is added is also converted to mass of CO<sub>2</sub>. Thirdly, the two mass values are subtracted to obtain the mass of CO<sub>2</sub> that is present in the 40-ml container. Lastly, the mass concentration is multiplied by the volume of the compost container to yield the mass of CO<sub>2</sub> that is present from the biodegradation process. As before, the mass of CO<sub>2</sub> can be converted to mass of carbon that will determine biodegradation rate of the composting materials.

**Appendix B. Pictures of Samples at the CSU, Chico Experimental Laboratory**



**Oven Line-up of Sample Jars**



**PASCO CO<sub>2</sub> Detector**



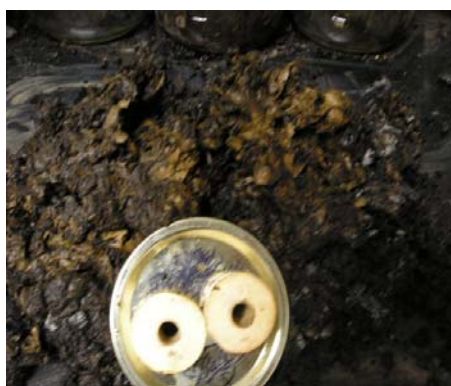
**Cellulose Start Sept 28, 2005**



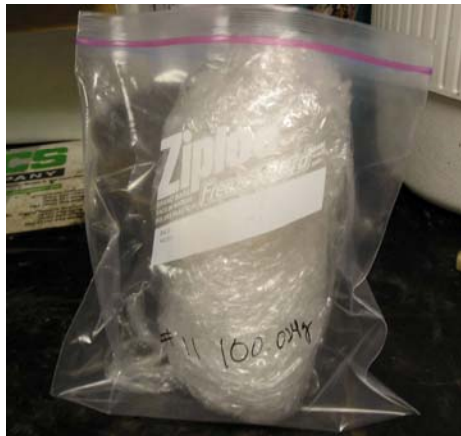
**End Nov 11 (45 days)**



**Kraft Paper Start Sept 28, 2005**



**End Nov 11 (45 days)**



**PE Wrap Start Sept 28, 2005**



**End Nov 11 (45 days)**



**BioBag Start Sept 28, 2005**



**End Nov 11 (45 days)**



**PLA Container Start Sept 28, 2005**



**End Nov 11 (45 days)**





**PLA Cup Start Sept 28, 2005**



**End Nov 11 (45 days)**



**Sugar Plate Start Sept 28, 2005**



**End Nov 11 (45 days)**

## ***Appendix C. Pictures of Samples at the CSU, Chico Farm***



**Compost Control June 13, 2005**



**CSU, Chico University Farm**



**Potato Starch Tray June 13, 2005**



**June 28, 2005 (15 days)**



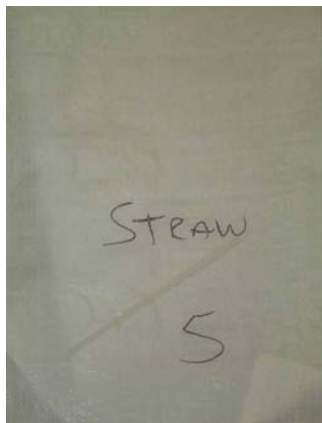
**PLA Plate June 13, 2005**



**June 28 (15 days)**



**August 1 (49 days)**



**PLA Straw June 13, 2005     June 28 (15 days)**



**August 1 (49 days)**



**PLA Fork June 13, 2005     June 28 (15 days)**



**August 1 (49 days)**

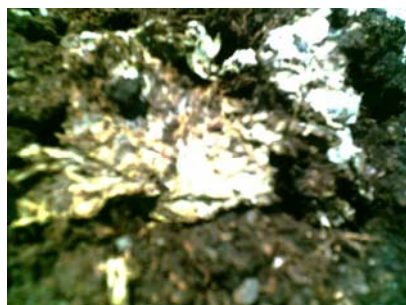


**PLA Cup June 13, 2005     June 28 (15 days)**



**August 1 (49 days)**





**BioBag Trash Bag June 13, 2005    June 28 (15 days)    August 1 (49 days)**



**PLA Container July 18, 2005    August 1 (14 days)    August 29 (43 days)**



## ***Appendix D. Pictures of Samples at the City of Chico Municipal Compost Facility***



**Compost Control July 28, 2005**



**City of Chico Compost Facility**



**Avicell Cellulose July 28, 2005**



**September 15 (7 Weeks)**



**PLA Knife July 28, 2005**



**September 15 (7 Weeks)**



**PLA Container July 28, 2005**



**September 15 (7 Weeks)**



**PLA Cup July 28, 2005**



**September 15 (20 Weeks)**



**BioBag Trash Bag July 28, 2005**



**Sept 15 (7 Weeks)**



**December 15 (20 weeks)**



**Kraft Paper July 28, 2005**



**Sept 15 (7 Weeks)**



**December 15 (20 weeks)**



**Sugar Cane Plate July 28, 2005**



**Sept 15 (7 Weeks)**



**December 15 (20 Weeks)**

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