

FINAL REPORT

Development of an Agile, Novel, Expeditionary Battlefield Manufacturing Plant using Recycled and Reclaimed Thermoplastic Materials

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March 2023

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REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188	
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.						
1. REPORT DATE (DD-MM-YYYY) 31/03/2023		2. REPORT TYPE SERDP Final Report			3. DATES COVERED (From - To) 6/21/2018 - 3/31/2023	
4. TITLE AND SUBTITLE Development of an Agile, Novel Expeditionary Battlefield Manufacturing Plant using Recycled and Reclaimed Thermoplastic Materials				5a. CONTRACT NUMBER 18-C-0017		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Prabhat Krishnaswamy and Richard Heggs Engineering Mechanics Corporation of Columbus (Emc2) Peter Stynoski, Jonathan Trovillion and Stephen Cosper U.S. Army Corps of Engineers, Engineering Research and Development Center-Construction Engineering Research Laboratory (ERDC-CERL), Champaign, Illinois Jose Castro The Ohio State University				5d. PROJECT NUMBER WP18-1047		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Engineering Mechanics Corporation of Columbus (Emc2) 3518 RIVERSIDE DRIVE, SUITE 202 COLUMBUS, OH 43221				8. PERFORMING ORGANIZATION REPORT NUMBER WP18-1047		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of the Deputy Assistant Secretary of Defense (Energy Resilience & Optimization) 3500 Defense Pentagon, RM 5C646 Washington, DC 20301-3500				10. SPONSOR/MONITOR'S ACRONYM(S) SERDP		
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) WP18-1047		
12. DISTRIBUTION/AVAILABILITY STATEMENT DISTRIBUTION STATEMENT A. Approved for public release: distribution unlimited.						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT This project involves both basic and applied research for the development of agile and mobile manufacturing technologies that take advantage of recycled and reclaimed plastics at forward operating bases (FOBs). The project offers a safe and environmentally responsible way to reduce disposal requirements and to turn specific thermoplastics waste-streams into value-added products for use by servicemembers.						
15. SUBJECT TERMS Waste to energy, emissions, waste reduction, manufacturing, additive, recycling, plastic, re-use, Thermoplastic						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UNCLASS	18. NUMBER OF PAGES 229	19a. NAME OF RESPONSIBLE PERSON Prabhat Krishnaswamy	
a. REPORT UNCLASS	b. ABSTRACT UNCLASS	c. THIS PAGE UNCLASS			19b. TELEPHONE NUMBER (Include area code) 614-459-3200	

FINAL REPORT
SERDP Project: WP18-1047

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LIST OF ACRONYMS

ABS	Acrylonitrile Butadiene Styrene
AM	Additive Manufacturing
AOR	Area of Responsibility
ARL	Army Research Laboratory
ASTM	American Society of Testing and Materials (International)
CB	Contingency Base
CDME	Center for Design and Manufacturing Excellence (OSU)
CERL	Construction Engineering Research Laboratory
CMU	Concrete Masonry Units (8x8x16 construction blocks)
CONUS	Continental United States
COR	Contracting Officer's Representative
DFP	Directly Funded Partner
DLA	Defense Logistics Agency
DoD	Department of Defense
Emc ²	Engineering Mechanics Corporation of Columbus
ERDC	Engineering Research and Development Center
FFF	Fused Filament Fabrication
FOB	Forward Operating Base
GDC	Goshen Die Cut (Goshen, IN)
IPR	Interim Progress Review
ISO	International Standards Organization
IV	Intrinsic Viscosity
JAMMEX	Joint Additive Manufacturing Model Exchange
LVDT	Linear Variable Displacement Transducer
MLSR	Monthly Letter Status Report
MRE	Meal, Ready-to-Eat
MSDS	Material Safety Data Sheet
NSWC	Naval Surface Warfare Center (Crane, IN)
OBE	Overcome by Events
OSU	The Ohio State University
PET	Polyethylene Terephthalate
PETG	Polyethylene terephthalate glycol
PLA	Poly Lactic Acid
PON	Point of Need
QPR	Quarterly Progress Report
rPET	reclaimed Polyethylene Terephthalate
SBIR	Small Business Innovation Research
TGA	Thermo-Gravimetric Analysis
UGR	Unitized Group Ration
USARCENT	US Army Central Command

ACKNOWLEDGEMENTS

The authors are very grateful to SERDP's Program Managers Dr. Robin Nissan and Dr. John La Scala, and also Mr. Braxton Lewis of Noblis for their efforts in guiding the project from initiation in 2018 to completion and their support as the project team maneuvered through numerous delays due to the pandemic. Dr. John La Scala's timely review and response to the draft Final Report are especially appreciated. We are also grateful to Mr. Marc Pepi of ARL and SERDP's Technical Oversight Committee for this project for all his valuable input and his feedback during In-Progress Review (IPR) Meetings, and introductions provided to others in the industry through his contacts. Ms. Katherine Tran of Noblis/SERDP provided administrative support with monthly and Quarterly Progress Reports and other programmatic issues.

At Emc², the support of Mr. Gary Hattery, Ms. Ann Roty, Mr. Russ Hattery, Mr. Mike Fishman Ms. Sushma Pothana, and Mr. Cameron Orth are gratefully acknowledged. At ERDC-CERL the contributions of Ms. Megan Krieger, Mr. Bruce Macallister, Mr. Tanner Wood, Mr. Robert Murphy, and Mr. Grant Siegmund are gratefully acknowledged.

ABSTRACT

INTRODUCTION AND OBJECTIVES

This project involves both basic and applied research for the development of agile and mobile manufacturing technologies that take advantage of recycled and reclaimed plastics at forward operating bases (FOBs). The project offers a safe and environmentally responsible way to reduce disposal requirements and to turn specific thermoplastics waste-streams into value-added products for use by servicemembers.

TECHNICAL APPROACH

Of the various plastics in the waste stream at FOBs, Polyethylene Terephthalate (PET, #1) and Polyethylene (PE, #2 and #4) constitute the most significant portion. Based on earlier research by the Principal Investigator for the US Army, a complete, mobile, extrusion plant for waste PE had been designed. This research effort focuses on reclaimed PET, (rPET) from beverage containers (such as water and beverage bottles) and involves waste stream characterization, source separation, sorting and cleaning technologies, development of rPET based material formulations, and processing technologies to convert rPET into high-valued, much-needed products at the Point of Need (PON) by servicemembers.

RESULTS

The major deliverables from this project involve the following four complete designs for mobile and agile manufacturing plants that can be fabricated and built in the continental United States. (CONUS) in 20-foot International Standard Organization (ISO) containers, shipped to the required bases and operated on site by a servicemember or contractor.

- Sorting and washing plant that can produce clean rPET flake at 300 pounds per hour,
- Filament extrusion plant for high-valued Additive Manufacturing of much-needed components at PON with rPET flake
- Dimensional rPET based plastic lumber extrusion plant that can operate at 200 pounds per hour, and
- Manufacturing plant to convert mixed plastics waste into light construction blocks at 30 tons per month.

BENEFITS

The clear benefits of this project involve not only significant improvements in the self-sustainability for the servicemember with a reduction in the logistics tail, but also the ability to manufacture value-added products on-demand, on-site at the FOBs. Additionally, the results of this project will reduce need for open burning which would benefit not only the environment, but the health of the personnel performing the burn as well.

EXECUTIVE SUMMARY

INTRODUCTION

This research project directly addresses the original Statement of Need (SON) for this SERDP solicitation which is to conduct basic and applied research for the development of agile manufacturing technologies that take advantage of recycled and reclaimed materials. This research focuses on waste thermoplastics streams generated at forward operating bases (FOBs). Polyethylene Terephthalate (PET) is the largest components of the plastics waste stream at FOBs. Therefore, this research involves the development of novel, mobile and agile manufacturing technologies that converts reclaimed PET (or rPET) raw materials into value-added end-product(s) that would be useful to servicemembers on-site at FOBs and Logistics Support Areas (LSAs).

OBJECTIVES

The objective of this project is to conduct primarily applied research to develop innovative agile manufacturing plants for onsite fabrication of value-added products from rPET materials in FOB waste streams. The specific questions to be addressed include (i) how much and what types of rPETs are available at FOBs (quantification and classification); (ii) how to adapt existing commercial technologies to convert waste rPET bottle into clean raw materials for agile manufacturing, (iii) what possible material formulations may be developed for field-manufacturing of products relevant to FOBs, (iv) relevant processing methods and equipment to fabricate products from rPET; (v) modifying existing manufacturing equipment so as to make it agile for mobility in 20-foot ISO containers, and (vi) developing product applications for on-site use by servicemembers.

TECHNICAL APPROACH

Emc²'s lead collaborator and directly funded partner (DFP) in the proposed work is the US Army Corps of Engineers, Engineer Research & Development Center, Construction Engineering Research Laboratory (ERDC-CERL), in Champaign, IL. Over the last two decades, CERL engineers have conducted extensive research and investigations on the applications and conversion of recycled plastics and other waste stream products into durable structural materials.

Specifically, in relation to this project, the US Army Logistics Innovation Agency has prepared a report on their work on "Contingency Base Waste Analysis (CBWSA)¹." A detailed review of this report and its classification of waste streams at FOBs indicates that plastics constitute approximately 16% by weight of the solid waste at FOBs. Amongst these, the two major components of plastics waste are Polyethylene Terephthalate (PET) at approximately 40% and Polyethylene (PE) at 23%. *It was estimated that up to 0.5 lbs/servicemember/day of waste PET could be produced at bases, and therefore targeting this largest source of plastics waste for recycling into useful products was considered to be of highest priority in this work.*

Recycling of PET is a very mature industry stateside. Many plants currently convert upwards of 60M lbs. per year of baled waste PET beverage containers into clean rPET pellets. The process of

¹ "US Army Central (USARCENT) Area of Responsibility (AOR) Contingency Base Waste Stream Analysis (CBWSA)," Final Report prepared by the U.S. Army Logistics Innovation Agency, March 2013.

grinding, sorting, separating, and washing waste PET materials and producing raw materials is highly automated and a complete circular economy exists to generate food grade rPET pellets from waste beverage containers. Given this background, the major focus of the applied research in this project involves adapting and modifying the existing recycling technologies to make it (i) agile and mobile, that is, to 'miniaturize' the available recycling equipment and systems to be installed and operated in 20-foot ISO Containers (ii) select only those product manufacturing technologies that are 'robust' enough for FOB use, including Additive Manufacturing (AM) methods that could use rPET to make replacement 'parts' at point of need (PON), and, (iii) to 'evaluate' the performance characteristics of the products developed with rPET and provide guidance for their applications for FOBs for servicemembers.

With the above background, the five major Tasks undertaken in this project are as follows:

- Task 1 - Sorting of FOB Waste Streams for rPET
- Task 2 - Development of rPET based Formulations
- Task 3 - Modification of Extrusion Processing Technologies
- Task 4 - Evaluation of rPET Products for use at FOBs
- Task 5 - Program Coordination, Reports and Deliverables

A schematic of the four technical tasks is shown below in Figure ES-1 which is followed by a summary of the work done in each of them.

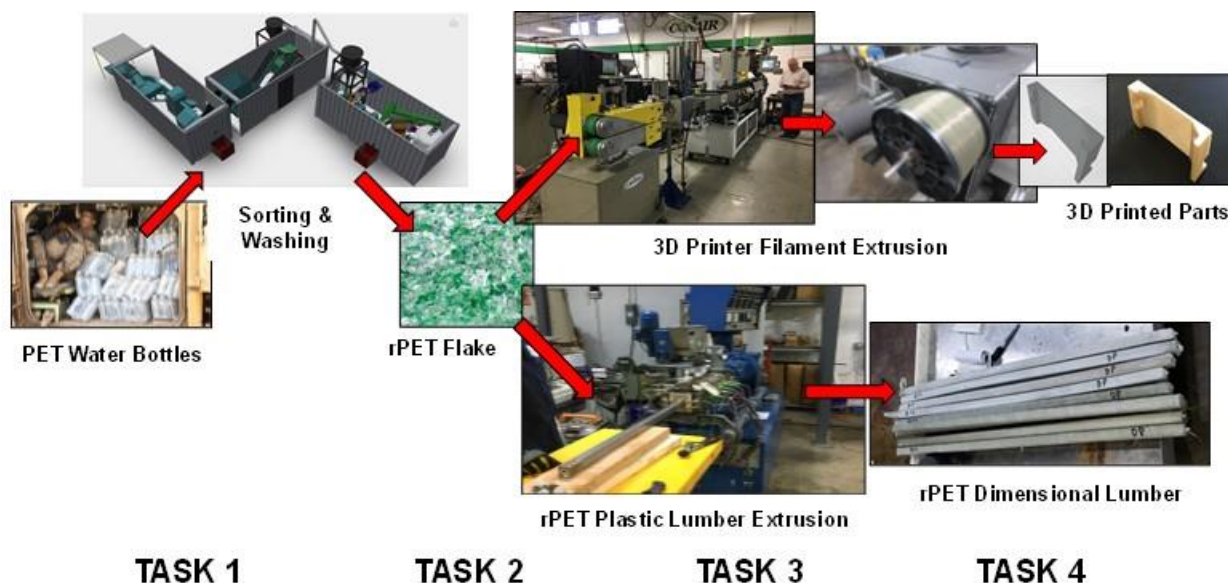


Figure ES-1 – Schematic showing the various steps in the technical tasks

Task 1 efforts involved design of a recycling system that could be fabricated and installed in 20-foot ISO containers. This system is designed to accept rPET water bottles 'as is', that is, with or without labels and caps, and convert them through a series of processing steps to clean uniform rPET flake at approximately 300 lbs/hr. The rPET flake from this system would then serve as feedstock raw material for manufacturing useful products at the PON in FOBs.

Task 2 involves characterizing the rPET flake available from the recycling system and then developing appropriate material formulations for various products that could be manufactured at FOBs, also in 20-foot ISO containers. While one of the major thrust areas of this work involved Additive Manufacturing (AM), as called out by the original SON, it was determined that AM alone could only consume a very small fraction of the approximately 0.5 lbs/day/person of rPET generated by servicemembers. Therefore, formulations and products that could consume much larger quantities of rPET at FOBs were investigated as part of this effort.

Task 3 was the primary thrust of the entire project. The efforts in this task involved modifying plastics processing technologies (such as extrusion) to design mobile, agile manufacturing plants in modules, so that they can be fabricated, transported and operated in 20-foot ISO containers at bases by service members with limited training. Three separate mobile manufacturing plants were designed to use the rPET flake produced by the system designed as feedstock:

- i. *Filament extrusion plant*: an extrusion plant was designed to use clean, consistent 4-mm rPET flake to produce both 1.75mm and 2.85 mm diameter filament used widely by most 3D printing systems for additive manufacturing, also known as ‘fused filament fabrication (FFF).
- ii. *Plastic lumber plant*: a robust extrusion system for flow-molding dimensional rPET based ‘plastic lumber’ was designed that can tolerate a higher degree of contaminants in the waste stream. Samples of 1x1 and 2x4 dimensional rPET lumber were produced and evaluated for structural properties to determine design limitations and outdoor structural applications in the field.
- iii. *Construction block plant evaluation*: ‘ByBlock’ is a compression molded construction block manufacturing technology developed by ByFusion of Gardena, CA for handling not only rPET but also converting all types of mixed plastics waste into blocks dimensioned as a standard concrete masonry unit (CMU) for light outdoor construction. The validation of this technology could potentially convert very large quantities (up to 30 tons/month) of mixed plastics waste into useful products for on-site structures at bases. The structural performance of these ByBlocks were first characterized and a demonstration structure was built at ERDC-CERL to determine its feasibility at bases. ByFusion also showed that their standard manufacturing plant in 40-foot containers could be converted to two 20-foot containers for FOB applications.

Task 4 involves characterization of the properties of products developed from the trials conducted to develop and validate the three manufacturing plants designed in Task 3. The rPET filaments produced from the trials while designing the extrusion plants were used to print calibration parts, ASTM specimens and other parts from the DoD’s Joint Additive Manufacturing Model Exchange (JAMMEX) database to evaluate the material properties and confirm the feasibility of this AM technology. As part of this task a recent breakthrough AM technology from two commercial companies involving printing directly from plastic resin pellets and flakes (instead of filament) is evaluated.

The final Task 5 in this effort involved program management, reporting and preparation and submission of deliverables. As part of this task a number of publications and presentation on this project were made at national and international conferences and published in their proceedings. A complete list of these publications is included in the report.

RESULTS AND DISCUSSION

The major accomplishments during the course of this project were the designs of the four mobile, agile manufacturing plants in 20-foot ISO containers to convert waste PET bottles into useful products at PON at FOBs. These results are summarized next.

Figure ES-2 shows a schematic of the rPET ‘Sorting & Washing’ line that has been designed to receive waste PET water and convert it to clean rPET flake to be fabricated, installed, shipped and operated in three (3) 20-foot ISO containers.

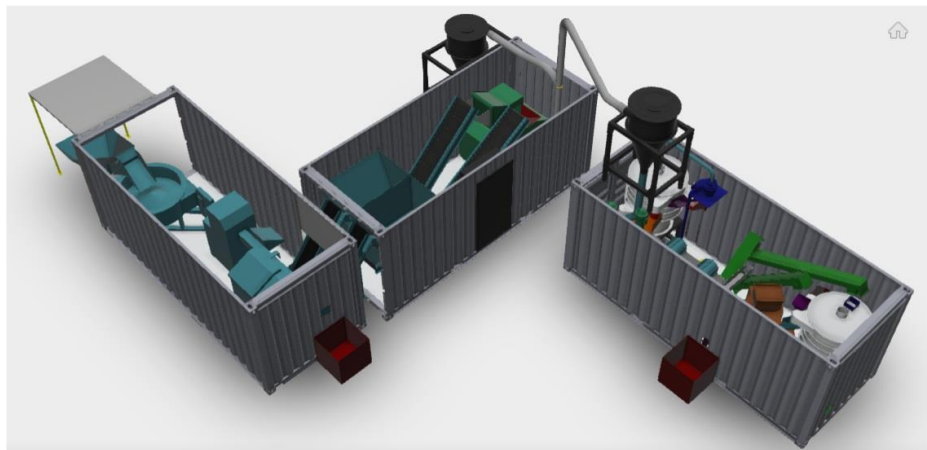


Figure ES-2 – PET water bottle sorting and washing manufacturing line

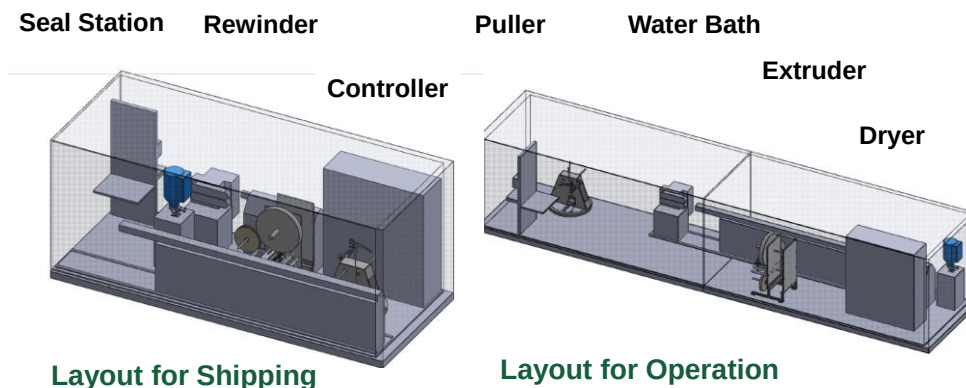
Container 1 receives the PET water bottles, sorts, singulates, and flattens them. Container 2 separates the flattened bottles into bins, grinds them to size and separates the rPET with a cyclone. The final Container 3 washes the ground flakes with a hydro-cyclone, separates contaminants and lighter plastics from the PET using a float-sink tank, and delivers clean, dry, consistent flake using a spin-dryer. The clean rPET flake, such as that shown in Figure ES-3 may be used to manufacture products at PON as described in the following section. The above plant has been designed to operate at 300 lbs per hour, (approximately 14,000 bottles).



Figure ES-3 – Sample of clean 4-mm rPET flake delivered from ‘sorting and washing line’

The second major deliverable from this effort is a mobile manufacturing line that converts the clean, consistent rPET flake in filament for 3D printing, or Additive Manufacturing. Figure ES-4 shows the schematic of the various pieces of equipment designed and configured in a single 20-

foot container for shipping, and to be operated on-site in two (2) 20-foot containers due to the length of the cooling tanks needed. The various pieces of equipment include a dryer (as PET is very sensitive to moisture), extruder, controller, tooling for both 1.75 mm and 2.85 mm filament extrusion, water bath, puller, winder and a seal station for the finished spools.



Figures ES-4 – Filament extrusion line for Additive Manufacturing (AM) or 3D printing

The filament produced from equipment similar to that designed in the above plant during the trials showed excellent characteristics and could be used to print parts using a variety of commercially available 3D printers. The properties of the printed parts with rPET were comparable to those of standard engineering resins used in 3D printing such as ABS, PLA, etc.

The third mobile extrusion plant designed during the effort is to convert much larger amounts of rPET flake (up to 200 lbs/hr; much higher than those used in AM filament) into dimensional ‘plastic lumber’ such as those available commercially using high density polyethylene (HDPE). A very robust, but simple flow-molding extrusion process was selected due to its ease of operation. Figure ES-5 shows a schematic of the plastic lumber extrusion line designed to be installed and operated in two (2) 20-foot ISO containers.

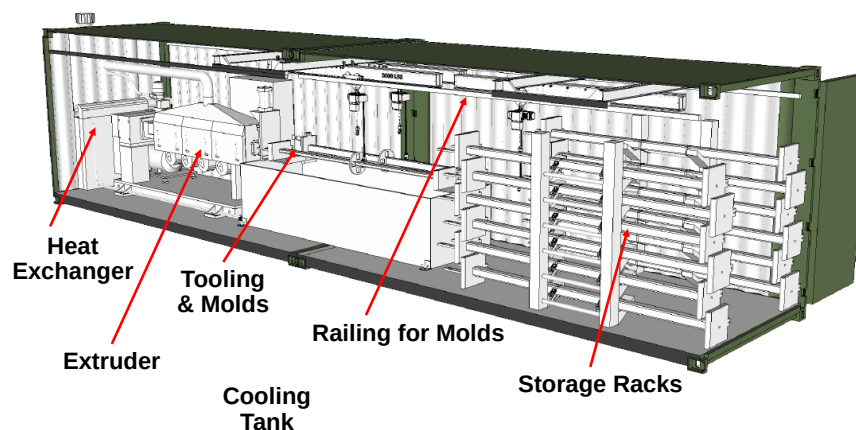


Figure ES-5 – Plastic lumber extrusion line using rPET flake in two 20-foot ISO containers joined together for manufacturing on-site

As indicated in Figure ES-5, the major pieces of equipment designed and sized for this plant include a heat exchanger for the cooling water, a single-screw extruder, tooling and molds for various lumber dimensions, cooling tank and storage racks for molds. The rPET based plastic lumber samples produced using similar equipment to that designed showed excellent flexural properties much higher than those from HDPE based lumber available commercially.

The fourth mobile, manufacturing plant investigated in this work involved evaluation of a commercially available technology to convert large quantities of mixed plastics waste into standard dimensional (8-in x 8-in x 16-in) construction blocks by ByFusion of Gardena, CA. The construction ByBlocks were tested to characterize its load carrying and structural capacity and its relevance to light-structural applications at FOBs. The commercial version of the manufacturing plant is designed to operate in a single 40-foot ISO container. During this effort the design was modified so the plant could be split into two 20-foot ISO containers for shipping and operations. A full-scale demonstration project with these ByBlocks was initiated at the US Army Corps of Engineers ERDC-CERL where a standard storage shed was designed and built to assess its applicability to FOBs. Figure ES-6 shows the schematic design of the storage shed, a photo of the construction process, and the photo of the shed after completion.



Figure ES-6 Demonstration project at ERDC-CERL - a storage shed designed and built with mixed plastic waste ByBlocks

As can be seen, the structures such as the storage shed provide an excellent use for both rPET based and other mixed plastics waste based on the evaluation of this unique technology during this effort.

IMPLICATION FOR FUTURE RESULTS AND BENEFITS

Based on the results obtained during the SERDP project involving extensive applied research, and the data obtained from the trials involving preparing and converting rPET flake into useful products it is recommended that further demonstration and validation be undertaken. Of the four agile manufacturing systems described above, developing both the sorting and washing line as well as the rPET plastic lumber extrusion facility should be pursued.

The AM work conducted during this effort has already been recognized by the DoD for its potential to convert rPET into high-valued, key components that can be produced at PON. The project team has partnered with a manufacturer of 3D printers that can use rPET pellets and a project for the US Army has recently been initiated to explore the demonstration and validation of this technology further. With regard to the ByBlock technology evaluation, ERDC-CERL is already working on a follow-on project with the Defense Logistics Agency (DLA) to further evaluate the structure that was built (storage shed) on site, and transfer of this technology to FOBs.

BENEFITS

The overall benefits of this project included improved self-sustainability of the warfighter in-theater, increased operational readiness, a reduction of the logistics tail, and the ability of the warfighter to manufacture on-demand at the PON. The successful results described above show that FOB plastics streams can be reused, with field-capable manufacturing processes in 20-foot ISO containers allowing the warfighter to potentially fabricate replacement parts or perform repair operations, while keeping subject waste streams out of open burn pits, or incinerators currently used on many FOBs. Reducing open burning would benefit not only the environment, but the health of the personnel performing the burn.

1.0 OBJECTIVE

The objective of the Statement of Need (SON) for this project is to conduct basic and applied research for the development of agile manufacturing techniques that take advantage of recycled and reclaimed materials. The proposed research focuses on plastics waste streams generated at forward operating bases (FOBs). In keeping with the objective, this research project offers a safe and environmentally responsible way to reduce disposal requirements and to turn thermoplastics waste-stream into value-added products for use by the warfighter. This work focuses specifically on waste Polyethylene Terephthalate (PET) from beverage containers, which is the largest components of the plastics waste stream at FOBs. This research involves technologies to convert reclaimed PET (rPET) raw materials into value-added end-product(s) that would be useful to the servicemembers on-site at FOBs and Logistics Support Areas (LSAs).

The specific topics addressed in this research project include (i) review of various types of rPET containers available at FOBs; (ii) modifying existing technologies to convert waste rPET bottle into clean raw material feedstock,, (iii) material formulations for field-manufacturing of products relevant to FOBs, (iv) plastics processing methods and equipment to fabricate products from rPET, and (v) adapting existing manufacturing equipment to make it agile for mobility in 20-foot ISO containers, and, (vi) recommending possible applications for rPET products manufactured for use by the servicemember in theater.

2.0 BACKGROUND

Forward operating bases generate considerable amounts of refuse including recyclable thermoplastic resins such as polyethylene (PE) and polyethylene terephthalate (PET). It is desirable to recycle these materials rather than disposing of them in a pit or open burning.

Emc²'s lead partner in this project work is the US Army Corps of Engineers, Engineer Research & Development Center, Construction Engineering Research Laboratory (ERDC-CERL), Champaign, IL. Over the last two decades, CERL engineers have conducted extensive research and investigations on the applications and conversion of recycled plastics and other waste stream products into durable structural materials. Along with Dr. Prabhat Krishnaswamy of Emc², Mr. Richard Lampo of CERL (now retired), co-chaired the ASTM D20 Plastics Committee. group and co-authored all of the ASTM standards relating to recycled plastic lumber materials.

Specifically in relation to this work, the US Army Logistics Innovation Agency has prepared a report on base waste stream analysis¹. A detailed review of this report and its classification of waste streams in FOBs indicates that plastics constitute approximately 16% by weight of the solid waste at FOBs. Amongst these, the two major components of plastics waste are Polyethylene Terephthalate (PET) at approximately 40% and Polyethylene (PE) at 23%.

With regard to the PE waste stream, Emc² had successfully completed a study in this technical area under a Small Business Innovation Research (SBIR) Phase I Project titled "*Innovative Mobile Extrusion Plant for On-site Fabrication of Ammunition Packaging Materials from Composite*

*Recycled Plastics.*²” This project was supported by the U.S. Army Armaments Research, Development & Engineering Center at the Rock Island Arsenal. As seen in Figure 1 and Figure 2 below, the entire manufacturing design for the composite recycled plastic plant from this effort could be transported in standard 20-foot ISO containers on aircraft and installed and operated in the FOBs.

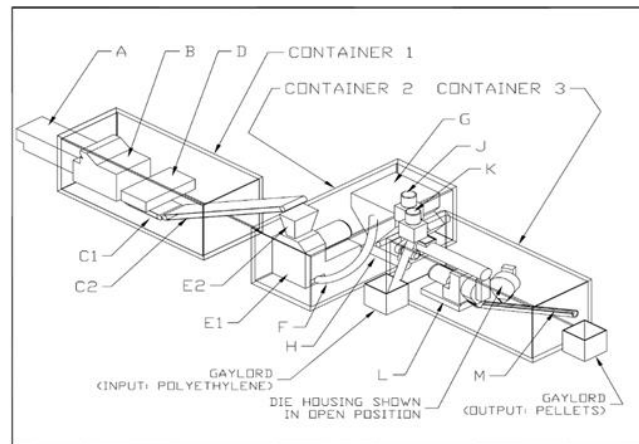


Figure 1. Composite Lumber Pelletizer Plant in ISO Containers in Operational View

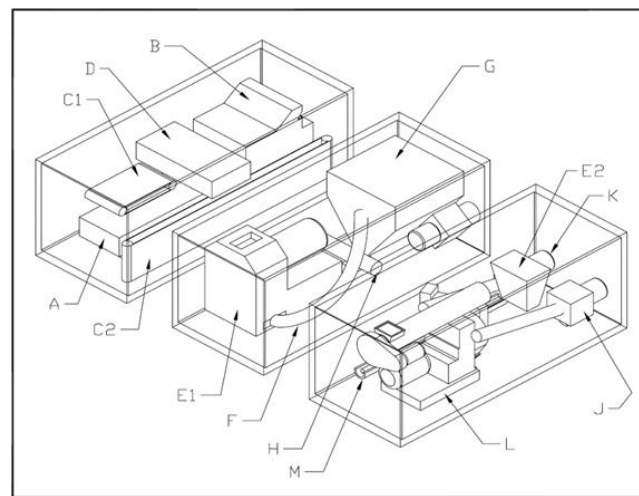


Figure 2. Composite Lumber Plant Assembled for Shipping in ISO Containers

Given the successful completion of the above work previously, PE recycling at FOBs was not considered as a focus area in this project. Therefore, the sole focus of this SERDP Project involved waste rPET, the largest component of the plastics waste stream at bases.

Recycling of PET primarily from beverage bottles and development of useful products from rPET stateside has been studied extensively since the 1980s and a comprehensive review of the state of

² Krishnaswamy, P. and others, “Innovative Mobile Extrusion Plant for On-site Fabrication of Ammunition Packaging Materials from Composite Recycled Plastics,” US Army SBIR Phase 1 Final Report by Emc², Contract Number: W15QKN-05-C-1187, July 2005.

the art is available in the literature³. Based on these technologies, a complete patented system for recycling of RPET stateside was developed by Rutgers University and licensed to many Material Recycling Facilities (MRFs) world-wide as far back as 1987⁴. Since then, there have been more recent review articles published on rPET⁵. Additionally, beverage companies such as PepsiCo and Coca Cola have actively been engaged in developing and implementing technologies to re-use rPET into the bottle manufacturing processes. PET recycling therefore is a very mature industry stateside, and, there are many recycling plants that currently convert upwards of 60-80M lbs/year of baled waste PET beverage containers into clean rPET pellets. The process of grinding, sorting, separating, and washing waste PET materials and producing raw materials is highly automated and a complete circular economy exists to generate food grade rPET pellets from waste beverage containers. The above technologies are mature and have been used to convert RPET into the following end-products stateside:

- Depolymerization into monomer (ethylene glycol and either terephthalic acid or dimethyl terephthalate) for re-use in beverage bottle/food container production
- Clean injection moldable pellets for beverage bottle/food containers
- Fiber for carpets and geotextiles
- Fiber for textile fabrics and nonwoven blankets
- Fibers for insulation applications
- Commingling of rPET fibers with other lignocellulosic fibers for composites
- Extruded sheets for packaging
- Extruded banding straps for shipping

A thorough review of the above technologies was conducted so as to focus only on those products and manufacturing processes that could be directly applicable in the forward operating environment. This review led to the technical approach described in the next section and the tasks that are conducted in the subsequent work.

³ Milgrom, J., “*Polyethylene Terephthalate (PET)*”, Chapter 3, *Plastics Recycling - Products and Processes*, J Ehrig, Editor, Hanser, 1992.

⁴ Nosker, T. and others, “*Technology Transfer Manual, Plastics Beverage Bottle Reclamation Process*,” Center for Plastics Recycling Research, Rutgers - The State University of New Jersey, 1987.

⁵ “*Waste Management of PET Bottles*,” *Journal of Environmental Research and Development*, pp 862, Vol. 2, April – June 2008.

3.0 TECHNICAL APPROACH AND RESULTS

With the above background, there were five major Tasks undertaken in this project as follows:

- Task 1 - Sorting of FOB Waste Streams for rPET
- Task 2 - Development of rPET based Formulations
- Task 3 - Modification of Extrusion Processing Technologies
- Task 4 - Evaluation of rPET Products for use at FOBs
- Task 5 - Program Coordination, Reports and Deliverables

A schematic material flow chart of the relation between the four technical tasks is shown below in Figure 3 which is followed by a summary of the work done in each of them.

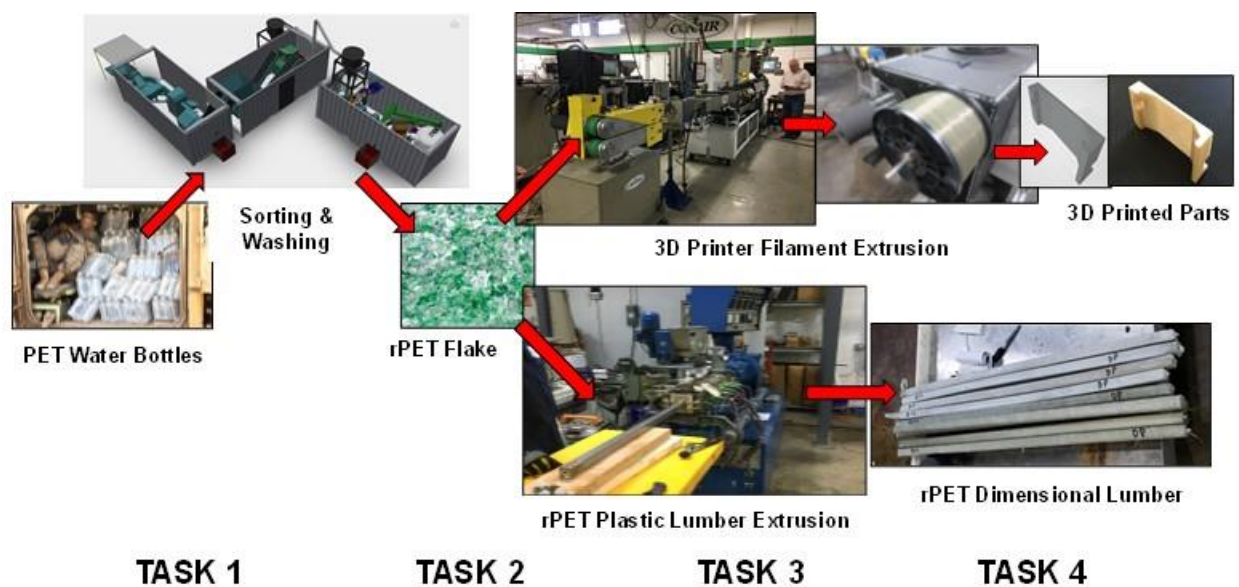


Figure 3. Schematic showing the various steps in the technical tasks

The various subtasks and activities in each of these tasks are detailed next. Only the key accomplishments during the four-year effort are summarized below with details provided in the various documents listed in Appendix A.

3.1 TASK 1 - SORTING OF FOB WASTE STREAMS FOR RPET

The overall objective of Task 1 involves design of a recycling system that could be fabricated and installed in 20-foot containers to accept rPET waste and convert it to feedstock for manufacturing products. The five subtasks involved are listed below:

- *Subtask 1.1 - Attend coordination meeting with ERDC-CERL and NSW*
- *Subtask 1.2 - Contact/ Visit rPET suppliers and processors*
- *Subtask 1.3 - Estimation of rPET available at FOBs*
- *Subtask 1.4 - Equipment specifications for rPET sorting*

- *Subtask 1.5 - Final configuration of FOB rPET sorting facility*

The technical approach and results for each of these is described next.

3.1.1 Subtask 1.1 Attend coordination meeting with CERL and NSWC

The meeting with ERDC-CERL was the first step to coordinate all tasks and activities on the project as they are a directly funded partner (DFP) on this project. Emc² was also requested to coordinate efforts on this project with NSWC in Crane, IN as they apparently had similar work underway. During the visit it was found that NSWC had only one staff member who worked on additive manufacturing (AM) with recycled plastics. And after review of this work was determined that the results were not directly relevant to the objectives of this project. Furthermore, the staff member at NSWC moved on, and hence, further coordination was deemed unnecessary.

3.1.2 Subtask 1.2 Contact/ Visit rPET suppliers and processors

As indicated above, since PET recycling is a mature industry stateside, several recycling operations were contacted and extensive discussions held prior to conducting site visits to three (3) plants in Ohio. Emc² staff visited Phoenix Technologies in Bowling Green, OH to review their sorting and cleaning operations, equipment and system configurations for their rPET bottles to pellet line. Appendix A-1 provides the trip report from this visit which also details the various steps and the equipment configuration used in their rPET recycling plant.

Phoenix was specifically very helpful in defining possible configurations for a smaller ‘Sorting and Washing’ for work under Subtask 1.4 and 1.5 below. And, they were also a source for purchasing both rPET flakes and pellets, that would be representative of the material at FOBs. Therefore, Emc² purchased two grades of their rPET pellets and two grades of flakes from Phoenix to conduct conducting the experiments and trials in other tasks described below.

3.1.3 Subtask 1.3 Estimation of rPET available at FOB

This subtask was led ERDC-CERL, the DFP on this project. The input from CERL and their analysis regarding volume of rPET available, level of sorting and percent contamination and proposed scale of operation at FOB was very critical to sizing, designing, and specifying the equipment configuration and system for rPET recycling at FOBs. Based on the analysis of the data presented in the USARCENT report¹ the approximate weight of rPET that is available at five (5) Contingency Bases (CB) in the middle-east region is summarized below in Table 1.

Table 1. Estimated Amounts of rPET Available at Five Contingency Bases

Data for	Kuwait	Afghanistan	Afghanistan	Afghanistan	Afghanistan
Contingency Base (CB)	CB#1	CB#2	CB#3	CB#4	CB#5
Population	10000	10000	7500	10000	3500
MSW tons/day	106	100	80.625	255	16
lbs/day	212,000	200,000	161,250	510,000	32,000
lbs/person /day	21.2	20	21.5	51	9.2
PET % of MSW	10.60%	7.00%	5.50%	6.10%	3.20%
lbs/day	22,472	14,000	8,869	31,110	1,024
lbs/year	8,202,280	5,110,000	3,237,094	11,355,150	373,760
lbs/person /day	2.25	1.40	1.18	3.11	0.29
lbs/hour	936	583	370	1,296	43
RPET: lbs/day for 8-hr x 5-day shift	31,461	19,600	12,416	43,554	1,434
Equipment Capacity Needed, lbs/hr	3933	2450	1552	5444	179

The most significant finding from the above referenced study and analyses of these data is that the quantities of waste PET available for recycling *is far greater than originally anticipated ranging from 373,760 lbs/year to over 8M lbs/year*, as seen in Table 1. This would require appropriate scale of equipment to convert rPET to useful end products that can handle 1,434 lbs to 31,461 lbs/day for a 40 hour/week operation. This scale of operation can pose significant challenges in modular design of manufacturing equipment that can be assembled stateside, shipped to and then operated at FOBs.

CERL further refined the estimate of the amount of waste PET available per person at an FOB to the range of 0.3 to 0.5 lb of rPET per servicemember per day. This figure was therefore used to size the equipment needed to process rPET bottles to feedstock material. Additionally, it was determined through discussions with CERL that the equipment should be sized to accommodate a contingency base with 1000 to 2000 servicemembers, which could be scaled if needed with multiple units for larger bases (> 5000 persons).

The above results were discussed with both CERL and SERDP staff and the Technical Oversight Committee (TOC) during the In-Progress Review (IPR) meeting. It was then decided that other exemplar products that could consume much larger volumes of rPET materials in agile manufacturing plants (in 20-foot ISO containers) should be considered and were pursued in this work as described below in Tasks 2 through 4.

3.1.4 Subtask 1.3 Equipment specifications for rPET sorting

Emc² conducted a detailed review of the various vendors for Sorting and Washing lines for waste PET including attending the Plastics Recycling Conference & Trade Show in March 2019 in Washington, DC. After several follow up meetings with vendors, Mr. Curt Cozart of CommonSense Solutions Inc., NJ, a world-renowned industry expert in the design of rPET (and other waste plastics) recycling operations was engaged to work with the team to design the modular ‘Sorting & Washing’ line in 20-foot ISO containers.

After extensive discussions and review of the estimates for waste rPET bottles available as detailed above and the constrain of operating in 20-foot containers it was decided that the design of this

‘Sorting and Washing’ plant *would be restricted to waste PET water bottles with or without labels and would be sized to operate at 300 lbs/hour*. Based on this analysis, a complete process flow diagram with specific equipment needed for sorting and washing the PET bottles was designed in a three (3) 20-foot ISO container system as shown in Figure 4, Figure 5 and Figure 6.

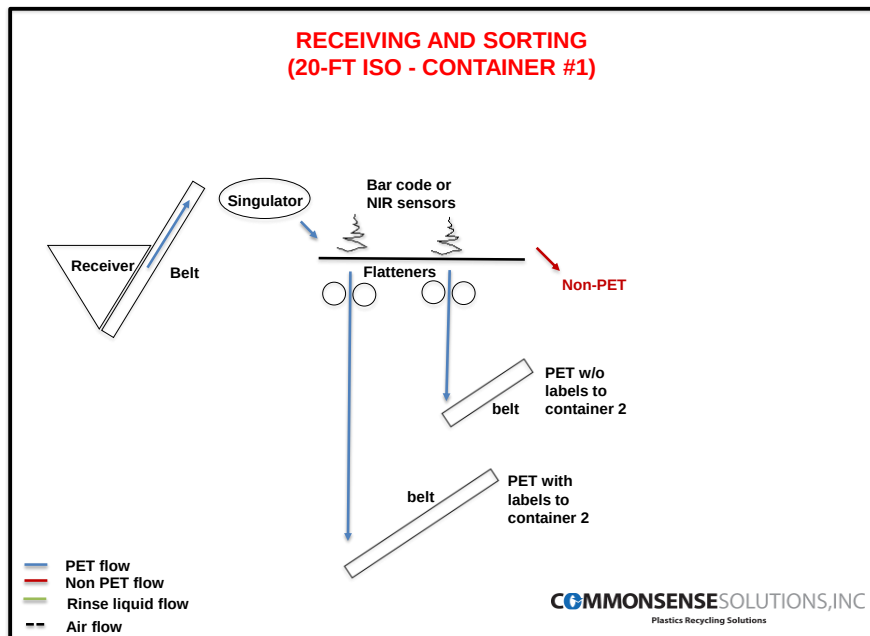


Figure 4. Process flow diagram for rPET Sorting and Washing Line – Container 1

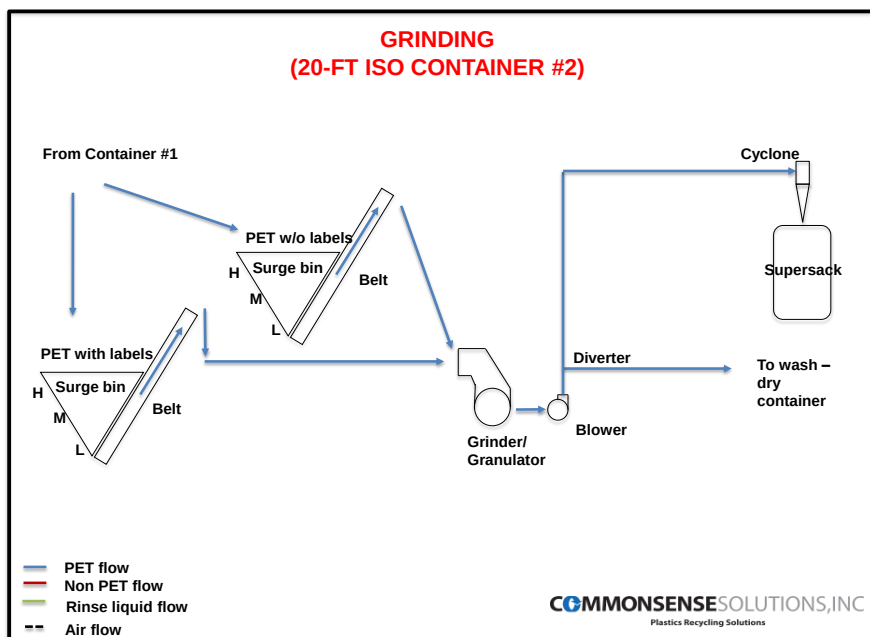


Figure 5. Process flow diagram for rPET Sorting and Washing Line – Container 2

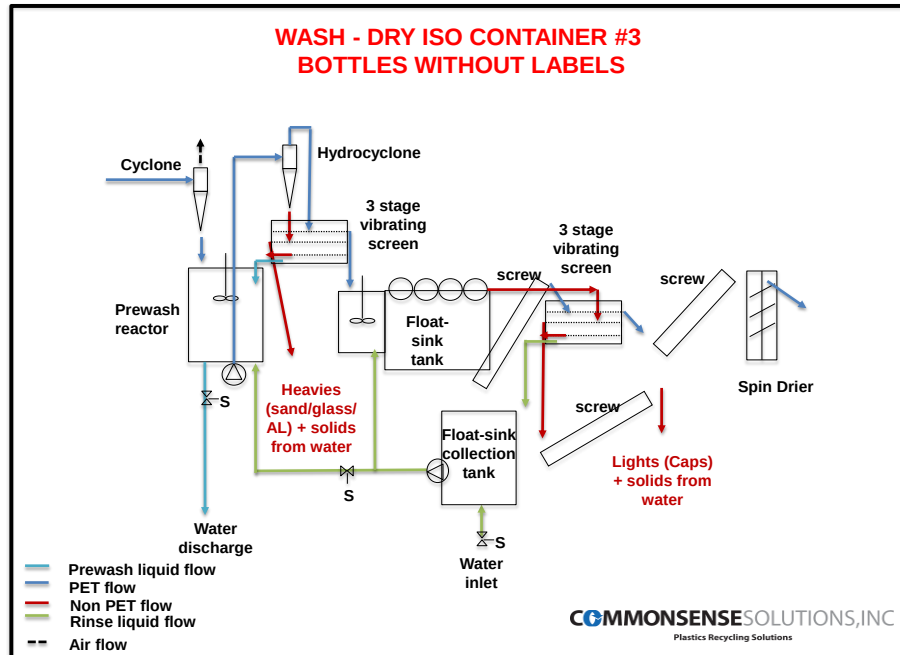


Figure 6. Process flow diagram for rPET Sorting and Washing Line – Container 3

A complete report with details of the above mobile, manufacturing plant to produce clean rPET flake along with all the equipment specifications, utility requirements and budgetary estimates is provided in Appendix A-2 (Note: Some information about this system is considered proprietary by the vendor and is therefore marked as such and may need to be restricted for use outside of federal government agencies.)

3.1.5 Subtask 1.5 Final configuration of FOB rPET sorting facility

Once the above design was completed, the next step involved conducting confirmation trials of key pieces of equipment in the above design that are unique to this ‘small-scale’ 300 lb/hour mobile sorting and washing line. These trials were conducted at equipment vendor sites for the singulators and sorters for bottles, and at commercial recycling facility for the hydrocyclone operation. These trials were recorded on video (due to the pandemic staff was unable to attend) to confirm that waste PET bottles mixed with contaminants could be successfully converted to clean rPET flake. Figure 7 shows photographs from these trials of the mixed water bottles and also the clean rPET flake generated from these trials – confirming that this mobile system designed for FOBs would work successfully.

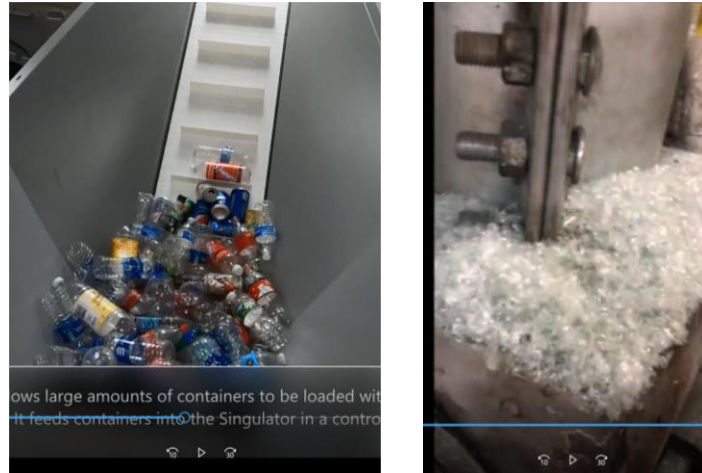


Figure 7. Photos of PET bottles used in trials and discharged clean dry rPET flake

Upon successful completion of these field trials, the original design was modified as needed to finalize the configuration of the mobile ‘Sorting and Washing Line’ that could be built during any follow-on demonstration and validation phase undertaken in a follow-on project. Figure 8 shows a schematic overview of the three 20-foot ISO containers of this finalized design with further details provided in Appendix A-2 (Note: Some information about this system is considered proprietary by the vendor and is therefore marked as such and may need to be restricted for use outside of federal government agencies.).

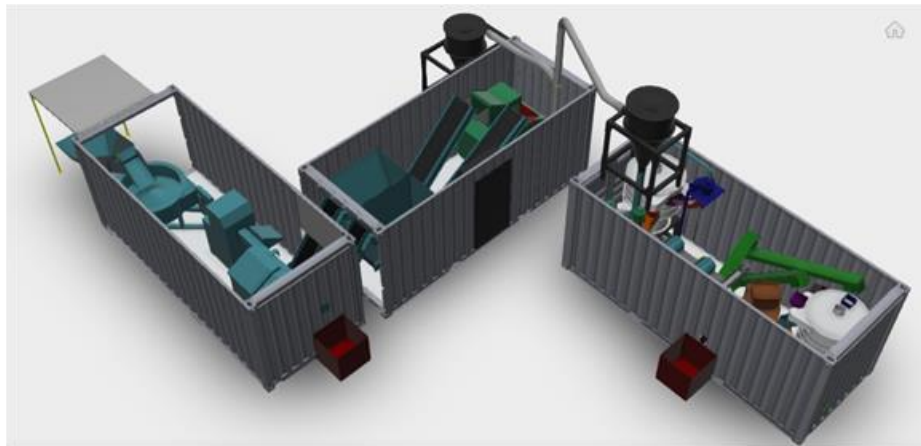


Figure 8. Final configuration of PET water bottle sorting and washing line

Container 1 receives the PET water bottles, sorts, singulates, and flattens them. Container 2 separates the flattened bottles into bins, grinds them to size and separates the rPET with a cyclone. The final Container 3 washes the ground flakes with a hydro-cyclone, separates contaminants using a float-sink tank, and delivers clean, dry, consistent flake using a spin-dryer. The clean rPET flake, such as that shown in Figure ES-3 may be used to manufacture products at PON as described next. The above plant has been designed to operate at 300 lbs. per hour, (14,000 bottles).

Upon review of the above system, and further discussions with the ERDC-CERL staff, there was a suggestion to further ‘miniaturize’ this Sorting and Washing Line solely for AM applications. Since AM requires much smaller quantities of rPET, this design was further modified with the constraint of using only PET water bottles without labels (labels significantly complicate the washing process detailed above) and target a capacity of only 100 lbs. per day (< 15 lbs. per hr).

Figure 9 shows this ‘small-scale’ system designed to be installed in a single 20-foot ISO Container along with a 3D printer that is designed to work with rPET flakes as feedstock material as will be discussed below in Task 3

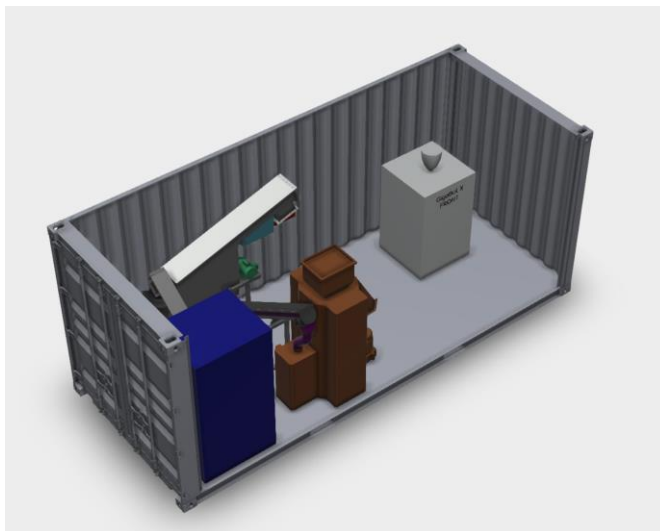


Figure 9. Final design of ‘small-scale rPET sorting line for AM applications

A detailed report on this configuration from CommonSense Solutions along with equipment specifications, utility requirements is provided in Appendix A-3. (Note: some information from vendors on this system is proprietary and is for federal government agency use only.)

3.2 TASK 2 - DEVELOPMENT OF RPET BASED FORMULATIONS

The objective of Task 2 involves characterizing the rPET flake available from the recycling system and then developing appropriate material formulations. The five subtasks planned under this task originally were:

- *Subtask 2.1 Review current formulation for rPET products*
- *Subtask 2.2 Initial rPET formulation for 3D filament*
- *Subtask 2.3 Finalize rPET formulation for 3D filament*
- *Subtask 2.4 Initial rPET formulation for strapping tape*
- *Subtask 2.5 Final rPET formulation for strapping tape*

While the major thrust area of this work involved feedstocks for 3D filament for Additive Manufacturing (AM), as called out by the original SON, it was determined that AM could only

consume a very small fraction of the approximately 0.5 lbs/day/person of rPET generated by service members. Therefore, formulations and products that and applications that could consume much larger quantities of rPET at FOBs as other exemplar products were investigated.

The final two subtasks involved strapping tape as a larger volume application for rPET was not pursued after preliminary analysis showed that this product was not suitable for FOB applications. Other exemplar products that consume larger quantities of rPET such as dimensional plastic lumber and construction type ‘ByBlocks’ were explored in this project. These additional rPET products are detailed under Task 3 below. No further efforts were undertaken under Subtasks 2.4 and 2.5 above since they were ‘Overcome by Events’ (OBE). With this background Subtasks 2.1 through 2.3 are detailed next.

3.2.1 Subtask 2.1 Review current formulation for rPET products

There were extensive discussions held with CERL staff, the material vendor Phoenix Technologies, as well as other companies working with rPET in OH, IN, and FL regarding rPET formulations that would be most suitable for the applications being considered – AM, flow-molded dimensional lumber and compression molded construction blocks (ByBlocks). Also reviewed was the use of rPET flake versus pellet for each process being considered, as the former would require much smaller equipment footprint and operator skills to use at FOBs.

It was first determined that the rPET materials available stateside, from a commercial recycling company such as Phoenix would be procured for all trials in this work, as material from actual FOBs was not feasible to procure for logistical reasons. The following grades for rPET were therefore deemed feasible for further FOB applications:

- rPET pellets from clear bottles with an intrinsic viscosity (IV) range of 0.65 to 0.75 that are dried to < 100 ppm moisture and fully crystallized with no contaminants for 3D printer formulation,
- clean, clear rPET flake from ‘mid-stream’ process could be used for AM applications if they had < 100 ppm moisture and fully crystallized,
- rPET flakes from clear or green bottles with an IV =0.75 dried to < 100 ppm moisture for trials to manufacture dimensional lumber,
- rPET flakes that are sufficiently dried (that could contain other contaminants) could also be used for plastic lumber and shapes using flow molding intrusion process,
- rPET with even significant components of other waste plastics resins and contaminants could be used for the ByBlock process using the ByFusion technology.

3.2.2 Subtask 2.2 Initial rPET formulation for 3D filament

The rPET grades purchased from Phoenix effort are shown in Table 2 below.

Table 2. List of rPET Materials Procured from Phoenix for Various Application

Material	Specimen ID	Specification / Notes	Applications
rPET Pellets - NLP Clear	rPET - 1	IV = 0.7; fully crystallized; ~5% amorphous	filament and plastic lumber
rPET Pellets - NLP-H	rPET - 2	IV = 0.75	plastic lumber
rPET 4mm white flake	rPET - white flake	4mm fully crystallized flake; noncommercial	filament and plastic lumber
rPET green Flake	rPET - green flake	green clean flake post wash + sorted	Plastic lumber

Figure 10 below shows photographs of the four resin samples procured. As may be seen the rPET-1 and rPET-2 grades visually do appear to be the same. Material Safety Data Sheet (MSDS) sheet for all rPET grades along with the Technical Specification for each of the three commercial grades (other than the non-commercial rPET-white flake) along with an injection molding processing guide are provided in Appendix A-4. For reference, a technical data sheet for virgin PET material from the literature is also included in Appendix A-4.



rPET pellet fully crystallized



rPET pellet (amorphous)



rPET white flake (water bottle)



rPET green flake (beverage bottle)

Figure 10. Photos of rPET sample formulations purchased for trials

3.2.3 Subtask 2.3 Finalize rPET formulation for 3D filament

Before finalizing the rPET formulation for 3D filament in AM, several activities in Tasks 3 and 4 needed to be completed, to confirm that the recommended formulation would be suitable for FOB

applications. As discussed in Task 3 below, it was first shown that clean rPET white flakes such as those shown in Figure 10 above could be used directly to make 1.75mm and 2.85 mm filaments spools, shown in Figure 11 for AM without the need to convert them to pellets.



Figure 11. Photo of 2.85 mm AM filament spool made with clean rPET flake

After completing the material property evaluation of AM parts printed described in Task 4 below, the final formulation for rPET based 3D printing filament would require the following steps: (i) the waste water bottles had to be cleaned so as to be free from labels, adhesives, and caps (usually made of PP or PE), (ii) the ground material had to undergo a size-reduction and sifting step that would result in a uniform, clean 4-mm size flake, (iii) the flake has to be dried to less than 100 ppm % moisture at 225 F for a minimum of 4 hours, and (v) all spools of filament need to be shrink-wrapped to prevent any moisture uptake by the rPET filament until used at the PON.

The conclusion from this task is that AM filament with rPET flake processed using the above outlined steps can be successfully used to print replacement parts made with other engineering plastics such as ABS at FOBs. Further enhancement of both part quality and ductility if needed, can be achieved with the use of 10% Kraton (FG 1924) as an additive in filament manufacturing as described in Task 3 below.

3.3 TASK 3 - MODIFICATION OF EXTRUSION PROCESSING TECHNOLOGIES

This task constitutes the primary thrust of the overall project. The overall goal involves modifying plastics processing technologies (such as extrusion) to design mobile, agile manufacturing plants in modular units to be fabricated, transported and operated in 20-foot ISO containers at bases by servicemembers with limited training. Following is the list of subtasks:

- *Subtask 3.1 Initial trials for rPET filament extrusion*
- *Subtask 3.2 Final trials for rPET filament extrusion*
- *Subtask 3.3 Configuration of rPET filament facility at FOB*
- *Subtask 3.4 Initial trials for rPET strapping extrusion (OBE)*
- *Subtask 3.5 Final trials for rPET strapping extrusion (OBE)*

- *Subtask 3.6 Configuration of rPET strapping facility at FOB(OBE)*
- *Subtask 3.7 Processing and evaluation of rPET based plastic lumber and shapes*
- *Subtask 3.8 Processing and evaluation of rPET based ByBlocks*

As indicated previously in Task 1, the work on rPET strapping extrusion was determined not to be suitable for FOB applications very early during this project and therefore Subtasks 3.4, 3.5 and 3.6 will not be discussed further. Instead, two other exemplar products involving dimensional plastic lumber with rPET formulations under Subtask 3.7 and ByFusion construction ByBlocks under Subtask 3.8 were investigated as they would consume significantly higher amounts of waste rPET and other plastics at contingency bases and are detailed below.

3.3.1 Subtask 3.1 Initial trials for rPET filament extrusion

Two methods of filament production were investigated, Filament Spinning, and Conventional Extrusion and are described next.

Filament Production Trials with Fiber Spinning: Fiber spinning is a mature technology that has been used extensively in converting rPET materials into very small diameter monofilament for textile and carpet applications. Therefore, this was the first technology investigated to produce rPET filament for AM applications. Hills Inc. from West Melbourne, FL is a major worldwide supplier of equipment in this industry. After review of their capabilities, their laboratory was contracted to conduct trials for filament production, specifically because their equipment configuration is very compact and could readily be installed in a single 20-foot ISO Container for FOB applications. After resolving some equipment and processing issues, the trials at Hills were successful in producing both the 1.75 and 2.85mm AM filament from rPET pellets with an intrinsic viscosity (IV) of 0.7. Figure 12 below shows the results of these trials and the AM filament being spun into a spools.

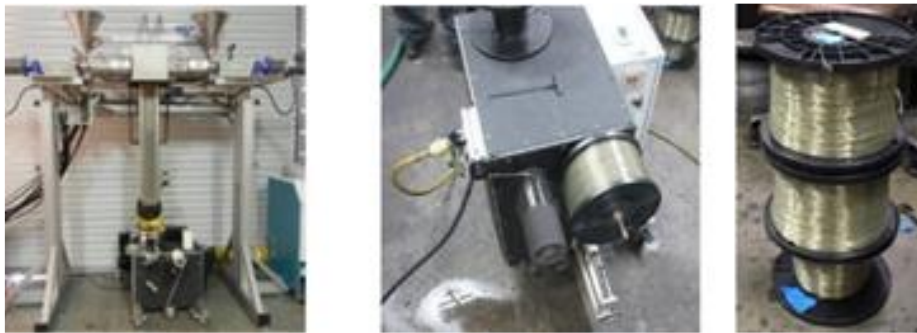


Figure 12. Compact lab equipment used for fiber spinning 3D printer filament

After being dried per the specifications (250 F for a minimum of 4 hours or equivalent), the AM filaments from fiber spinning were used with several printers at Emc² and ERDC-CERL. *While simple calibration items could be printed successfully, it was found that the consistency and ovality of the filament varied beyond the tolerance specifications of the 3D printers.* One frequent problem involved ‘clogging’ of the printer head due to excess ovality in the filament. Therefore, a second set of trials were conducted at Hills to investigate if filament ovality could be controlled to the tolerance required. These trials indicated that Hills’ equipment and technology is ideally suited

for a much finer (smaller diameter) textile grade fiber as ovality requirements are not stringent. However, controlling the ovality of larger diameter (1.75 mm and 2.85 mm) AM filament to the required specifications was not very reliable. After further discussion with Hills, the fiber spinning technology was not deemed feasible for FOB applications and was therefore not pursued further.

Filament Production Trials with Conventional Extrusion: The next set of trials conducted involved investigating conventional extrusion technologies used commercially for AM filament. The goal was to determine if the system can be modified for installation and operation in one or possibly two 20-foot ISO containers for FOB applications. These trials were conducted successfully at three separate production / lab facilities as summarized below.

FilaMint, Chesterland, OH: After several preliminary discussions and at the recommendation of IC3D, a local manufacturer of commercial 3D filament in Central Ohio, FilaMint was selected for the initial trials. FilaMint is a small company with equipment that can easily be re-configured to suit rPET filament manufacturing. Emc² staff worked closely with FilaMint to conduct several trials modifying the extrusion conditions and processing parameters as required for rPET materials.

The results from the FilaMint trials and their reports and recommendations for some *unique process settings required for rPET* are included in Appendix A-5. Both 1.75mm and 2.85 mm filaments were successfully made using not only the rPET pellets used previously, but also clean 4-mm rPET flakes. This latter result is a major breakthrough in rPET filament extrusion technology as it eliminates the need for pelletizing the flake prior to filament production. Based on the success of these trials at FilaMINT, a complete 3D filament extrusion plant was designed, The design allows for shipping the system in a single 20-foot ISO container and then operated at PON with an additional 20-foot container. Figure 13 shows the various pieces of equipment and their configuration for this mobile filament extrusion line for FOBs.

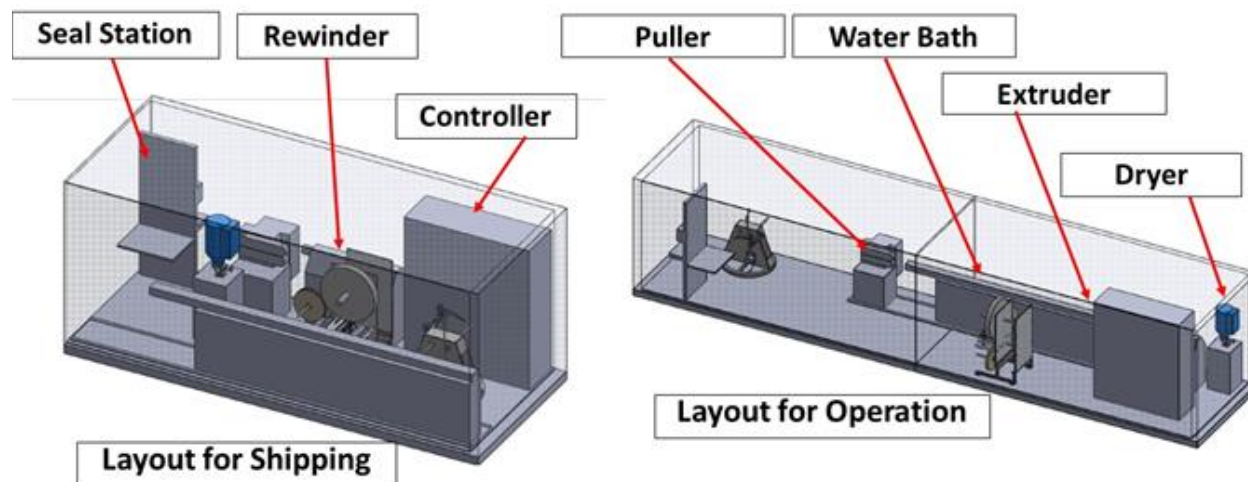


Figure 13. Mobile 3D printer filament extrusion line in 20-foot ISO containers

Conair, Pinconning, MI: Conair is one of the premiere suppliers of plastics processing equipment in the USA. They are the best known producer of complete filament extrusion lines for the 3D printing industry. Based on the success of the above trials at FilaMint, a second trial was scheduled at Conair to determine if any major modifications to the system design and shown in Figure 13

was necessary for FOB applications. Trials were therefore conducted at Conair using both rPET pellets and clean 4-mm flake to make 2.85 mm diameter filament, see Figure 14 below. These trials were coordinated under Emc² staff supervision using *the exact same unique processing conditions that were deemed optimal for rPET* based on the earlier effort at FilaMint described above.



Figure 14. Photograph of rPET 3D printer filament extrusion trials at Conair

The trials at Conair established that rPET flake could successfully be used to meet commercial grade filament with < 0.05% ovality at a rate that exceeded 50 feet per minute. The test report along with Conair's commercial brochure for 3D filament production is provided in Appendix A-6.

3.3.2 Subtask 3.2 Final trials for rPET filament extrusion

The final trials to confirm that rPET flake could be successfully converted into AM filament were conducted at IC3D, Hilliard, OH under supervision of Emc² staff. At the 2021 IPR meeting, one of the recommendations made was to investigate the use of additives to enhance the material properties of rPET parts. Specifically, since rPET items can have very low ductility, additive packages could enhance the elongation to failure and also the impact strength. Therefore, a third set of 2.85 mm 3D printer filament extrusion trials were undertaken at IC3D, a commercial filament manufacturer for 3D printers. The primary objective of this trial was to repeat the production of rPET filament from clean flake from the FilaMint and CONia and also produce filament with a 10% by weight Kraton (FG1924) additive package. The choice of additive and the percent by weight loading was based on earlier trials conducted in a separate activity in the project involving rPET based plastic lumber extrusion.

The IC3D trials were again very successful in making 2.75 mm filament both with and without the additive, see Figure 15. *It was noted that the additive package enhanced both the processability, printability and the surface finish of the final part along with enhancing the ductility by 46%, as*

discussed in Task 4. Photos of filament spools with and without the additive are shown in Figure 16 below. The complete report from the IC3D trials is included in Appendix A-7.

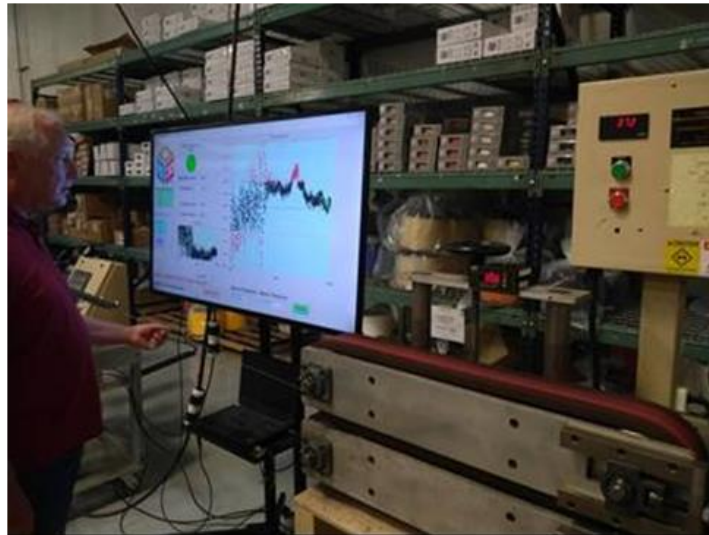


Figure 15. Photo of IC3D trials to make 3D filament on commercial equipment



Figure 16. rPET filament spools with (opaque) and without (translucent) additive

3.3.3 Subtask 3.3 Configuration of rPET filament facility at FOB

Based on the above trials, it was concluded that the preliminary design for the filament extrusion line, shown in Figure 13 above would be the most optimal option for FOB operation of mobile manufacturing for AM applications. Complete details of this design of this mobile filament extrusion system with equipment specifications and budgetary costs are provided in Appendix A-8.

3.3.4 Subtask 3.7 Processing and evaluation of rPET based plastic lumber and shapes

Background: Plastic lumber as a potential product for agile manufacturing at FOBs was selected for further evaluation as it would (i) consume much larger quantities of the waste rPET than AM, (ii) it is relatively tolerant process with regard to contamination in the PET waste stream, and (iii) provides much needed construction material for light outdoor structural applications. All three grades of rPET procured from Phoenix were used in these trials: rPET pellets with an IV=0.7 rPET white flake (from water bottles), and rPET green flake (from soda bottles).

Experimental Trials with rPET: Plastic lumber is manufactured generally using waste and recycled resins by two main processes: continuous profile extrusion, and flow molding⁶. In flow molding a closed cavity in the shape of the desired lumber (e.g. 2"x4", 4"x4" etc.) is filled with plastics melt at low pressure by an extruder. When the cavity is filled it is allowed to cool either in air or in a water bath. The formed lumber then shrinks during the cooling process and therefore is relatively easily removed from the mold cavity. This process can be manual with the molds being removed and placed by hand or automated using multiple molds on a turret⁷.

The first step in this experimental effort involved evaluating the processing conditions and their effect on the properties of the rPET formulations for lumber. Four small 1"x1"x36" stainless steel molds were made to minimize the amount of material needed to determine and optimize these processing conditions. Two of these molds are shown in Figure 17 and have a volume of 590 cc and result in a part that weighs approximately 800 gms.



Figure 17. Molds for plastic lumber test specimens (1" x 1" x 36")

As is well established, rPET is a hygroscopic material and needs to be dried prior to processing to prevent degradation during the extrusion process. All grades for rPET used in these trials were dried prior to extrusion as follows using a Matsui Dehumidifying Hopper Dryer DMZ-80 at 130 °C for 12-hrs minimum.

⁶ <http://www.globalre-energy.com/flow-molding-process/>

⁷ http://americancierra.com/Recycling_Lines.php?Line=1

The extrusion equipment used was a Leistritz 34-mm co-rotating twin screw, L/D ~25:1, and the conditions are provided below in Table 3.

Table 3. Extrusion conditions used for rPET plastic lumber

PET Feed Rate		Estimated Fill Time	Extruder Speed	Temp Profile
(g/min)	(lb/min)	(min)	(rpm)	deg C
346	0.76	2.4	200	270 (Flat)

Using these basic conditions all three rPET materials: pellets, white flake and green flake were used to make lumber test specimens by water cooling and/or air cooling. Figure 18 shows some representative test specimens. A complete test report on these trials with details is included in Appendix A-9.



Figure 18. rPET plastic lumber (1" x 1") test specimens with white flake

Results: The test specimens made from all three types of rPET were tested using the procedures found in ASTM D6109⁸. For comparison, samples of representative commercially available high-density polyethylene (HDPE) plastic lumber and pine wood were also tested. The flexural testing was conducted on a 20 kip (98 kN) servo hydraulic Universal Testing Frame (UTF) controlled with an MTS digital system was used to perform the test on each of the specimens. The test was run using a four-point bend fixture with two Linear Variable Differential Transformer sensors (LVDTs). The fixture was set with an outer span of 16-inches and an inner span of 5.3-inches (one-third of outer span). The LVDTs were set at the center span of the fixture to measure center line displacement. Figure 19 below shows the experimental set up for the four-point flexural test.

⁸ ASTM D6109 – 19, *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastic Lumber and Related Product*, 2019.



Figure 19. Four-point bend fixture for testing plastic lumber

Table 4 shows the samples tested and their designations. The rPET pellet material was both water cooled and cooled with a combination of initial air cooling for 15 minutes followed by water cooling. All the other rPET samples were water cooled for approximately 10 minutes before demolding.

Table 4. rPET Specimens and Cooling Type

Specimen ID	Material	Cooling Type
B1T	White Pellet	Air/Water
B2T	White Pellet	Water
White	White Flake	Water
Green	Green Flake	Water

As indicated above comparison specimens with commercially available Pine Wood and HDPE based plastic lumber were also used for the flexural testing. Figure 20 below shows the stress-strain curves for the flexural testing of the four rPET materials at Emc²'s laboratory, and the comparison samples of standard polyethylene lumber and pine wood along with the loading diagram per ASTM D6109⁸ in the inset. The data is shown up to 3% strain, since this is the limit specified in this test method. Several samples including rPET pellets cooled in air/water (B1T), rPET White Flake, and wood all failed before 3% strain. The best performing rPET in this test was the green flake sample followed by the pellet sample cooled only in water (B2T). All the rPET samples flexural strength at either 3% strain or failure is significantly higher than the polyethylene specimen, on average about 5 times higher (2175 psi vs. 10870 psi at 3% strain).

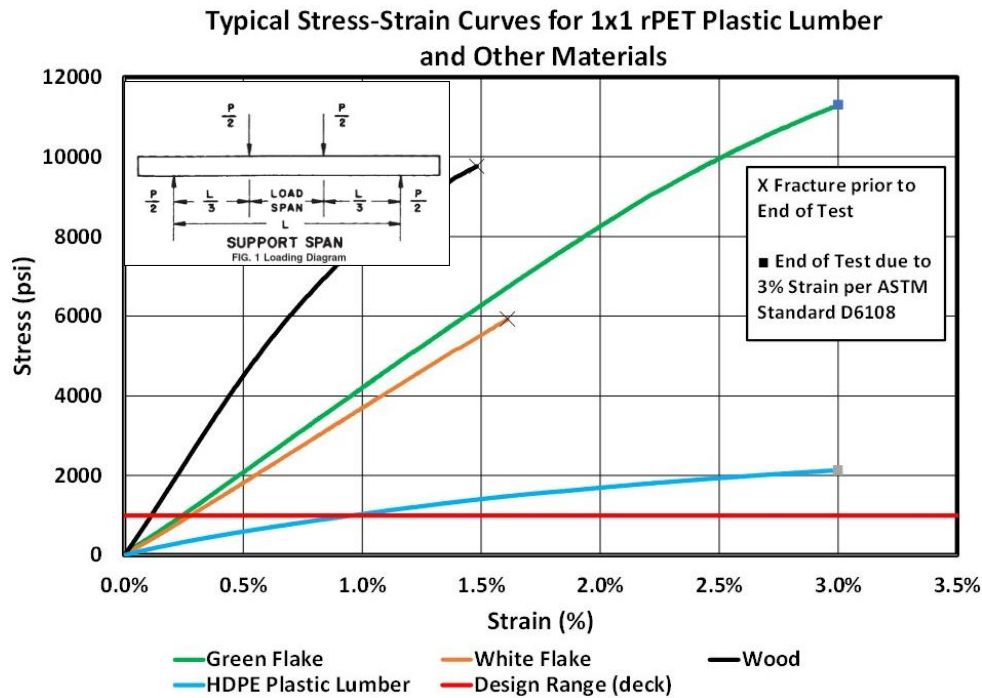


Figure 20. Stress-Strain Curves for rPET and other plastic lumber test specimens

The modulus or stiffness of the rPET, which is the initial slope of the stress strain curves is significantly higher than the polyethylene specimens although not as high as the wood specimen. One important observation was that the failure mode for all the rPET samples was observed to be brittle failure that occurred with fragmentation of specimen.

Additional evaluation of the rPET plastic lumber samples involved fastener withdrawal testing per ASTM D6117⁹. These set of tests were indicated that screw withdrawal forces exceed 1000 lbs in all the specimens tested, that is it well exceeded the minimum values required to meet plastic lumber specifications in ASTM D6662¹⁰.

Use of additives to enhance rPET properties: Given the brittle failure observed during the flexural tests (although they occurred well above the useful design range for plastic lumber), a recommendation was made by the technical committee to investigate the use of additives to enhance properties of rPET. Therefore, additive technologies were investigated to improve ductility and strain to failure of rPET based plastic lumber formulations.

Two types of additives were used to modify the rPET green flake - Chain Extenders, and, Impact Modifiers. Chain extenders are meant to increase molecular weight of the rPET that can be reduced via hydrolysis during previous melt processing excursions. Additives of this type contain groups that are reactive to terminal hydroxyl or acid groups that would be present on hydrolyzed polyesters. One of the most common chain extenders used for PET is pyromellitic dianhydride

⁹ ASTM D6117, *Standard Test Methods for Mechanical Fasteners in Plastic Lumber and Shapes*, 2018.

¹⁰ ASTM D6662, *Standard Specification for Polyolefin-Based Plastic Lumber Decking Boards*, 2022.

(PMDA)¹¹, which was sourced from Sigma Aldrich. Impact modifiers are typically elastomers that are mixed into the PET and form a phase dispersed morphology the dispersed elastomer phase functions to interrupt microcrack growth resulting in improved ductility and toughness. Three types of impact modifiers were evaluated. All of these impact modifiers have some structural moiety in their composition that is capable of interacting with the rPET polymer. The Kraton materials both were grafted with maleic anhydride which could react with hydroxyl end groups on the rPET and the Lotader material is a terpolymer of ethylene–ethyl acrylate–glycidyl methacrylate, where the glycidyl group could also interact with hydroxy end groups. The Kraton material differed in the amount of maleic acid functionalization. The functional additives were hand mixed in the formulations at various levels shown in Table 5.

Table 5. Additives used in rPET plastic lumber formulations

Material	Source/grade	Addition Levels
Pyromellitic Dianhydride (PMDA)	Millipore/Sigma Aldrich Chemical	0.15%
SEBS-MA	Kraton FG1901	10%
SEBS-MA	Kraton FG1924	5 and 10%
E-EA-GMA	Lotader AX8900B	5 and 10 %

Using the same basic processing conditions as done previously, these rPET green flake formulations with additives were made into test specimens of four replicates each. Figure 21 shows some representative test specimens.



Figure 21. rPET plastic lumber (1" x 1") test specimens with green flake

The addition of either the Kraton FG 1924 or Lotader AX8900B at 10% resulted in a more ductile failure without fragmentation showing that the impact modifiers at this level result in a significantly improved product. The stress/strain curves from ASTM D6109⁸ tests conducted during this project at Emc²'s laboratory is shown in Figure 22 for the four replicates of the 10% Kraton FG1924 composition shows that the curve flatten indicating a more ductile failure mode

¹¹ Scheirs, J. (2004). *Additives for the Modification of Poly(Ethylene Terephthalate) to Produce Engineering-Grade Polymers*. In *Modern Polyesters: Chemistry and Technology of Polyesters and Copolyesters* (eds J. Scheirs, J. Scheirs and T.E. Long).

with strain at failure of 0.05 (5%) which far exceeds the strain of 3% observed earlier (Note: the one specimen that failed at low strain is most likely due to an external defect)

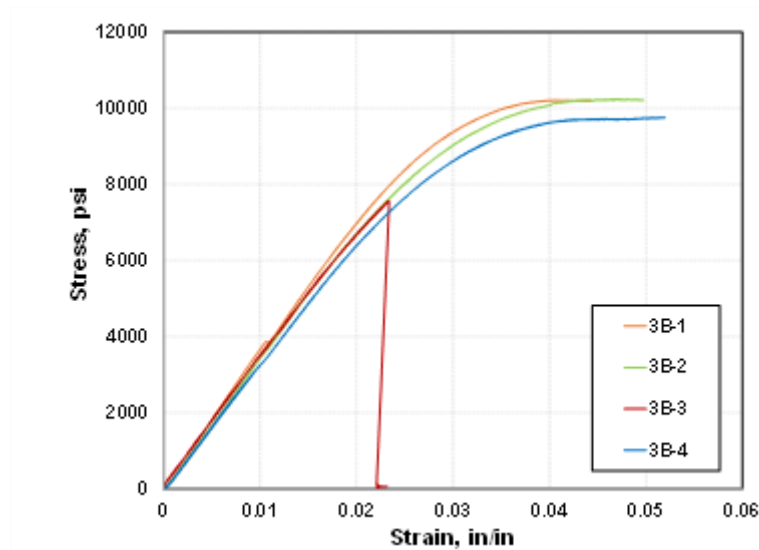


Figure 22. Stress-Strain curves plastic lumber sample for rPET with 10% Kraton

Design of mobile rPET plastic lumber manufacturing system: Based on the success of the above trials, an equipment fabricator that specializes in flow-molding systems for plastic lumber manufacture, American Cierra, Auburn, NY was contacted. After extensive discussions about the scale, mobility, and modularity of the system required for FOBs, a complete manufacturing system in two 20-foot ISO ‘high’ containers was designed as shown in Figure 23. Appendix A-10 provides all the details of the equipment specifications as well as budgetary costs for this flow molding system (Note: some information from vendors on this system is proprietary and is for federal government agency use only.)

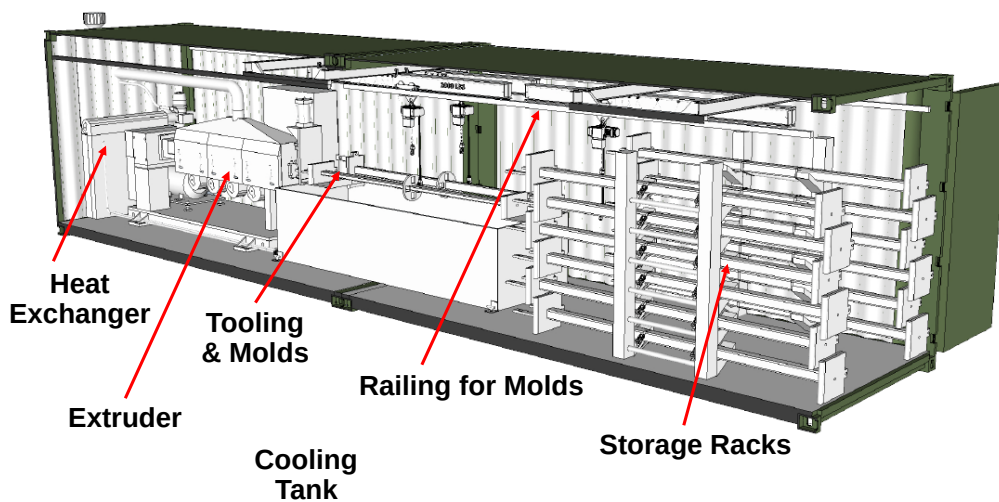


Figure 23. Schematic of the mobile manufacturing system for rPET plastic lumber

To further confirm that the equipment selected in the above design would work for rPET, one of the commercial manufacturers with a similar system, EcoProducts, Wilmington, DE was contracted to produce larger 2x4x8-ft long dimensional lumber specimens with the same rPET formulations successfully, see Figure 24. This confirmed that the mobile manufacturing system would work reliably as designed.



Figure 24. 2x4 rPET lumber produced with commercial flow molding equipment

3.3.5 Subtask 3.8 Processing and evaluation of rPET based ByBlocks

In addition to rPET based plastic lumber one other commercially available technology that was evaluated for FOB applications involved the production of construction type ByBlocks from ByFusion of Gardena, CA. This is especially relevant to address the large volume of both waste PET (up to 0.5 lbs/person/day) at contingency bases but also the fact that other technologies are needed to address mixed plastics waste.

The ByFusion ‘Blocker’ machine produces ByBlock from recycled plastics, including those normally considered not recyclable. ByBlocks have an overall rectangular prismatic shape with nominal exterior dimensions of 16 x 8 x 8 in. (40 x 20 x 20 cm), similar to plain-end Concrete Masonry Units (CMU) as shown in Figure 25.



Figure 25. Photograph of typical ByBlock made with mixed plastics waste

The final shape of ByBlock may include a boss and pocket for self-loading walls and/or through-hole bores for reinforcing bars, threaded rods, or cables. ByBlocks weigh approximately 22 lbs. (10 kg), roughly 30% less than typical CMU of equivalent size and design. Based on this claimed weight, each block would require approximately 800 to 1,200 empty 16.9 oz. (500 mL) water bottles, which would have contained about 130 gallons or 1,000 lbs. of water. This calculation does not consider binding agents (like low melting polymers PE or PP) that might be added to ByBlock during production. Appendix A-11 provides detailed information about this technology from ByFusion including demonstration projects conducted by them at various sites.

As part of this effort three activities were undertaken to evaluate the relevance of ByBlocks as described below:

- Determine the compressive structural properties of ByBlocks, the primary mode in which they are loaded during use,
- Construct a small ‘storage shed’ at ERDC-CERL to assess ease of use in light structural applications outdoors, and
- Modify the design of ByFusion’s existing manufacturing system to a more agile, modular units in 20-foot ISO Containers.

Evaluation of ByBlock compressive properties: While commercially available ByBlocks are made with a range of mixed plastics waste, of specific interest in this effort is the use of rPET materials in this product. Hence, ByFusion was contracted to produce sample ByBlocks with two levels of waste PET 40% and 75% by weight and produce three replicate samples for each of the formulation shown in Table 6 below. Each of these specimens was tested in compression at Emc²’s structural testing laboratory using the entire ByBlock, i.e. a full-scale specimen test as shown in Figure 26

Table 6. Formulations for rPET ByBlocks used in Evaluation

Specimen ID	rPET	HDPE	Mixed Plastics
ByBlock Formulation 1	75%	25%	0%
ByBlock Formulation 2	40%	25%	35%



Figure 26. ByBlock samples under compression testing

During the test, load, load point deflection, and compression strain for the ByBlock was measured to determine maximum load carrying capacity, mode of failure and compressive stress-strain behavior. Table 7 below shows the summary of critical compressive properties for the two formulations shown in Table 6.

Table 7. Compression properties for ByBlock samples with formulations in Table 6

Specimen ID	Max Load	Max Stress	Max Strain	Load at 3% Strain
Formulation	kips	psi	%	kips
ByBlock Formulation 1	41.9	360	23.40%	5.67
ByBlock Formulation 2	46.4	395	24%	5.02

The most significant result from this testing was that in each case the maximum load carrying capacity of the ByBlock in compression exceeded 40,000 lbs (40 kips) without catastrophic or brittle failure. The compressive strain approached 23% at a maximum stress which was under 400 psi, that is the compressive modulus for ByBlocks is relatively low as would be expected of plastics. Appendix A-12 has the experimental test report. These results confirmed that ByBlocks could be considered for use at FOBs for light, outdoor construction applications as is recommended by the vendor.

Evaluation of building structures with ByBlocks: The next step in the evaluation of ByBlock technology involved building an actual structure on site by US Army Corps personnel at ERDC-CERL. With the help of ByFusion a small storage shed (outer dimensions of 143" x 120" x 114" high) was designed, see Figure 27. This storage shed was completed successfully and was moved outdoors for use at CERL as shown in Figure 28.



Figure 27. Design of storage shed for building at ERDC-CERL



Figure 28. Photo of storage shed under construction and completed at ERDC-CERL

Appendix A-13 includes the detailed construction guide for this structure provided by ByFusion, a report on ByBlock technology by CERL, and their final evaluation in a ‘Lessons Learned’ and recommendations from their evaluation. Also included in Appendix A-13 is the ERDC-CERL report on estimates of rPET available at CBs. Most of these recommendations are minor and can readily be addressed as may be noted by ByFusion’s responses, which are also included in Appendix A-13. In summary the ERDC-CERL effort established conclusively that ByBlocks made from mixed waste plastics could be used on site for many light, outdoor structural applications at FOBs. (Note: Some information in Appendix-13 is considered proprietary by the vendor and is therefore marked as such and may need to be restricted for use outside of federal government agencies.)

Modification of ByFusion system into 20-foot ISO Containers: The existing manufacturing system for ByBlocks is proprietary to ByFusion and normally consists of a mobile unit designed to operation in a single 40-foot container. However, to address the goals of this project, that is, to design agile manufacturing with wasted plastics, ByFusion under Emc²’s coordination modified their design into two 20-foot containers. A rendering of this agile ByBlock manufacturing plant is shown in Figure 29.



Figure 29. Schematic of ByBlock manufacturing plant in two 20-foot ISO containers

The report from ByFusion on this design modification is included in Appendix A-14 and does contain information that is proprietary and confidential information that is restricted only to federal agency uses.

3.4 TASK 4 - EVALUATION OF RPET PRODUCTS

The subtasks conducted under Task 4 are as follows:

- Subtask 4.1 Evaluation of rPET material properties
- Subtask 4.2 Interim report that provides a description of rPET material properties
- Subtask 4.3 Design guide for rPET 3D printers
- Subtask 4.4 Evaluate rPET strapping properties (OBE)
- Subtask 4.5 Design guide for rPET strapping for packaging (OBE)

The activities described below are primarily related to Subtask 4.1 and 4.3 as Subtask 4.2 was included at the request of the SERDP Technical Committee as an ‘interim’ deliverable. As previously explained, Subtasks 4.4 and 4.5 were ‘Overcome by Events’.

3.4.1 Subtask 4.1 Evaluation of rPET material properties

This subtask and the corresponding work in developing both rPET based formulations in Task 2 above and design of the mobile filament manufacturing technology in Task 3 are interlinked and were carried out simultaneously as the results are interdependent affecting the other tasks directly. The material properties for rPET based formulations were therefore evaluated at several stages of this research project and adjustments were made to the formulations based on the feasibility of filament processing and AM with the filament specimens. Both the interim and final reports on this effort are provided in Appendix A-15 for completeness and the key results summarized below. This material property characterization is significantly important for AM applications as it requires the cleanest feedstock with consistent processing properties for manufacturing of products at PON.

As detailed under Task 2 (see Table 2 and Figure 10), four grades of rPET were used in developing formulations and material properties for AM: two types of rPET pellets rPET-1 and rPET-2 which were clean and melt-filtered with IV = 0.70 and 0.75 respectively, a clean 4 mm white flake (water bottles), and green flake (soda bottles). The key material properties from this subtask are described below.

Thermogravimetric Analysis (TGA): The first material property characterized was a complete Thermogravimetric Analysis (TGA) of both rPET-1 and rPET-2 pellets to determine both the processing conditions limits and degradation temperatures for the resin. Appendix A-15 provides the TGA curves for all specimens, and, as may be noted in Figure 30 below both grades of resin are identical indicating that the various grades of rPET would be expected to have properties in the same range.

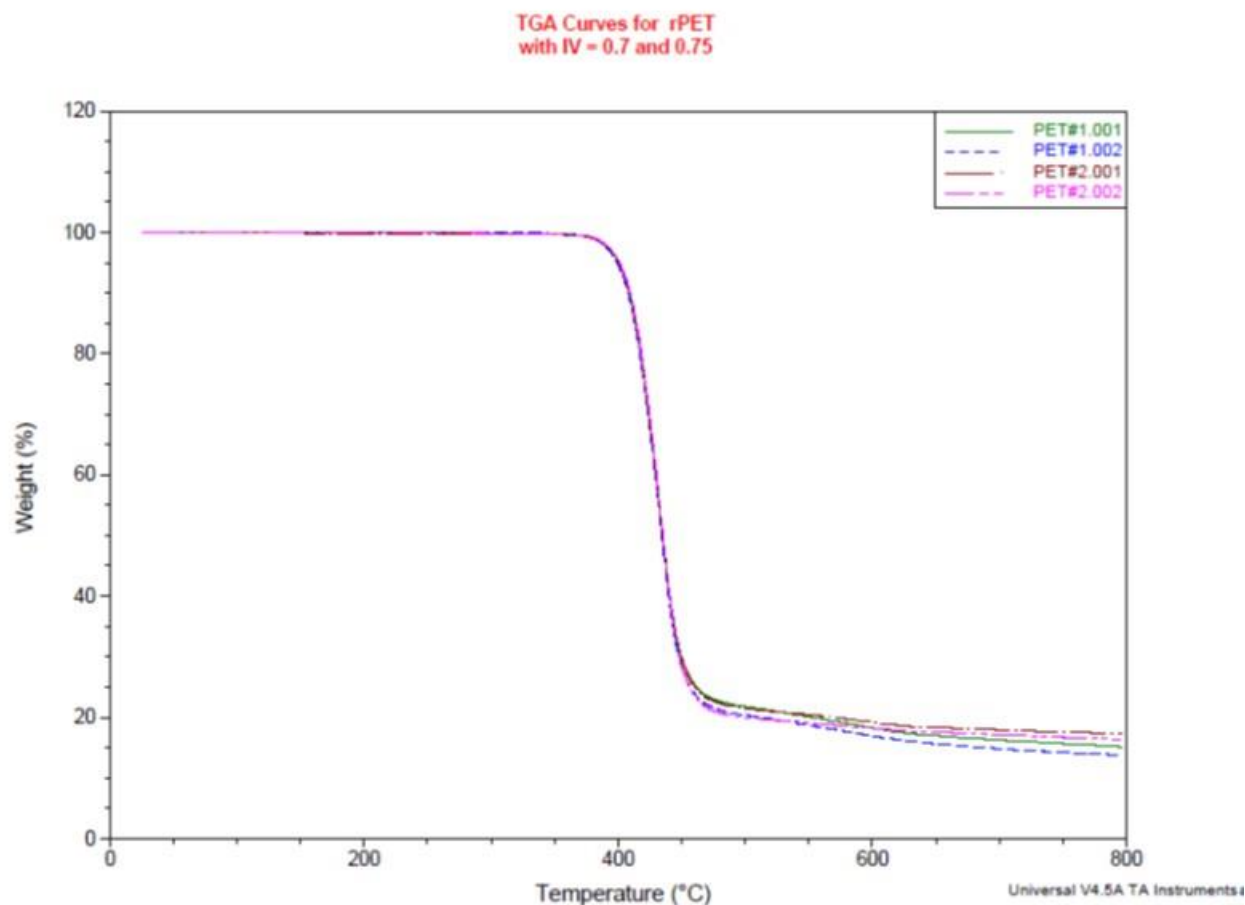


Figure 30. TGA Curves for rPET-1 and rPET-2 pellet samples (two replicates each)

Physical and Mechanical Properties: The next step involved evaluating standard physical, mechanical and thermal properties using the relevant ASTM test method]. Since one of the goals was to determine if rPET could be used to make filament for 3D printers, both injection molded and 3D printed (from rPET filaments made as described under Task 3) ASTM specimens were used to determine material properties. Two sets of ASTM specimens were molded using the rPET materials – one at GDC in Goshen, IN and the other at The Ohio State University’s (OSU) Center for Design and Manufacturing Excellence (CDME), OH. Appendix A-15 provides the injection molding conditions used to make standard ASTM specimens. Also provided in Appendix A-15 are the processing conditions and settings used to make the ASTM specimens on a Lulzbot Pro dual filament printer. The properties evaluated, the ASTM test methods used as well as the mean values for each case are summarized below in Table 8. All tests were conducted by an A2LA certified laboratory at Ineos of Dublin OH. The detailed reports from the ASTM tests from the laboratories are provided in Appendix A-15 some of which is proprietary and is for federal government use only.

Table 8. Material Properties for Injection Molded and 3D Printed Specimens

Property	ASTM Method	rPET Injection	rPET- 3D Printer	Difference. %
Laboratory		Ineos/ GDC	Ineos	
Processing Method		Inj Molded	Lulzbot Pro	
Specific Gravity	D792	1.333	1.262	-5.33%
Tensile Strength, psi	D638	7794	4089	-47.54%
Tensile Modulus, ksi	D638	342	259	-24.27%
Tensile Elongation, %	D638	3.44	1.77	-48.55%
Heat Deflection Temperature, F	D648	139.46	150.26	7.74%
Flexural Strength, psi	D790	11402	5031	-55.88%
Flexural Modulus, ksi	D791	352.5	146.2	-58.52%
Flexural Strain at Yield,in/in	D792	0.0441	0.0606	37.41%
Hardness, Shore D	D2240	77	50.7	-34.16%
Izod impact - Notched, ft-lbs/in	D256	0.64	0.5	-21.88%

The major conclusions from the above material property evaluation is that the 3D printed specimens have lower mechanical properties (tensile, flexure, hardness and Izod) compared to the injection molded parts. This is not unexpected as the packing density of the material from injection molding is higher than that during 3D printing which is reflected in the lower specific gravity of the 3D printed parts as well. However, the properties of the 3D printed specimens are still well within the range of those required for printing parts as described below.

Effect of in-fill and comparison to engineering plastics: The next step in evaluating material properties of rPET for AM was to assess the effect of in-fill percent (%). The lower the infill level the lighter the part and shorter the time for manufacturing. However lower infill levels do affect properties. Flexural and tensile properties determined from ASTM specimens printed at 100% and 50% infill are shown in Figure 31. Also shown are properties for ABS, a very commonly used engineering plastic resin used extensively in both non-structural and structural parts.

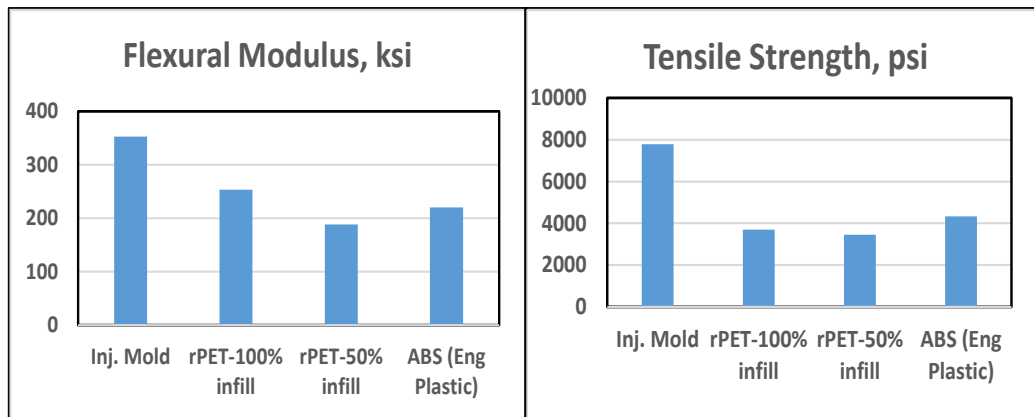


Figure 31. Comparison of flexural and tensile properties of 3D printed rPET materials

As may be seen in Figure 31, the properties decrease with the lower 50% infill. But the key finding is that *properties of 3D printed rPET compare very favorably with ABS, indicating that for many*

applications at FOBs, rPET could be a direct one-for-one replacement for parts originally made by injection molding with engineering grade resins such as ABS.

Effect of additives on rPET material properties: The final evaluation involved determining if rPET material formulations could be modified to enhance properties. Based on the effect of additives plastic lumber properties discussed in Task 3 rPET flake with 10% Kraton was used to manufacture 2.85 mm AM filament as shown in Figure 16 above in Task 3. These material property data along are shown below in Table 9 along with those for other standard materials used for AM, it. PLA, PETG, and ABS. As highlighted in Table 1, and shown in Figure 32 the tensile elongation value (ductility) of the rPET increases significantly with the use of 10% Kraton (FG1924), from 1.82% to 2.66% , that is an increase of 46%. This would significantly reduce the ‘brittleness’ of rPET materials making it much more effective for use in printing at FOBs. The tensile elongation property is also shown in Figure 32 below along with those for injection molded rPET specimens. Also seen is the fact that the ductility of rPET with 10% Kraton is directly comparable to other engineering resins such as ABS, PETG, and PLA.

Table 9. Properties of 3D printed rPET compared to other AM materials

Property	ASTM Test		Units	rPET- without Kraton	rPET-with 10% Kraton	PLA	PETG	ABS
Sp. Gravity	D792			1.149	1.344	1.177	1.247	1.006
Flexure	D790	Strength	psi	8334	7758	12616	9436	6806
	D790	Modulus	ksi	253	254	461	277	220
	D790	Yield Strain	in/in	0.054	0.0454	0.047	0.053	0.054
Tensile	D638	Strength	psi	3697	3065	5824	5958	4336
	D638	Modulus	ksi	230	164.2	401	276	262
	D638	Elongation	%	1.820	2.66	2.160	2.930	2.160

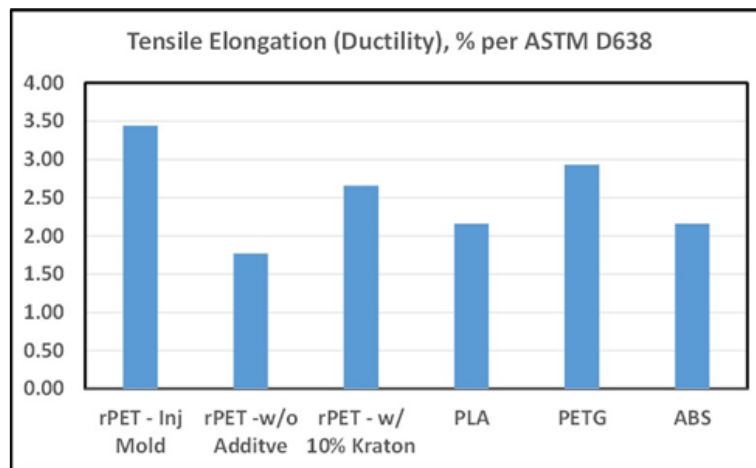


Figure 32. Tensile Elongation (ductility) of rPET and other materials used for AM

3.4.2 Subtask 4.3 Design guide for rPET 3D printers

The effort in this subtask was to compile a design guide for use of rPET in AM based on the results and analysis presented Tasks 2 and 3 along with the above results. This ‘Design Guide for rPET 3D Filament in Printers’ was submitted as a separate deliverable and is included in Appendix A-16. *The major conclusion from this subtask, as detailed in the design guide is that the mobile filament extrusion system shown in Figure 13 above would work successfully at FOBs for the rPET flake generated with the mobile ‘Sorting and Washing Line’ shown in Figure 8 (or Figure 9, both with or without additives. And, that the properties of the parts produced with AM using any of the commercially available printers would be acceptable for replacement parts for PON manufacturing as described next.*

Design considerations for rPET in 3D printers: To confirm the recommendation from the above design guide printing of sample parts especially representative of and relevant to FOBs was undertaken with rPET filaments printers at several laboratories. Besides the ASTM specimens printed for material property evaluation, several sample calibration parts were also printed as shown in Figure 33. This includes successfully printing a ‘radio bracket’ using the drawing files provided by ARL staff, a 90-degree gear transmission example for assessing precision and tolerance, and other common parts used for confirming printer parameters.

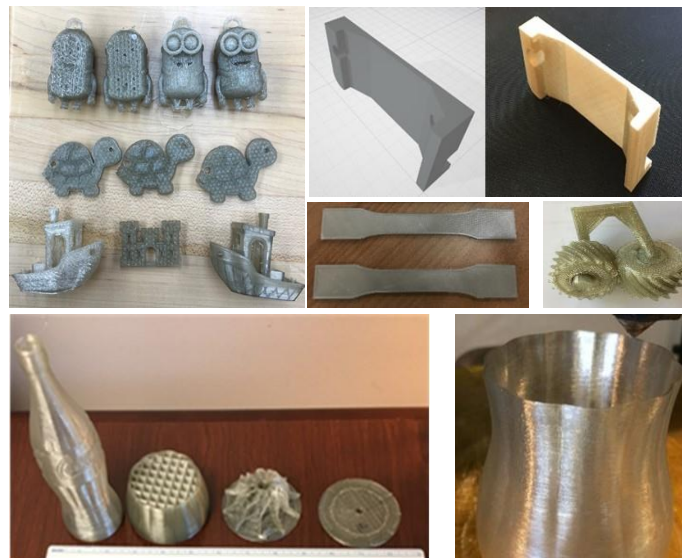


Figure 33. Calibration test specimens and components printed with rPET filament

To further establish the feasibility of using rPET filament for engineering components, several drum wrench, or ‘bung wrench’ designs (for 55-gallon drum opening) were printed. Figure 34 shows two of these wrench designs that were printed successfully with rPET filament without (translucent) and with the 10% Kraton (FG 1924) additive package (opaque). Both designs worked successfully and had sufficient strength to provide the torque required to open and close standard drum caps. Additionally, ERDC-CERL printed some components from the DoD database

JAMMEX¹² in their laboratory. One such part as it was being printed is shown in Figure 35. However, this finished print, as well as the other components printed with the rPET filaments are sensitive and therefore available, if needed, directly from ERDC-CERL staff who were the DFPs on this project.



Figure 34. Drum wrench printed with both rPET filament with and without additive

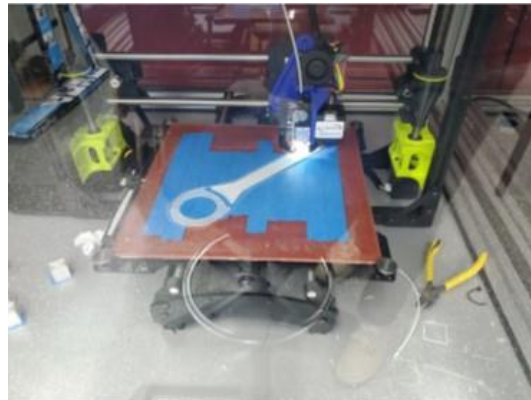


Figure 35. Photo of part from DoD's JAMMEX database during print

AM with rPET pellets and flake: One unique breakthrough AM technology developed recently involves printing directly from plastic resin pellets, without the need to produce filament. After extensive discussions with the project team, the Army Corps ERDC-CERL staff, and ARL, two such technologies were evaluated since they were considered high priority and be significant for mobile manufacturing systems at FOBs. *Specifically, if proven feasible, the rPET clean flake produced with the 'Sorting & Washing' systems described in Task 1(Figure 8 and Figure 9) could directly be used to print components at PON.* This evaluation was undertaken towards the end of the project performance period and is briefly summarized next.

Titan 3D Printer Technology: Titan, Colorado Springs, CO, is one of the US companies that has commercially available AM equipment to print large components from pellets. Sample rPET

¹²https://www.army.mil/article/260930/army_logistics_modernization_program_enhances_advanced_manufacturing_for_all_services

pellets (from Table 1) were sent to Titan to run some preliminary trials. Figure 36 shows the model of the Titan printer used for the trials as well as the ‘calibration’ sample of a 12”x12”x12” hollow box printed. Due to the proprietary nature of their printer design, modification to the equipment to also handle rPET flake in addition to pellets and address feeding and fluctuations in melt flow rate could not be addressed - and therefore not pursued further. However, several Titan printers have apparently been procured by DoD laboratories, and additional research trials and equipment modifications may be pursued directly by these entities with assistance from Titan.



Figure 36. Photo of Titan printer and hollow box specimen printed with rPET pellets

Evaluation of RE3D 3D Printer Technology for FOBs: Emc² was introduced to RE3D by ARL staff, since this AM technology also involved printing directly from resin pellets, and possibly plastic flakes as well. Several trials were conducted at RE3D with the Gigabot-X model, shown in Figure 37 below. Preliminary results with rPET pellets and flakes showed limited success as seen by the photos of the 4-inch calibration cylinder and the company ‘logo’ printed in Figure 38.

Additional efforts to use rPET flake to print and evaluate properties from standard ASTM specimens showed significant inconsistency. The results indicated that significant modifications to the printer as well as processing conditions are needed and was beyond the original scope of the project and not pursued further.



Figure 37. Photo of Gigabot X printer from RE:3D



Figure 38. Calibration cylinder and ‘logo’ printed on Gigabot-X with rPET flake

However, RE3D, being an ‘open-source’ AM technology, is willing to work with Emc² to modify their printer configuration and optimize processing conditions with rPET flake. This follow-on effort is being undertaken separately as discussed in Section 5.0 below.

3.5 TASK 5 - PROGRAM COORDINATION, REPORTS AND DELIVERABLES

The final Task 5 in this effort involved program management, monthly and quarterly progress reports, and submission of deliverables in the various tasks, including presentation at IPR meetings and SERDP webinar. As part of this task a number of publications and presentations on this project were made at national and international conferences and published in their proceedings. A complete list of these publications and talks are included in Appendix B.

4.0 SUMMARY OF RESULTS

The major results from this project involve the following four complete designs for mobile and agile manufacturing plants that can be fabricated and built in the continental U.S. (CONUS) in 20-foot International Standard Organization (ISO) containers, shipped to the required bases and operated on site by the servicemember.

- i. A Sorting and washing plant that converts waste PET water bottles with or without label into clean, dried, 4-mm rPET flake at a rate of 300 pounds/hour which serves as a consistent feedstock for a number of products that can be manufactured at PON at CBs. This plant is designed to be fabricated, shipped and operated in three (3) 20-foot ISO containers. A smaller version of this mobile plant was also designed in just one (1) 20-foot container using only PET water bottles without label, and produce sufficient quantities of flake needed for AM applications only.
- ii. A Filament extrusion plant that was designed to convert the rPET flake from the Sorting and Washing plant into high-valued 3D printed, much-needed components at PON. The rPET filament in standard 1.75 or 2.85 mm diameter could work with any commercially available printer and can be used to fabricate parts to replace components made with other engineering grade resins such as ABS. Recommendations for an additive package involving Kraton has been developed to further enhance the properties of the filament and the final components printed.
- iii. An rPET based dimensional plastic lumber flow-molding plant is designed to convert larger amounts of rPET flakes produced by the Sorting & Washing line into dimensional plastic lumber (1x1, 2x4 etc.). This plant is designed to operate in two (2) 20-foot ISO container and use 200 pounds/hour of the rPET, and can tolerate higher levels of contaminants than the requirements for AM. The rPET based dimensional lumber has much higher structural properties than conventional HDPE based lumber and may be used for many light outdoor structural applications at bases, where such construction material are in short supply.
- iv. A ByBlock fabricating plant is designed to operate in two (2) 20-foot ISO containers and can convert both rPET flake and mixed plastics waste into light construction blocks with the same dimensions as CMU's (8x8x16) at 30 tons/month. The ByBlocks were shown to have adequate compressive properties for light outdoor structures and based on a demonstration unit (storage shed) built at ERDC-CERL, detailed guidelines and recommendation have been developed.

5.0 IMPLICATIONS FOR FUTURE RESEARCH/ IMPLEMENTATION

Future research efforts recommended or currently underway as a follow on to the four major agile, manufacturing systems designed as deliverables in this project are discussed below.

Additive Manufacturing: AM at PON with waste plastics such as rPET is of highest priority to benefit the servicemember. The final results summarized in Section 3.4.2 (Task 4.3) that proved the feasibility of using rPET flakes directly for 3D printing without the need for manufacturing filament could be the breakthrough technology needed. Emc² has already partnered with RE3D, the manufacturer of Gigabot-X to pursue this technology further. Recently the team has been awarded a Small Business Innovation Research (SBIR), Phase II grant by the US Army XTECH office in late 2022 to modify the Gigabot-X printer to handle rPET flakes (and other plastics) and build an off-the-grid manufacturing system in a single 20-ft ISO container to convert waste plastics to clean flakes that can then be used for AM applications.

ByBlock Manufacturing: As this project came to a close, ERDC engaged the Defense Logistics Agency (DLA), an organization which, “Disposes of excess property by reutilization, transfer and demilitarization; conducts environmental disposal and reuse.” The DLA recently awarded an SBIR contract (Solicitation DLA202-006, Contract SP4701-21-P-0014) to study responsible disposition of Meal, Ready-to-Eat (MRE) and Unitized Group Ration (UGR) packaging through a, “Separation, Composting, Recycling and Repurposing System.” Based on the results of this project, the awardee took interest in developing a ByBlock source material formula that incorporates MRE and UGR packaging. Follow-on efforts to continue structural and seismic validation of ByBlock structures are expected to occur at the ERDC during FY23 in collaboration with the SBIR contractor.

Demonstration and Validation Project: Based on the design and the feasibility established of the Sorting and Washing plant as well as the Plastic Lumber Extrusion Plant, it is recommended that a demonstration and validation project be undertaken by SERDP-ESTCP at a suitable base in the CONUS. Given the detailed design already developed in this work along with the field trials using similar equipment available at commercial plants, the successful implantation of this project can be achieved by the same project team. The installation of these demonstration plants would be significant in providing a basis for similar system at remotely located FOBs,

Benefits: The overall benefits would be improved self-sustainability of the warfighter in-theater, increased operational readiness, a reduction of the logistics tail, and the ability of the warfighter to manufacture on-demand at the point of use. The successful results described above show that FOB plastics streams can be reused, with field-capable manufacturing processes in 20-foot ISO containers allowing the warfighter to potentially fabricate replacement parts or perform repair operations, while keeping subject waste streams out of open burn pits, or incinerators currently used on many FOBs. Again, reducing open burning would benefit not only the environment, but the health of the personnel performing the burn.

6.0 LITERATURE CITED

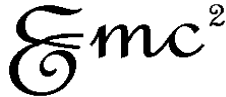
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APPENDIX A LIST OF APPENDICES WITH SUPPORTING DATA

- A-1 Phoenix Technologies Plant Visit Trip Report
- A-2 CommonSense Solutions Report on rPET Sorting and Washing Plant
- A-3 CommonSense Solutions Report on “Small-Scale’ Sorting & Washing Plant
- A-4 Phoenix Technologies rPET Technical Specifications
- A-5 FilaMint Report on rPET Filament Extrusion Trials
- A-6 Conair Report on rPET Filament Extrusion Trials
- A-7 IC3D Report on rPET Filament Extrusion Trials
- A-8 FilaMint Report on rPET ‘Filament Extrusion System’ for AM
- A-9 METSS Report on rPET Plastic Lumber Flow Molding Trials
- A-10 American Cierra Report on Mobile Plastic Lumber System Design
- A-11 ByFusion Technology Overview
- A-12 ByBlock Compression Test Report
- A-13 ERDC-CERL Report on ByBlock Evaluation
- A-14 ByFusion Report on Mobile ByBlock Manufacturing System
- A-15 rPET Material Property Evaluation Report
- A-16 Design Guide for rPET Additive Manufacturing

Appendix A-1

Phoenix Technologies Plant Visit Trip Report



Engineering Mechanics Corporation of Columbus
3518 Riverside Drive - Suite 202
Columbus, Ohio 43221

Phone: (614) 459-3200

Fax: (614) 459-6800

E-mail: rheggs@emc-sq.com

Date: August 20, 2018

To: Prabhat Krishnaswamy, Emc²

From: Rick Heggs, Emc²

Subject: Trip Report for visit to Phoenix Technologies, Bowling Green, OH; August 14, 2018

Phoenix is a PET bottle recycler that specializes in producing clean pelletized RPET that is food grade and is suitable for going back into the PET bottle manufacturing system. The Phoenix facility consists of two plants—a wash and separation plant that produces RPET flake and palletization plant that converts the flake into pellets. The wash plant is large and utilizes bales of PET bottles supplied by various recycle collection centers and material recovery facilities (MURFs) in Ohio. The bales are large approximately (4X4X4 feet) and in addition to PET bottles contain contaminants like aluminum, ferrous metals, and bottles made from other polymers like polyvinyl chloride and polyethylene. The 2 plants are designed to produce approximately 20 million pounds of RPET pellets per year.

Wash and Sort Plant

The wash and sort plant converts the baled post-consumer waste to RPET flake and has several unit operations as shown in Figure 1 below.

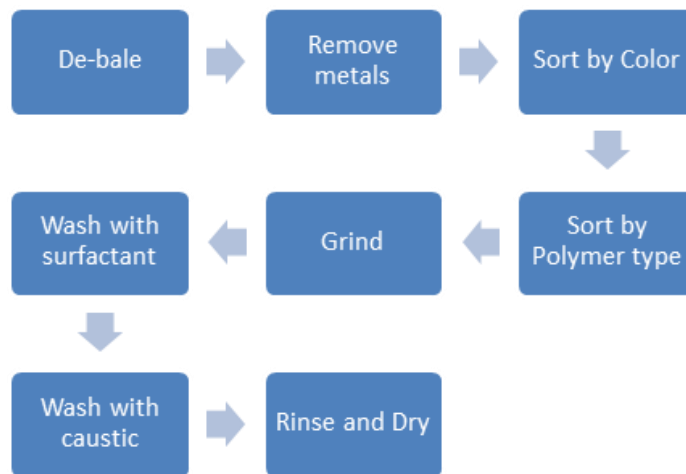


Figure 1- Wash Plant Schematic

The individual processes are:

Debaling: The bales consist of compacted bottles are held together with metal bands or wires which need to be removed and the material is then subjected to an auger which separates the individual bottles from the compacted bale. The debaling step also helps to remove the labels from the bottles. The material is then conveyed on a belt through the process.

Remove Metals: The bales have some aluminum cans, steel cans, metal closures and other metallic contaminants that need to be taken out before the PET can be recovered. In this step the ferrous metals are removed with a large magnetic belt running perpendicular to the conveyed materials and the non-ferrous metals are removed by their greater density.

Sort by Color: Since the goal of this plant is to produce clear flake the colored bottles need to be removed by an optical sorter that excludes every color but blue and clear. This is accomplished automatically with the material coming off the belt into a zone when air jet segregates the colored bottles from the clear and blue ones.

Sort by Polymer Type: The bales contain bottles made from other polymers like high density polyethylene (HDPE) and polyvinyl chloride (PVC) which cannot be melt processed with PET and must be removed. This sorting is done with a near infrared sorter (NIR) which also utilizes an air jet to reject the bottle made from anything but PET.

Grind: The sorted PET bottles are then conveyed to a grinder that reduces the bottles to flakes that are approximately ½ inch in size. This step creates some fine particulates that are removed by air and collected in filter sacks.

Wash by Surfactants: The first wash step is done with detergent to remove any residual contaminants that may be in the bottles and to float and separate the HDPE and polypropylene (PP) closures that may have been on the bottles. This polyolefin (HDPE and PP) waste stream is collected and also sold as a commodity. After this wash step is a dryer.

Wash by Caustic: The second wash step is done with a caustic (sodium hydroxide) solution to remove the label adhesive and any residual label material. This step is followed by a rinse and dry station.

Rinse and Dry: This step removes the caustic solution and removes surface moisture from the flake. The flakes are then put into super-sacks containing about 1500 lbs. These flakes are then sold as a commodity or transferred to the pelletizing and melt filtration plant for further processing.

Melt Processing Plant

The melt processing plant converts the clean flake into pellets suitable for reintroduction into the PET bottle making supply chain. The unit operations in this plant are shown in Figure 2.

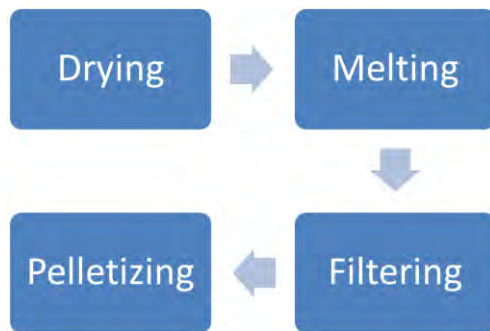


Figure 2 Melt Processing Plant

The unit operations are:

Drying: Since PET is a hygroscopic polymer it must be dried to around 50 ppm of moisture before it is melted to prevent foaming and molecular scission and molecular weight reduction during the extrusion

process. The dryers are commercial units that utilize desiccated air for drying. The dried flake is directly air conveyed to the hopper of the extruder which is also flushed with dried air.

Melting: The RPET flake is then melted in a single screw extruder and conveyed in the extruder to a melt filtration unit.

Filtering: The melt is filtered at the end of the extruder using either a continuous belt filter or a dual filter pack that is exchanged when the pressure increases beyond a set point. The filter is designed to remove small particulates that could cause processing problems in making fibers, preforms, strapping or other products.

Pelletizing: The melt is then forced through a pellet die and cut underwater and conveyed in a water slurry to a pellet separator and then dried. The dried pellet is still amorphous and therefore relatively clear and can be inspected for trace particulate contaminants. The RPET pellet has a final intrinsic viscosity of from 0.71 to 0.80 and is available in food or non-food grade.

Summary

The Phoenix plant is a state of the art PET recycling facility that converts baled compressed post-consumer bottles to food grade pellets suitable for reintroduction into the PET bottle supply chain or used for other product manufacture like strapping or fibers.

The Phoenix personnel were very open and helpful and agreed to assist EMC2 in this project by supplying RPET pellet based on our specification and will help in recommending suitable equipment for washing, sorting, grinding and extrusion processing based on the volume generated at an FOB and on the required specifications of the material for making strapping and 3D printer filament.

Next steps are for Emc² to specify the type and amount of RPET pellets required for plant trials and to give Phoenix information on volume and composition of the FOB waste stream.

Appendix A-2

CommonSense Solutions Report on rPET Sorting and Washing Plant

CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE INFORMATION

Access to Appendix A-2 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

Appendix A-3

CommonSense Solutions Report on “Small-Scale’ Sorting & Washing Plant

**CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE
INFORMATION**

Access to Appendix A-3 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

Appendix A-4

Phoenix Technologies rPET Technical Specifications

SAFETY DATA SHEET

Polyethylene Terephthalate or PET Flakes

1. CHEMICAL PRODUCT/COMPANY IDENTIFICATION

PRODUCT IDENTIFIER: Polyethylene Terephthalate or PET Flakes (RPET flake, NLF™ and RPET RESIN, Lno™, NLP™, LNOCTM)

RECOMMENDED USE AND USE RESTRICTIONS: Polymer for plastics industry. Not intended for internal use or medical implantation.

MANUFACTURER / SUPPLIER:

Phoenix Technologies International LLC.
1098 Fairview Ave.

Bowling Green, OH 43402

EMERGENCY PHONE NUMBERS:

2. HAZARD(S) IDENTIFICATION

OSHA HAZARD CLASSIFICATION: COMBUSTIBLE DUST – WARNING! MAY FORM COMBUSTIBLE DUST CONCENTRATIONS IN AIR.

HAZARDS NOT OTHERWISE CLASSIFIED: CAUTION! MOLTEN MATERIAL WILL PRODUCE THERMAL BURNS. Molten polymer will adhere to skin and may cause severe burns.

Eye contact with polymer particles may cause mechanical irritation with discomfort, tearing, or temporary blurring of vision.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Material: Polyethylene Terephthalate CAS Number 25038-59-9 Variable concentration >99%

Residual additives, modifiers, colorants / impurities <1%

4. FIRST AID MEASURES

INHALATION: Polyethylene Terephthalate is not likely to be hazardous by inhalation. If exposed to fumes from overheating, move to area with fresh air. If not breathing contact emergency services.

SKIN CONTACT: Polyethylene Terephthalate is not likely to be hazardous by skin contact. If molten material contacts skin, cool immediately with cold water. Do not attempt to remove material from skin. Obtain medical treatment.

EYE CONTACT: In case of contact, immediately flush eyes with water for 15 minutes. If contact with molten material, seek medical attention immediately.

INGESTION: Polyethylene Terephthalate is not intended to be ingested as part of normal use. If ingested, consult a physician.

5. FIRE FIGHTING MEASURES

SUITABLE EXTINGUISHING MEDIA: Water, Foam, Carbon Dioxide (CO₂), or Dry Chemical.

SPECIFIC HAZARDS ARISING FROM CHEMICAL

HAZARDOUS COMBUSTION PRODUCTS: Carbon dioxide and carbon monoxide.

SPECIAL PROTECTIVE EQUIPMENT AND PRECAUTIONS FOR FIRE-FIGHTERS

Keep personnel removed and upwind of fire. Wear self-contained breathing apparatus. Wear full protective equipment.

6. ACCIDENTAL PRECAUTIONS / PROTECTIVE EQUIPMENT / EMERGENCY PROCEDURES

The interior of molten masses may remain hot for some time because of the low heat conductivity of the polymer. Use care when handling / disposing of molten masses.

Review Section 5. FIRE FIGHTING MEASURES and Section 7. PRECAUTIONS FOR SAFE HANDLING before proceeding with clean-up. Use appropriate Personal Protective Equipment during clean-up. Thermal protective equipment should be used when handling molten material (See Section 8. For further details).

METHODS AND MATERIALS FOR CONTAINMENT AND CLEANUP

Sweep up and recover, or mix material with moist absorbent and shovel into suitable chemical waste container.

7. HANDLING AND STORAGE

PRECAUTIONS FOR SAFE HANDLING: Do not breathe gases, vapors or fumes that may be evolved during processing. Caution and suitable thermal eye, face, and body personal protective equipment must be used if handling hot or molten material. Contact with molten material can cause burns, so unprotected contact with molten material must be avoided.

Polyethylene Terephthalate can cause slip hazards in multiple forms (Flake, Pellet, and Dust) and should be kept clear of all walkways. Avoid dust generation to prevent dust accumulation to minimize explosion hazard. Grinding of Polyethylene Terephthalate can create dust and potential dust explosion hazard. To reduce risk follow National Fire Protection Association's codes and standards for handling combustible dust.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS: There are no established exposure limits for Polyethylene Terephthalate for PEL or TLV. Particles not otherwise regulated the limit is 15mg / m³ total dust 5 mg / m³ respirable fraction (PEL). For particles not otherwise specified 3mg / m³ respirable particles and 10mg / m³ for inhalable particles. All mentioned limits are 8 hour time weighted average limits.

APPROPRIATE ENGINEERING CONTROLS: Use local ventilation to control gasses, vapors and fumes from hot processing. Use static controls. Static charges can build up and ignite dust or solvent laden atmospheres. Design precautions into processes that can create dust, such as pneumatic conveying systems, grinding and other physical operations. There is the potential for a dust explosion hazard. Ensure that dust handling systems are designed in a manner to prevent the escape of dust into work areas.

INDIVIDUAL PROTECTION MEASURES / PERSONAL PROTECTIVE EQUIPMENT

EYE/FACE PROTECTION: Wear coverall chemical splash goggles when the possibility of eye or face contact from airborne material exists. Wear a face shield when working with molten material.

RESPIRATORY PROTECTION: Respirators are not needed for normal use. When airborne concentrations are expected to exceed exposure limits, a NIOSH approved respirator should be selected based on the form and concentration of the contaminant in air and in accordance with the OSHA Respiratory Protection Standard (29 CFR 1910.134).

PROTECTIVE CLOTHING: If there is potential for contact with hot/molten material, wear heat resistant impervious clothing and footwear. Special protective clothing is not needed for normal use. Gloves are recommended as good industrial practice.

RECOMMENDED DECONTAMINATION FACILITIES: Eyewash station, washing facilities.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance:	Solid, clear/colored flakes	Flammability Limits (upper/Lower):	No data available
Odor:	Odorless	Vapor Pressure:	Not Applicable
Odor Threshold:	No data available	Vapor Density:	Not Applicable
pH:	Not Applicable	Specific Gravity:	1.33 – 1.45 g/cm ³
Melting Point:	225 – 250 °C	Solubility in Water:	Insoluble
Initial Boiling Point/Range:	No data available	Partition coefficient(n-octanol/water):	No data available
Flash Point:	Not applicable, combustible solid	Auto-Ignition Temperature:	No data available
Evaporation Rate:	No data available	Decomposition Temperature:	330 °C
Flammability:	No data available	Viscosity:	No data available

10. STABILITY AND REACTIVITY

REACTIVITY: None known

CHEMICAL STABILITY: Stable at normal conditions. Polymerization will not occur

POSSIBILITY OF HAZARDOUS REACTIONS: None known

CONDITIONS TO AVOID: Temperatures above 330 °C

INCOMPATIBLE MATERIALS: Incompatible or can react with strong oxidizers.

Hazardous decomposition products: Thermal decomposition products caused by overheating polymer can include carbon monoxide, carbon dioxide, acetaldehyde and ethylene. Decomposition products (gases, vapors and / or fumes) may cause skin, eye or respiratory tract irritation, and other adverse health effects.

11. TOXICOLOGICAL INFORMATION

INFORMATION ON LIKELY ROUTES OF EXPOSURE: Polymer dust may be inhaled, and come in contact with skin and eyes. Thermal decomposition products may be inhaled.

SYMPTOMS RELATED TO PHYSICAL, CHEMICAL AND TOXICOLOGICAL CHARACTERISTICS:

Skin contact with molten material will produce thermal burns.

Eye contact with polymer dust may cause mechanical irritation with discomfort, tearing, or blurring of vision. Polyethylene terephthalate is a mild eye irritant.

Eye contact with molten material will produce thermal burns.

Decomposition products (gases, vapors and / or fumes) may cause skin, eye or respiratory tract irritation, and other adverse health effects.

ACUTE, DELAYED, AND CHRONIC EFFECTS FROM SHORT AND LONG-TERM EXPOSURE:

Polyethylene terephthalate animal testing indicates

- No carcinogenic, mutagenic, developmental or reproductive effects
- No adverse effects from short exposures by inhalation or ingestion.

Polyethylene terephthalate patch tests with humans resulted in no skin irritation or skin sensitization.

NUMERICAL MEASURES OF TOXICITY: Polyethylene Terephthalate: Oral Approximate Lethal Dose (ALD): > 10,000 mg/kg in rats

CARCINOGENICITY INFORMATION: None of the components present in this material at concentrations equal to or greater than .1% are listed by NTP, IARC, OSHA or ACGIH as carcinogenic.

12. ECOLOGICAL INFORMATION

No toxicity data is available. The product is insoluble in water.

13. DISPOSAL CONSIDERATIONS

Treatment, storage, transportation and disposal must be in accordance with applicable Federal, State / Provincial, and Local regulations.

14. TRANSPORTATION INFORMATION

Shipping information

DOT	Not regulated
TDG	Not regulated
UN Proper Shipping Name/Number	Not regulated

IMDG
IATA

Not Regulated
Not Regulated

15. REGULATORY INFORMATION

U.S. FEDERAL REGULATIONS:

- TSCA Inventory Status: In compliance with TSCA Inventory requirements for commercial purposes.
- Under RCRA, it is the responsibility of the product user to determine at the time of disposal whether a material containing this product or derived from the product should be classified as a hazardous waste (40 CFR 261.20-24).
- If discarded in its purchased form, this product does not meet the RCRA characteristic definition for ignitability, corrosivity or reactivity and is not a RCRA listed waste: however, it has not been tested by the Toxicity Characteristic Leaching Procedure (TCLP).
- SARA, Title III: This material is not known to contain extremely hazardous substances.

INTERNATIONAL REGULATIONS:

- DSL (Canadian Domestic Substances List) and CEPA (Canadian Environmental Protection Act): This product listed on the DSL or otherwise complies with CEPA new substance notifications requirements.
- No components of this product are on the Mexican Raw Materials Regulation.

16. ADDITIONAL INFORMATION

The data in this Safety Data Sheet relates only to the specific material designated herein and does not relate to use in combination with any other material or in any process.

SDS Revision Date: March 2, 2016

End of SDS



Specifications for Crystalline RPET Phoenix Process

100% Post-Consumer PET Resin Grade: NLP Clear resin

GENERAL PURPOSE GRADE

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY	0.71 ± 0.02/-0.03 dL/gm
MELTING POINT	246° ± 3° C.
CRYSTALLINITY	36.0 % ± 5.0
UNDERCRYSTALLIZED PELLETS	< 5%
CIE (L*, a*, b*) COLOR †	L* 67.0 Min a* -1.50 + 1.0 b* + 2.0 Max
PELLET SIZE	0.120" ± 0.020 MAJOR DIA.
FINES	0.08 % MAX.
FILTRATION	filtered to 60 microns
MOISTURE CONTENT	0.2 % MAX.
BULK DENSITY	50 -60 lbs/ ft3

NLP Clear rPET resin is derived from diverse feed stocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

PHOENIX TECHNOLOGIES MAKES NO WARRANTY BEYOND THE SPECIFICATIONS AND DISCLAIMS ANY WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

† 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant; Reflectance method

†† As provided by continuous melt filtration by min. 200 Mesh screen and as measured in a single dimension by Phoenix test method LAB103.



Specifications for Crystalline RPET Phoenix Process

100% Post-Consumer PET Resin Grade: NLP H Clear resin

GENERAL PURPOSE GRADE

- trial

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY	0.75 ± 0.02/-0.03 dL/gm
MELTING POINT	246° ± 3° C.
CRYSTALLINITY	36.0 % ± 5.0
UNDERCRYSTALLIZED PELLETS	< 5%
CIE (L*, a*, b*) COLOR †	L* 67.0 Min a* -1.50 + 1.0 b* + 2.0 Max
PELLET SIZE	0.120" ± 0.020 MAJOR DIA.
FINES	0.08 % MAX.
FILTRATION	filtered to 60 microns
MOISTURE CONTENT	0.2 % MAX.
BULK DENSITY	50 -60 lbs/ ft3

NLP Clear rPET resin is derived from diverse feed stocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

PHOENIX TECHNOLOGIES MAKES NO WARRANTY BEYOND THE SPECIFICATIONS AND DISCLAIMS ANY WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

† 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant; Reflectance method

†† As provided by continuous melt filtration by min. 200 Mesh screen and as measured in a single dimension by Phoenix test method LAB103.



Specifications for RPET Phoenix Curbside Flake
100% Post-Consumer PET Resin Grade: NLF-GREEN

NON FOOD GRADE

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY (SOLUTION)	> 0.73 dL/gm
CIE COLOR [†]	a* -32 - 0
FLAKE SIZE	< 12mm
FINES	< 0.5% MAX.
MOISTURE CONTENT	≤ 1.0%
BULK DENSITY	≥ 20 lbs/ ft ³
PVC CONTAMINATION	< 50 ppm (lot average)
LOWMELT CONTAMINATION	< 250 ppm
NYLON CONTAMINATION	< 30 ppm
NON MELTING *	< 100 ppm
OTHER COLORS PET++	< 250ppm

No single package within a load should exceed these specifications, if the load is in a bulk truck the load average should meet these specifications

NLF grade RPET resin is derived from diverse feedstocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

[†] 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant. Reflectance method.

* non-melting to include items such as metal, glass, sand, stones, wood (any item with a melt point > 260 C)

++ applies to all other colored and opaque PET including but not limited to amber, pink, white, yellow

Phoenix Technologies Grade RPET Resins NLP™ and LNO™P/PT

Technical Processing Information

Recommended Drying Procedures

PET is a very hygroscopic material which readily absorbs moisture when exposed to ambient air. PET which is not properly dried will result in a lower IV and loss of other important physical properties. At time of processing, the moisture in PET must be less than **50 ppm** or 0.005% by weight.

Dry thoroughly in a high heat regenerative–desiccant drying system. DO NOT remove dried material from the drying hopper or expose it to ambient conditions prior to processing. Rapid degradation will occur when hot, dry PET is exposed, even briefly, to ambient air.

Recommended Drying Parameters

Drying Time	4-5 hours
Drying air temperature	295-330° F.
Drying air dewpoint	-40° C. / F.
Drying air flow rate	1 CFM per pound per hour processed

The drying temperature used should be kept as low as possible to minimize yellowing by oxidation during this portion of the process.

Material Handling

Phoenix RPET resin is produced from Recycled PET. It shows good material flow characteristics and blends well with major virgin PET resins. For best results blending should be done just prior to drying, and conveying blended materials long distances should be avoided, but Phoenix RPET resin can generally be handled as your virgin PET resin.

Processing Parameters

Phoenix RPET resin can be processed very easily over a wide range of conditions. Typically, processing temperatures range from 530-565° F. The main focus of processing should be to minimize process temperatures and melt residence times in an effort to maintain the molecular weight of the material and minimize any yellowing of the material during processing.

- Use the lowest possible barrel-temperature profile that will produce a clear product.
- Minimize melt-residence time.
- Minimize the back pressure used in extrusion to reduce shearing of the material.
- Use the lowest possible hot runner and nozzle temperature that will produce a clear part.
- Use the lowest possible mold coolant temperature to promote part clarity.

Phoenix Technologies

Our specialization in RPET has allowed us to concentrate all of our time and effort to produce the highest quality, contaminate-free recycled resin which is very close to virgin material in quality and performance.

All of us at Phoenix Technologies want to become your supplier of recycled PET resin and are capable and willing to assist you with specific processing questions on an individual basis. Please feel free to call us at 419-353-RPET.

Material Safety Data Sheets providing safety precautions that should be observed in handling and storing the products mentioned in this publication are available and should be reviewed before handling these products.

Phoenix Technologies International LLC provides NLP™ and LNO™ grade RPET resin to a variety of customers for specific applications depending upon their needs. We are not in the business of advising customers and suppliers generally as to usage of our product, including drying and processing procedures, or to the testing or other methodology which could require modification depending upon the specific applications desired. The above information is furnished without charge as an accommodation to our customers and suppliers and is of general application only. Phoenix Technologies International LLC and its affiliates do not warrant or represent the accuracy of such information in each and every application and disclaim any and all responsibility for the results or consequences of use of such information or the use of any product, method, or apparatus referred to above. It is the responsibility of each customer or user, based on whatever testing criteria it deems appropriate, to determine the suitability of our product or methods for any particular use or purpose.

GEHR Plastics PET Polyethylene Terephthalate

Categories: [Polymer](#); [Thermoplastic](#); [Polyester, TP](#); [Polyethylene Terephthalate \(PET\)](#)

Material Notes: Polyethylene terephthalate shows high tensile and mechanical strength, hardness and toughness, low friction and a high dimensional stability. PET may be used at temperatures ranging from -20°C to approximately 115°C.

Properties

- high mechanical resistance and tensile strength
- high surface strength
- easy to varnish and polish
- high dimensional stability
- high toughness
- good electrical insulating properties
- high chemical resistance
- limited dielectric properties
- sensitive to hydrolysis

Applications include bearings, pumps and parts for housings, frame components, tank lids, cogwheels, insulators for the electrotechnic, deflection rollers for the filament industry, levers, handles, and control disks.

Vendors: No vendors are listed for this material. Please [click here](#) if you are a supplier and would like information on how to add your listing to this material.

Physical Properties	Metric	English	Comments
Specific Gravity	1.39 g/cc	1.39 g/cc	ISO 1183
Moisture Absorption at Equilibrium	0.25 %	0.25 %	ISO 62
Water Absorption at Saturation	0.50 %	0.50 %	ISO 62

Mechanical Properties	Metric	English	Comments
Hardness, Rockwell M	96	96	ISO 2039
Hardness, Shore D	84	84	ISO 868
Ball Indentation Hardness	170 MPa	24700 psi	ISO 2039
Tensile Strength, Yield	90.0 MPa	13100 psi	ISO 527
Elongation at Break	15 %	15 %	ISO 527
Elongation at Yield	4.0 %	4.0 %	ISO 527
Modulus of Elasticity	3.445 GPa	499.7 ksi	ISO 527
Charpy Impact Unnotched	5.00 J/cm ²	23.8 ft-lb/in ²	ISO 179
	5.70 J/cm ²	27.1 ft-lb/in ²	round rods 220-300mm; ISO 179
	6.50 J/cm ²	30.9 ft-lb/in ²	round rods 160-200mm; ISO 179
Charpy Impact, Notched	0.200 J/cm ²	0.952 ft-lb/in ²	ISO 179

Electrical Properties	Metric	English	Comments
Volume Resistivity	3.00e+13 ohm-cm	3.00e+13 ohm-cm	IEC 60093
Surface Resistance	>= 1.00e+13 ohm	>= 1.00e+13 ohm	IEC 60093
Dielectric Constant	3.2	3.2	IEC 60250
	@Frequency 1.00e+6 Hz	@Frequency 1.00e+6 Hz	
Dielectric Strength	22.0 kV/mm	559 kV/in	IEC 60243-1
Dielectric Loss Index	0.014	0.014	IEC 60250
	@Frequency 1.00e+6 Hz	@Frequency 1.00e+6 Hz	
Comparative Tracking Index	600 V	600 V	IEC 60112

Thermal Properties	Metric	English	Comments
CTE, linear	80.0 µm/m-°C	44.4 µin/in-°F	ISO 11359

Thermal Conductivity	0.290 W/m-K	2.01 BTU-in/hr-ft ² -°F	ISO 22007-4
	@Temperature 20.0 °C	@Temperature 68.0 °F	
Melting Point	245 °C	473 °F	DIN EN ISO 3146
Maximum Service Temperature, Air	100 °C	212 °F	UL 746B
Deflection Temperature at 1.8 MPa (264 psi)	80.0 °C	176 °F	ISO 75
Minimum Service Temperature, Air	-20.0 °C	-4.00 °F	UL 746B
Glass Transition Temp, Tg	98.0 °C	208 °F	DIN EN ISO 3146
Flammability, UL94	HB	HB	UL 94
Oxygen Index	25 %	25 %	ASTM D2863

Compliance Properties	Metric	English	Comments
FDA	Yes	Yes	Physiological indifference

Descriptive Properties

Acid Resistance	yes
Aromatic Resistance	yes
Bondability	yes
CKW Resistance	limited
Color	natural
Hydrocarbonate Resistance	yes
Hydroxide Resistance	limited
Physiological indifference	yes, EEC
UV Stabilization	limited / no

Some of the values displayed above may have been converted from their original units and/or rounded in order to display the information in a consistent format. Users requiring more precise data for scientific or engineering calculations can click on the property value to see the original value as well as raw conversions to equivalent units. We advise that you only use the original value or one of its raw conversions in your calculations to minimize rounding error. We also ask that you refer to MatWeb's [terms of use](#) regarding this information. [Click here](#) to view all the property values for this datasheet as they were originally entered into MatWeb.

Appendix A-5

FilaMint Report on rPET Filament Extrusion Trials

Date: 1-31-2019

10:30 AM



10:50

Open hopper and start pushing PET through extruder

Evaluate PET coming out of die. Temperatures were set at recommended 560°F. PET was very viscous and had very little melt strength. It would pull apart as soon as it was touched.

Started lowering die and adapter temperatures to increase stability of melt.

Amp load on extruder motor was surging going at slow speed. Ranging from 2-17amps. Increased extruder throat temperature.



11:25



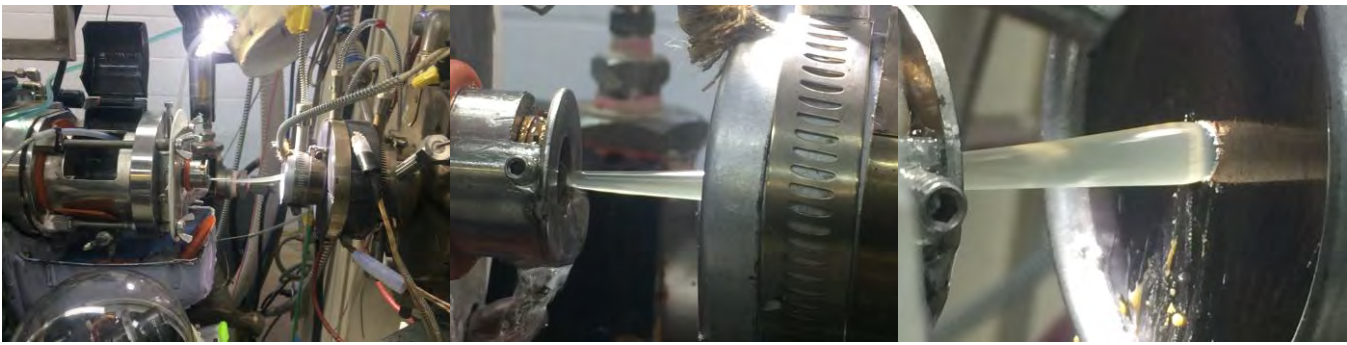
11:25

Kept decreasing die temperature to get more stable melt. At 400°F melt started to become manageable for threading through system.

11:55 AM

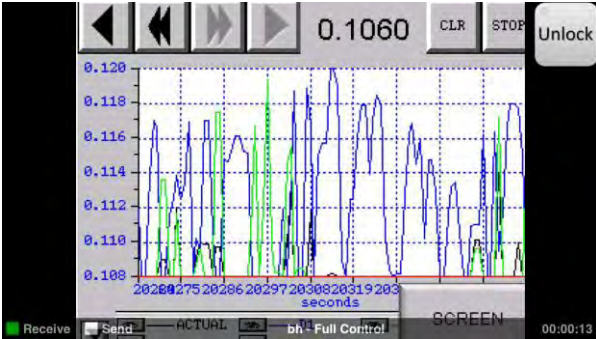


Threaded PET through water bath and puller.



Increased 30 RPM of extruder, amp load stabilized.

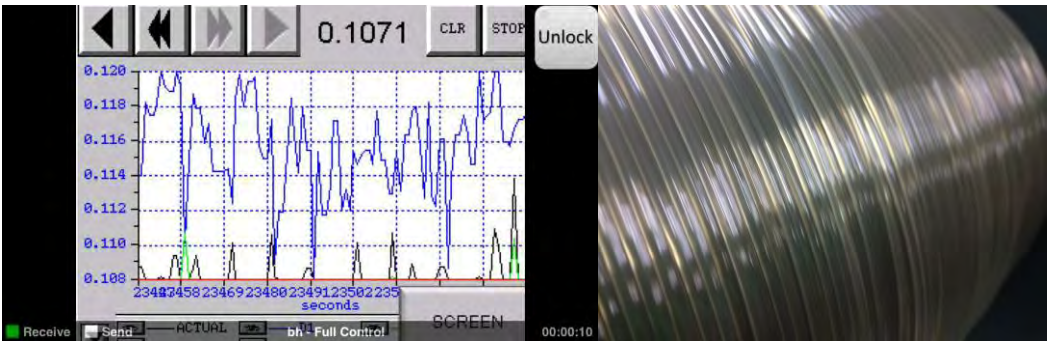
Attempted to get material to stabilize and concentric by adjusting water bath temperatures and adjusting puller speed.



12:55 PM

Tested other puller speeds

1:20



1:50

Raised die temperature to 430°F. Made material more unstable.

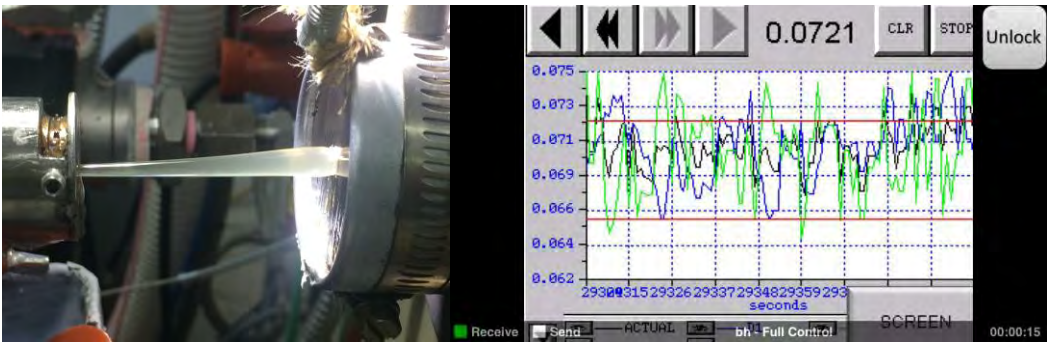
1:53

Attempted to draw material down to 1.75mm. Started having more success.

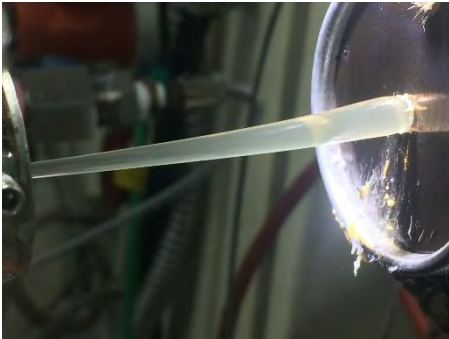


2:30 PM

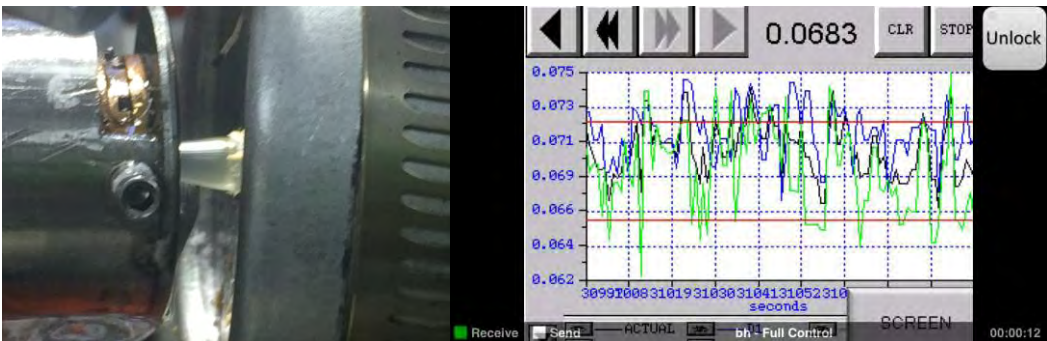
Moved water bath 1.5" from die face. Material started to get more stable.



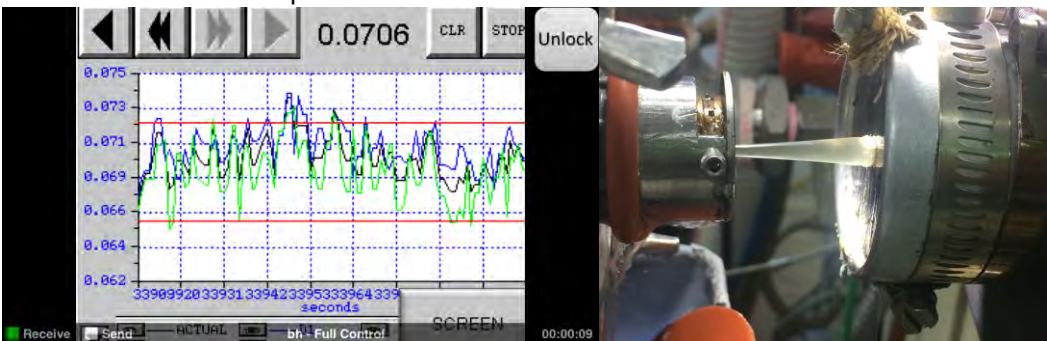
2:50
Moved water bath 2" from die face



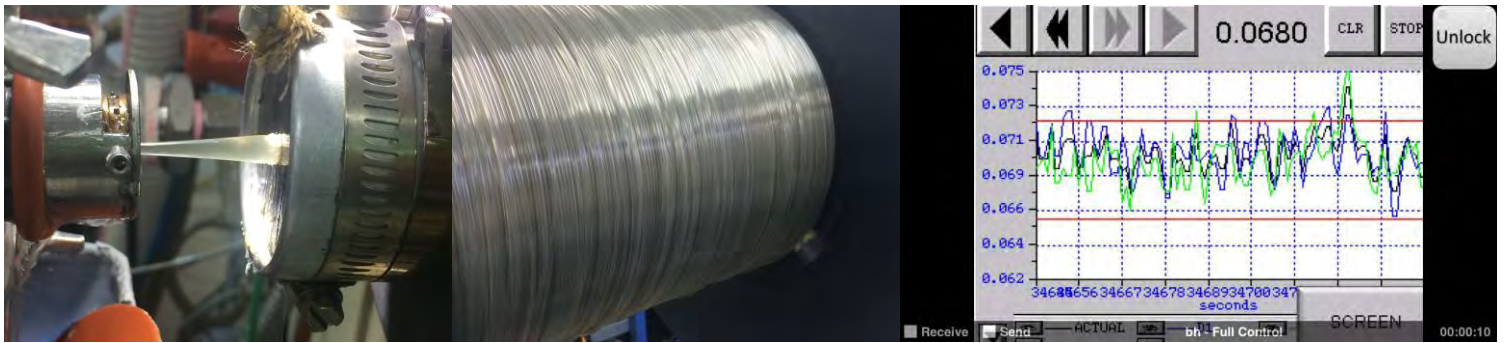
3:00
Moved water bath 0.5" from die face.



3:30
Started second master spool. Water bath 1" from die face



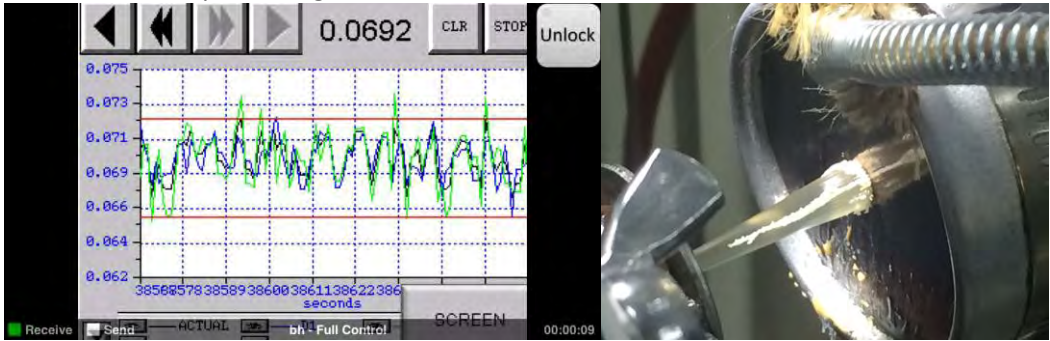
3:48
Material started to stabilize and make acceptable product.



4:17
Die build up starting to occur



4:49
More die build up occurring



4:55
Started shutting down.
Ended with 6702 feet on second spool

5:30
Completed purging with HIPS

2-1-2019

11:00 AM

Open gate and start purging.

The previous day we had transferred the flake from container we received to pail with vacuum to remove some of the dust. It did seem to reduce dust considerably.



HIPS PURGE



Flake start



12:15 starting to stabilize

Considerable amount of die build up.

Line broke after threading because of die build up clump.

12:35

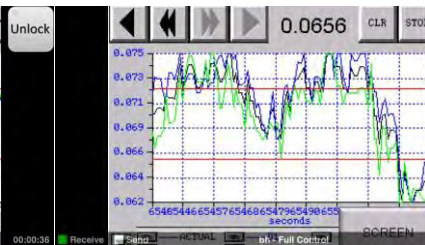
Started spooling.



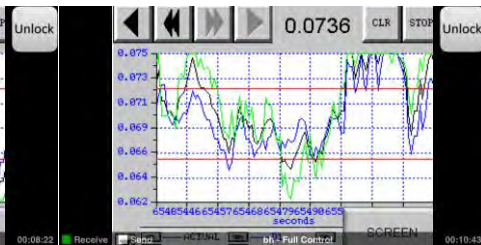
Material kept surging. It was ranging from 0.061-0.073. Surge cycle was approximately every 52 seconds to 2 minutes.



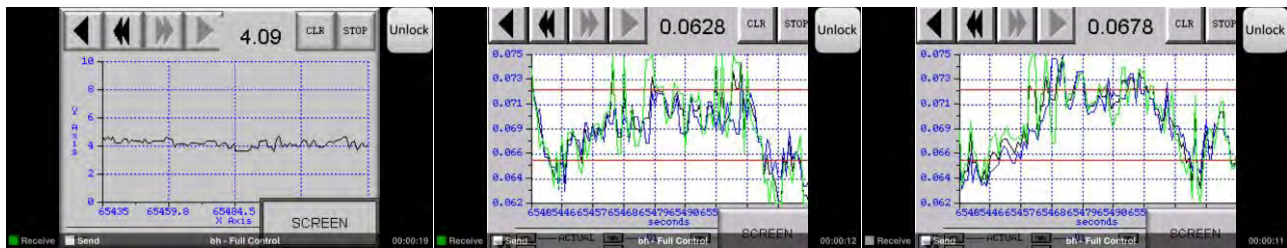
12:52



15:55



1:04

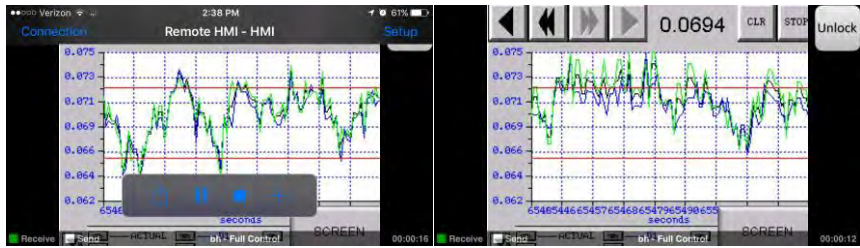


Amp load on extruder motor

1:49

1:54

Amp load on extruder stayed relatively steady. Fluctuations came mostly from the material. The steady amp load lead us to believe that it was not a feeding problem.

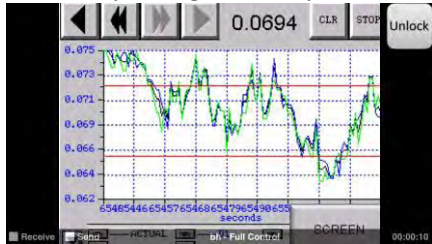


2:35

2:44

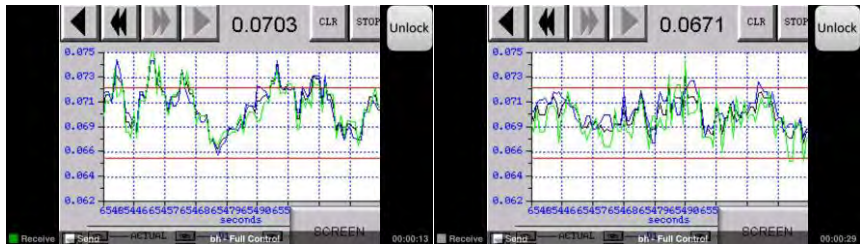
Line snap due to contamination at 2:50.

3:30
Started spooling on last spool.



3:44

4:45 started losing tolerance



4:59 stabilized

6:11



Class:

rPET Pellet

Date: 1-31-19

Material:	NLP 1	Manufacturer:	Phoenix Technologies
Grade:		Supplier:	Phoenix Technologies
Lot/Batch:	2011-11-9		

YEILD REPORT

STARTING WEIGHT

100

	1.75MM		2.85MM	START UP
Start Purge	9.4			
Settings Discovery	3.6			
Product Yield	26.874			
Out of Spec Product	30.53			
Left over	29.6			
Total				
Viscosity	560 – very low, 400-med	Draw down	38% (0.179" die)	
Melt Strength	560-low, 400-med	Die swell	Minimal	
Melting Point	slow	Set up	slow	

THREADING/START-UP REVIEW

With decent melt strength the material was strung up easily. Above 450° strand of PET was used for threading. Below 450° thread with loop used.

Processing Parameters

Dry Time	4 hours prior to start	Speed	Moderate
Dry Temperature	325°F	Pressure	Low
Screen Pack	60 mesh	Motor Draw	High until speeds increased
Temperatures	See attached	Cooling	Hot water bath

TENSION/WINDING

Tension and winding was easy to achieve desired results with normal set up

QUALITY

Tolerance	B
Ovality	C
Stability	C



Class:

rPET Flake

Date: 2-1-19

Material:	NLF 4 MM LNO FLAKE	Manufacturer:	Phoenix Technologies
Grade:		Supplier:	Phoenix Technologies
Lot/Batch:	2011-11-9		

YEILD REPORT

STARTING WEIGHT

100

	1.75MM		2.85MM	START UP
Start Purge	7.2			
Settings Discovery	2.4			
Product Yield	26.874			
Out of Spec Product	17.93			
Left over	45.60			
Total				
Viscosity	560 – very low, 400-med	Draw down	38% (0.179" die)	
Melt Strength	560-low, 400-med	Die swell	Minimal	
Melting Point	slow	Set up	slow	

THREADING/START-UP REVIEW

With decent melt strength the material was strung up easily.

Processing Parameters

Dry Time	4 hours prior to start	Speed	Moderate
Dry Temperature	325°F	Pressure	Low
Screen Pack	60-200-60 MESH	Motor Draw	High until speeds increased
Temperatures	See attached	Cooling	Hot water bath

TENSION/WINDING

Tension and winding was easy to achieve desired results with normal set up

QUALITY

Tolerance	C
Ovality	D
Stability	D

PROCESS SETTINGS

	PELLETS		FLAKE
MASTER POT	35		56
SCREW RPM/AMP	7.2		5.1
PULLER FPM	86		74.5
PULLER POT	66.5		37
MELT PRESS	3700		2100
MELT TEMP	516		519
SCREEN PACK	60		60-200-60
ZONE 1	570		570
ZONE 2	565		565
ZONE 3	560		560
ZONE 4	550		550
GATE	480		490
ADAPTER	430		440
CLAMP	380		390
DIE	390		420
CALIB WAT HEAT	140		150
WATER HEATER	140		140
BATH			
DISTANCE FROM DIE	1"		1"

SPOOL NUM	WEIGHT	QUALITY	NOTES
PELLETS			
1-1	2.738	B	more concentric
1-2	2.670	B	more concentric
1-3	2.684	C	oval shaped
1-4	2.688	C	oval shaped
1-5	2.718	C	oval shaped
1-6	2.734	C	oval shaped
	16.232		
2-2	2.508	A	Best overall quality
2-4	2.686	A	Best overall quality
2-5	2.660	A	Best overall quality
2-6	2.788	A	Best overall quality
	10.642		
FLAKE			
3-1	2.050	B	Surging by decent quality
3-2	2.762	B	Surging by decent quality
3-3	2.804	B	Surging by decent quality
3-4	1.236	B	Surging by decent quality
3-9	1.232	C	Contamination present
	10.084		
4-2	2.378	C	decent tolerance, contamination present
4-4	1.794	D	decent tolerance, contamination present
4-7	2.330	D	decent tolerance, contamination present
	6.502		
5-1	0.574	F	Good tolerance, contamination present
5-3	0.634	F	Good tolerance, contamination present
5-4	1.064	F	Good tolerance, contamination present
	2.272		
TOTAL	45.732		

Pellet total	26.874
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Flake total	18.858
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rPET Variables with most impact and Conclusion

After testing many different temperatures, pressures and speeds what we found to be the most important variables are:

Reverse temperature profile

Having the temperatures at an elevated temperature (570°F) at the feed throat and then gradually reducing them until reaching the lowest temperature at adapter and die of 400°F seemed to reduce surging, gave a consistent feed and provided a workable melt.

Drawdown

The drawdown from 0.179" to 0.112" (2.85 mm) was not enough. We had to draw it down to 0.0688" (1.75 mm) to get a stable product. Even a larger draw down may be considered to produce a more steady product.

Speed of extruder

At low speeds the extruder amps were spiking a lot. It would range from 2 – 17 amps. Upon increasing RPMs to 30 the amp load became steady.

Water bath distance

We tested different gaps between the water bath entrance and die. Too great of a gap (2") usually caused an oval shape product. Too small of a gap (0.5") seemed to cause the material to be unsteady and any die build up that was produced would immediately stick to the material and cause a problem. Around 1" distance from die face to water bath entrance produced the most desirable results. The little amount of air gap seemed to allow the material to stabilize and enter the water bath without any turbulence.

Water bath temperature

At 140°F throughout the entrance and the rest of the tank also seemed to produce the best results. We had tested tank temperatures ranging from 100°F to 150° and water bath entrance area from 100°F to 190°F. The material seemed to stabilize the best at 140°F throughout. It allowed it to start setting up but not quenching it too quickly.

Pellets vs flake

Being recycled material we did notice contamination in both the pellets and flake. We observed specs throughout the run. The flake seemed to have more contamination. We came to the conclusion that due to the material being compounded into pellets and making more of a homogenous mix.

Flake seemed to feed successfully without any bridging but it kept producing surge cycles in the product. The surges seemed to be steady at every 52 seconds to two minutes. Again, we would conclude that this is primarily due to a non-homogenous mix. We also noticed more unmelts.

Conclusion

Once the above variables were discovered the rPET seemed to run successfully. It was not a very steady product to run, it would have to be watched closely to see if it is going out of tolerance and adjustments made.

Date: 5-2-2019

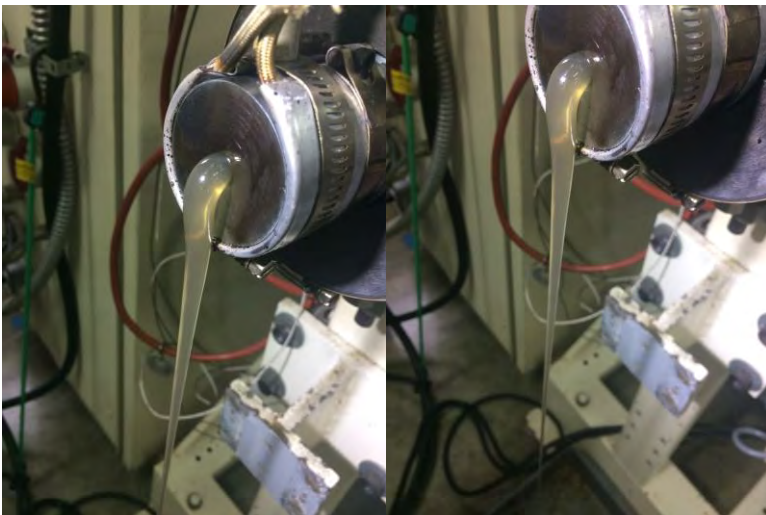
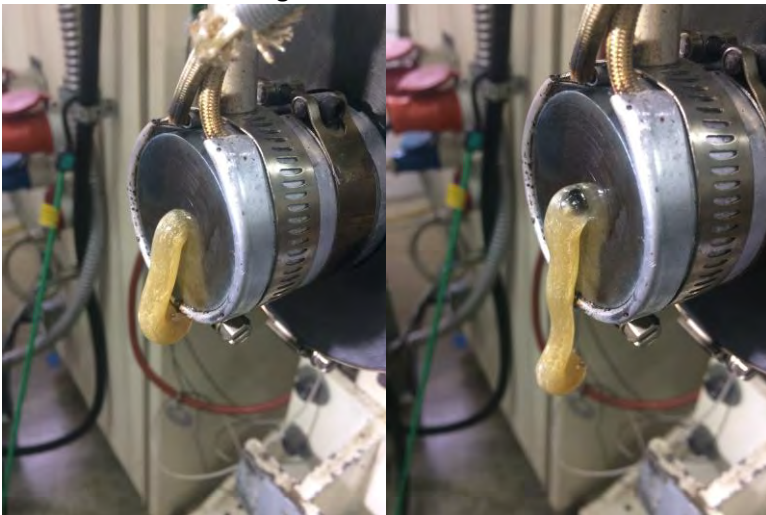
Sort material and try to remove as much excess aluminum and contamination as possible.



Date: 5-3-2019

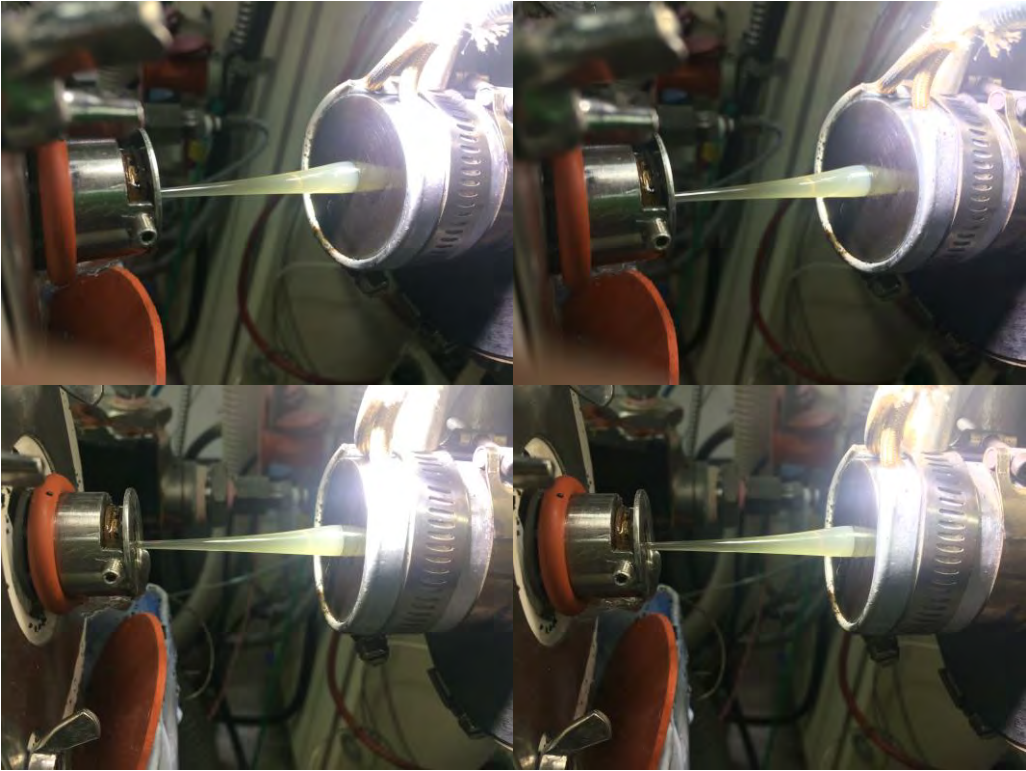
9:30 AM
Started to purge material.

10:05 AM
Material started coming out of die.



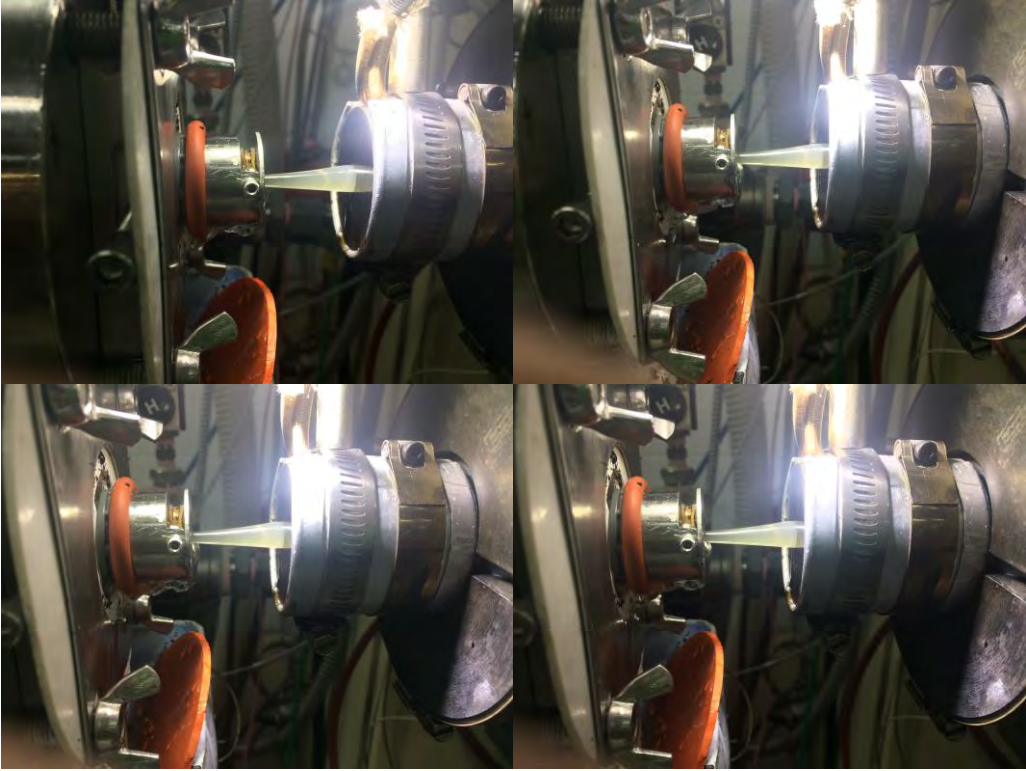
1:05 PM

Able to thread material to start producing a filament.

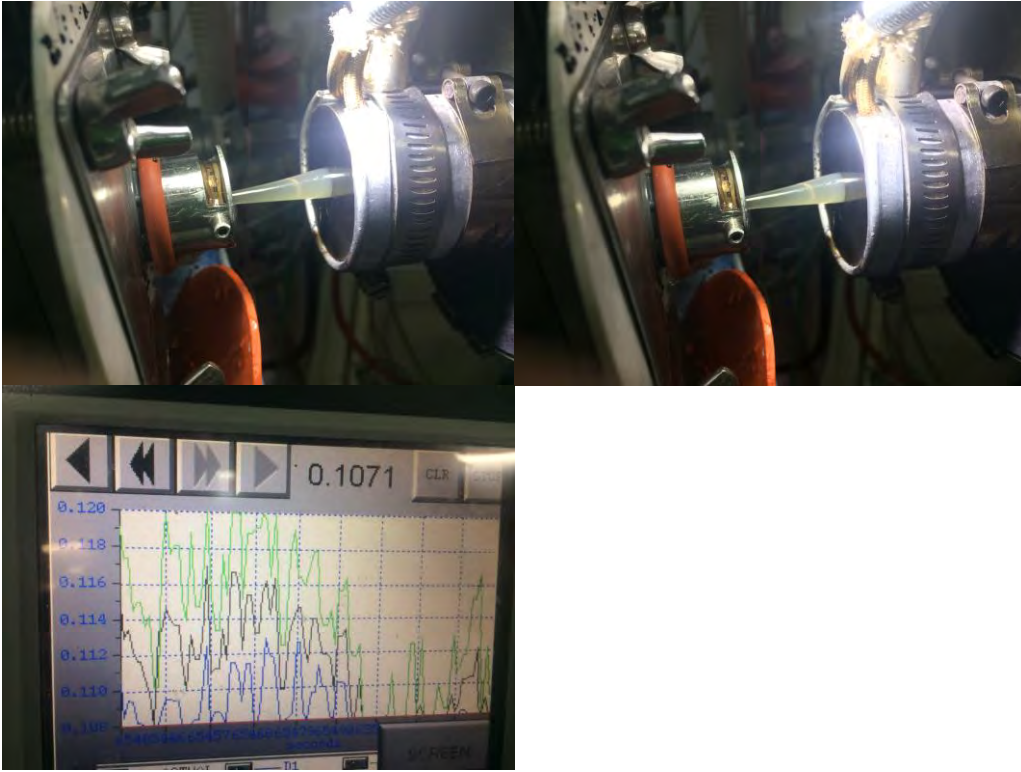


1:35 PM

Started winding



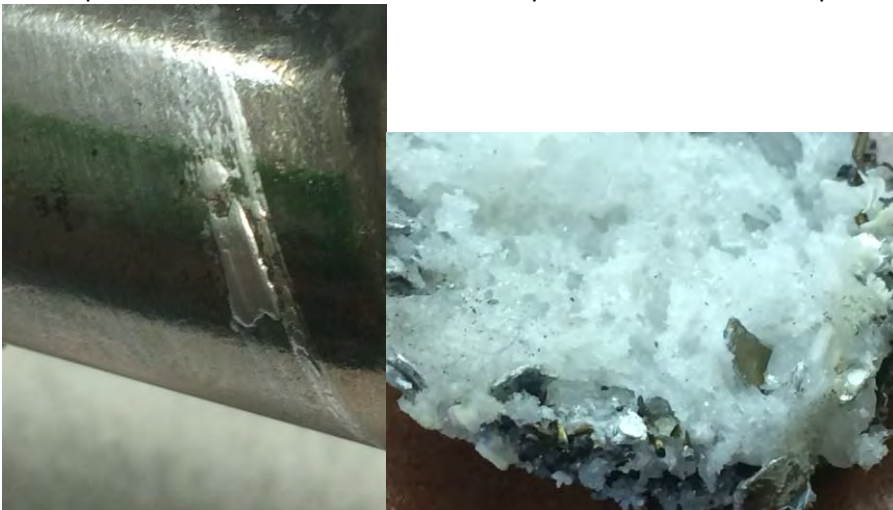
2:20 PM
Started second spool

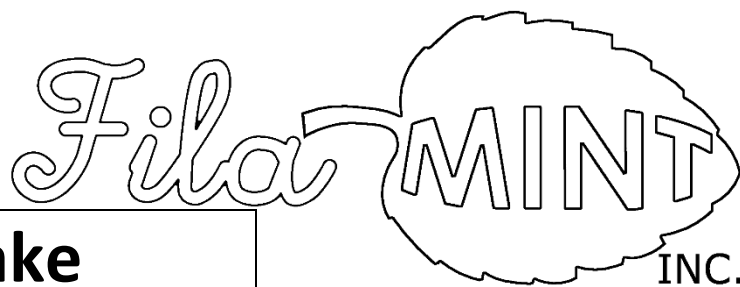


4:07 PM
Line snap caused by screen and breaker being destroyed



Build up on screw and contaminated screen pack with aluminum pieces.





Class:

rPET Flake

Date: 5-3-19

Material:	NLF 4 MM LNO FLAKE	Manufacturer:	Phoenix Technologies
Grade:		Supplier:	Phoenix Technologies
Lot/Batch:	2011-11-9		

YEILD REPORT

STARTING WEIGHT

41.0

	1.75MM		2.85MM
Start Purge			3.8
Settings Discovery			9.49
Product Yield			18.96
Out of Spec Product			7.302
Left over			1.44
Total			41.0
Viscosity	Low	Draw down	High-38% (0.325" die)
Melt Strength	560-low, 400-med	Die swell	Minimal
Melting Point	slow	Set up	slow

THREADING/START-UP REVIEW

Material had to be thread up by being approximately 4" from die pulled to a reduced size, threaded quickly, then slowly decrease puller speed to increase size of product.

Processing Parameters

Dry Time	4 hours prior to start	Speed	Slow
Dry Temperature	325°F	Pressure	Low until spike
Screen Pack	40-80-200-60-20 mesh	Motor Draw	Medium
Temperatures	See attached	Cooling	Hot water bath

TENSION/WINDING

Tension and winding was easy to achieve desired results with normal set up

QUALITY

Tolerance	D-F
Ovality	F
Stability	F

PROCESS SETTINGS

	FLAKE		FLAKE- ENDING
MASTER POT	40		40
SCREW RPM/AMP	4.7		5.1
PULLER FPM	27.8		25.9
PULLER POT	26		23.5
MELT PRESS	1700		3800
MELT TEMP	488		490
SCREEN PACK	40-80-200-60-20		40-80-200-60-20
ZONE 1	560		560
ZONE 2	555		555
ZONE 3	550		550
ZONE 4	540		540
GATE	480		480
ADAPTER	425		425
CLAMP	380		380
DIE	500		500
CALIB WAT HEAT	180		180
WATER HEATER	120		120
BATH			
DISTANCE FROM DIE	1"		1"

188 feet equal approximately 1 pound of material

Processing speed was approximately 8.3 pounds per hour

SPOOL NUM	WEIGHT	QUALITY	NOTES
FLAKE			
1-1	2.418	F	Ovality issues
1-2	2.044	F	Ovality issues
1-3	2.292	F	Ovality issues
2-4	2.802	F	Improved
2-5	2.186	F	Improved
2-6	2.306	D	Best quality of the spools
2-7	2.494	D	Ovality and tolerance
2-8	2.418	D	Ovality and tolerance – some contamination
TOTAL	18.96		

rPET Variables with most impact and Conclusion

After testing many different temperatures, pressures and speeds what we found to be the most important variables are:

Water bath distance

Die gap had to be large to be able to thread the material but once threaded it was reduced to about 1". Any gap larger produced a sag and increased to ovality.

Water bath temperature

The water bath temperature with a tank temperature of around 135°F and a tank entrance of 180°F produced the best results.

Die temperature

With having a much larger opening on die then previous test the die temperature played a key variable. Previously we had best results at 400°F to produce a stable product. Having a die opening that is 0.325" gave some unique results. Attempting to keep the temperature near 400°F to try to get a stable produce we experienced the material having a hotter center then exterior. As we attempted to thread the material the cooler exterior material would have an increased melt strength than the interior causing it to separate and cause surging. It also ended up producing a "skin" on the inside of the die of solidified material. We ended up getting the best results by increasing die temperature to match the melt temperature of the material and we had a more even draw down but the extrudate had less stability.

Conclusion

The rPET flake was a challenging material to run. It was difficult to balance a steady product and an even draw on the extrudate. The contamination and multiple grades of rPET posed the most significant challenge to get the material to be processed. This led to the increase in pressures and then the breaker being broken which allowed contamination through which caused a line break.

PRINTING WITH rPET

Print Settings

Basic

Extruder

Nozzle	0.5
Extrusion Multiplier	1
Extrusion Width	AUTO
Ooze Control	ON
Retraction	ON
Retraction Distance	0
Retraction Lift	1
Retraction Speed	600mm/min



Layer

	0.3
Infill	100%
Top Solid Layer	4
Bottom Solid Layer	3
Outline Shell	3

Temperatures

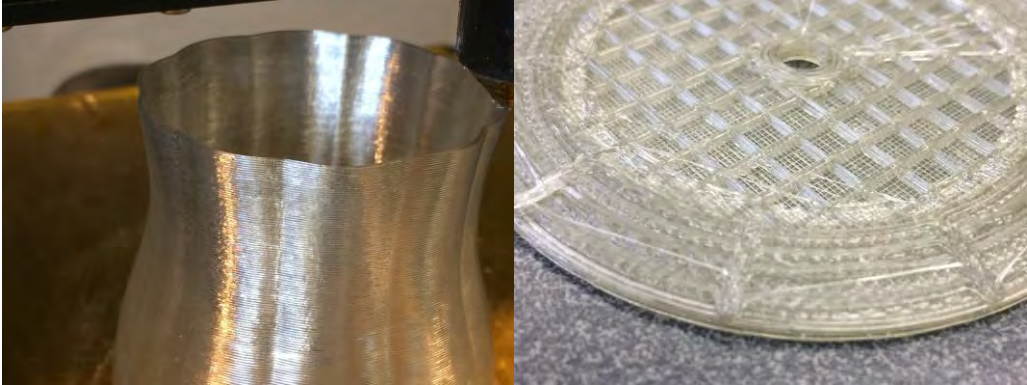
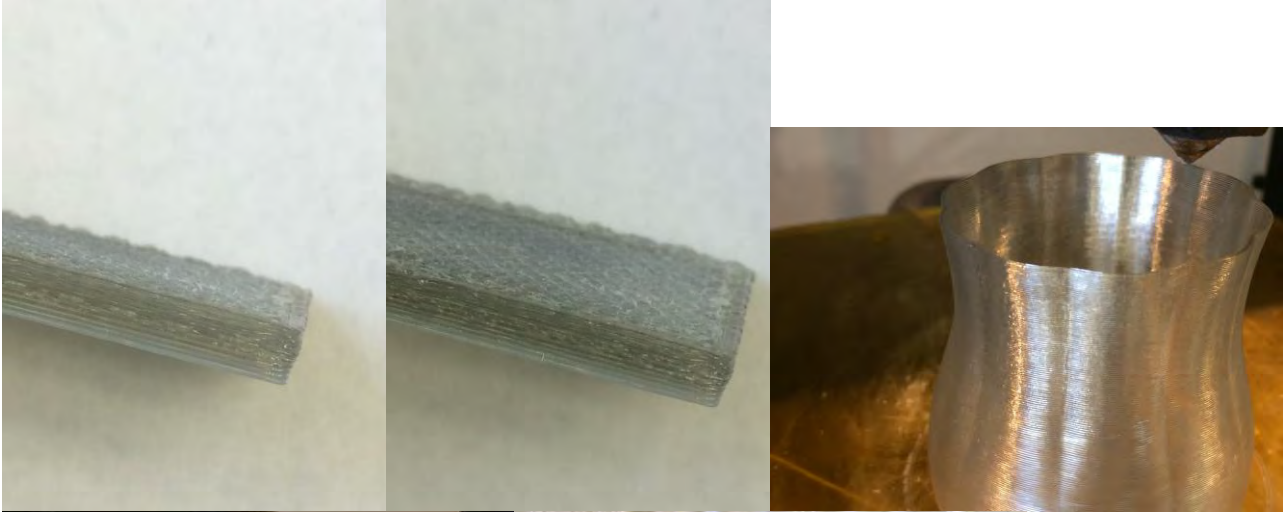
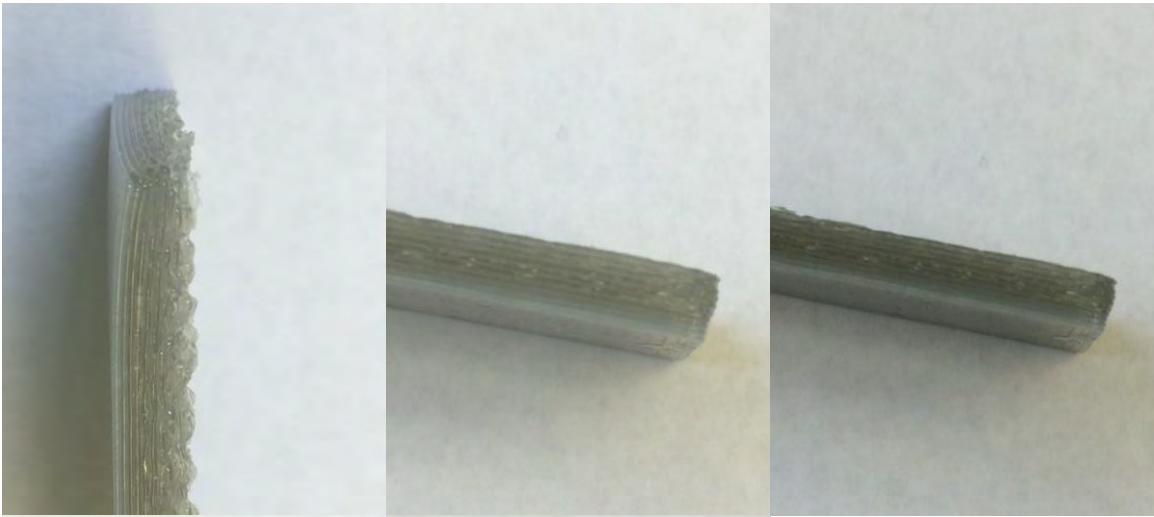
Heated Bed	110°C
Extruder	265°C
Cooling Fan	ON from start

Speeds

Print Speed	1500mm/min
Outline Speed	60%
Infill Speed	80%
Support Speed	80%
X/Y Movement	1800mm/min
Z Movement	600mm/min

Printing RPET

It is a challenging material to print. There seems to be a delicate balance of temperature, speed, part size and density. I have had to alter settings within a print to make sure that it continues to be a successful print. Examples of this would be changing the nozzle temperature and increasing/decreasing print speed. It does not seem to be universal settings. The tensile test model shows that the base layer crystallized, it had few successful layers, then the material started overheating. The second print I reduced the nozzle temperature by 5°C after 4 layers and got better results but the top few layers still showed signs of overheating. Decreasing die temperature again or increasing print speeds would probably produce better results. However altering and testing the parameters and observing the part is critical for print settings adjustments.





Appendix A-6

Conair Report on rPET Filament Extrusion Trials

CONAIR TECH CENTER DOWNSTREAM EXTRUSION RUN

Customer: <u>EMC</u>	Extruder: <u>1"</u>
Contact:	Tanks: <u>11th MT</u>
Date: <u>11-5-19</u>	Pin: Bushing:
Data by:	Calibrator:
Material: <u>PET ~~PP~~</u> <u>FLAKE</u>	Puller: Cutter:

0.75 IV FLAKE

	Run A	Run B	Run C	Run D	Run E
<u>throat</u>	<u>160</u>				
Extruder settings:					
Zn 1	<u>549</u>		<u>550</u>		
Zn 2	<u>520</u>		<u>520</u>		
Zn 3	<u>460</u>		<u>520</u>		
Zn 4	~~400~~				
Zn 5					
Zn 6					
Zn 7					
Zn 8					
Clamp	<u>480</u>		<u>520</u>		
Die 1	<u>480</u>		<u>520</u>		
Die 2	<u>500</u>		<u>520</u>		
Die 3					
Amps	<u>31%</u>				
RPM	<u>76</u>				
Die Pressure	<u>185</u>				
Vacuum (in H2O)	<u>3</u>				
Water Temp (F)	<u>145</u>				
Puller Speed (ft/min)	<u>55</u>				
Puller Ratio:					
Tube Dimensions					
diameter	<u>.112</u>				
<u>oval</u> ~~width~~ <u>length</u>	<u>1.002</u>				
length					

Comments: 1st Wafer 0.180
2nd Wafer 0.145
Die Bush 0.250

Run A Pellets + FLAKE

Run C START TEMP. AFTER FLOW
REDUCE TEMPS TO Run A condition

Technology Bulletin

By Dave Czarnik, R&D/Lab Manager, Downstream Extrusion



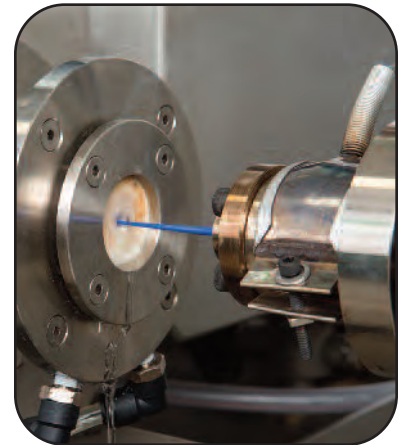
Optimizing Precision and Productivity in Extrusion of 3D Printing Filament

3D printing is taking off as an attractive technology for engineering, prototyping and other commercial uses, and demand for the plastic rod or filament that feeds the printers has never been greater. Yet, the extrusion lines producing filament today tend not to be as efficient as they could be and there is room for quality improvement as well. Taking advantage of experience in the production of high-precision profiles and tubing, including heart and brain catheters, Conair extrusion experts recently assembled a demonstration line to show how using the latest equipment and a complete systems approach can result in significant improvement over the current state of the art.

As 3D printing technology advances and as high-end printing equipment becomes more sophisticated, tight filament tolerances are becoming ever more critical. If the diameter is oversized, or ovality (roundness) varies, filament can misfeed or even jam in the feeder. If it is undersized, the feeder rolls may slip and the flow of polymer to the printer nozzle may be inconsistent. Most printers are calibrated volumetrically; that is, the amount of material delivered through the nozzle is assumed to be a function of the filament diameter, along with the speed at which it is being fed.



Conair extrusion experts recently assembled a demonstration line to show how using the latest equipment and a complete systems approach can result in significant improvement over the current state of the art.



In 3D printing filament extrusion, the latest equipment and a systems approach to can result in significant improvement in the productivity and quality.

An inch of filament of a given diameter is assumed to deliver a specific volume of polymer. If the diameter varies even slightly, the volume of resin deposited on the part will also vary, causing voids and other defects.

The best producers in the 3D-filament industry today are supplying product with tolerances within ± 0.002 inch (0.058 mm) on diameter and also on ovality, although much of the product on the market is more like ± 0.003 inch (0.075 mm).

The demonstration filament line, which was run-off at the Conair Extrusion Development & Testing Lab in Pinconning, MI, has achieved throughput rates of 400 to 600 ft/min (122 to 183 m/min) with ABS, which is three or four times greater than the typical production rate in the industry. Even at these high extrusion rates, quality is also much better than industry norms. Conair has recorded just ± 0.0005 inch (0.0127 mm) variation on diameter and less than 0.001 inch (0.0254 mm) on ovality. The system can also run other materials like PLA, PC, PETG and others.

The extrusion line starts with drying of the ABS resin in a Conair mobile drying and conveying system (model MDCW 100), which also loads the resin to the extruder hopper. A 2-inch Davis-Standard Super Blue extruder was used with the Conair GRH-1.0 extrusion die designed specifically for filament/rod production.

After emerging from the die, the filament passes into a Conair pre-skiner that begins the critical sizing and cooling process. From the pre-skiner the rapidly moving extrusion enters a Conair HTMP multi-pass cooling and sizing tank. The filament makes three passes through the tank before exiting to a precision belt puller and the Conair servo-driven automatic cut-and-transfer coiler. A laser gauge supplied by Zumbach Electronic, positioned between the cooling tank and the puller, continuously monitors diameter and ovality and provides a feedback loop to the extrusion line control to maintain consistent product.

The HTMP tank is a key component when it comes to both quality and productivity. Unlike a standard open water bath, which is still used in many filament installations, the Conair tank combines vacuum sizing and precision cooling in a very space efficient configuration. Cooling temperature in the initial sizing section is controlled separately from the rest of the tank by a Conair temperature control unit. Using warmer water in the vacuum section can help to prevent voids that may form in the center of the filament if the plastic cools too quickly. Vacuum sizing is usually reserved for the production of hollow shapes but Conair has found that the technique can also be used with solid filament to ensure the highest possible level of filament uniformity.

After exiting the vacuum section of the HTMP tank, the filament enters the primary cooling water bath, where water temperature is precisely controlled by a Conair chiller. When the filament reaches the end of the tank however, it does not exit as it



The Conair HTMP tank is a key component when it comes to both quality and productivity.

would in a conventional tank. Instead, it loops around an oversized servo-driven capstan and is directed back toward the extruder again before looping around another capstan and making a third pass through the cooling zone.

Taking up only about a third of the floor space that would be required by a standard open cooling tank, the Conair multi-pass tank still has plenty of cooling capacity so the filament can move at the speeds required to achieve high throughputs. Tight control over water temperature and pulling speed (the large driven capstan actually functions as the primary puller for enhanced precision), together with continuous laser gauging, ensure high-quality filament is produced even at up to 600 ft/min (183 m/min).

An air-wipe removes moisture and the 3-axis laser gage measures and confirms ovality and diameter. A relatively long belt puller is used to draw the filament out of the tank, providing a strong but delicate grip over a long length of the filament. Finally, a self-regulating loop control feeds an automatic cut-and-transfer coiler that smoothly wraps the filament onto production sized reels, ready for transfer to smaller spools in a separate operation.

The Conair Extrusion Development & Testing Lab is available to filament producers who conduct preproduction runoffs on new equipment or develop customized solutions that meet and even exceed the industry's highest standards for quality and productivity.



A Conair automatic cut-and-transfer coiler smoothly wraps filament onto production-size reels, ready for transfer to smaller spools in a separate operation.



Appendix A-7

IC3D Report on rPET Filament Extrusion Trials



IC3D Printers – Prepared by Luke DeBruin for EMC2
Date Prepared: 7-16-2021

Extrusion process pictures

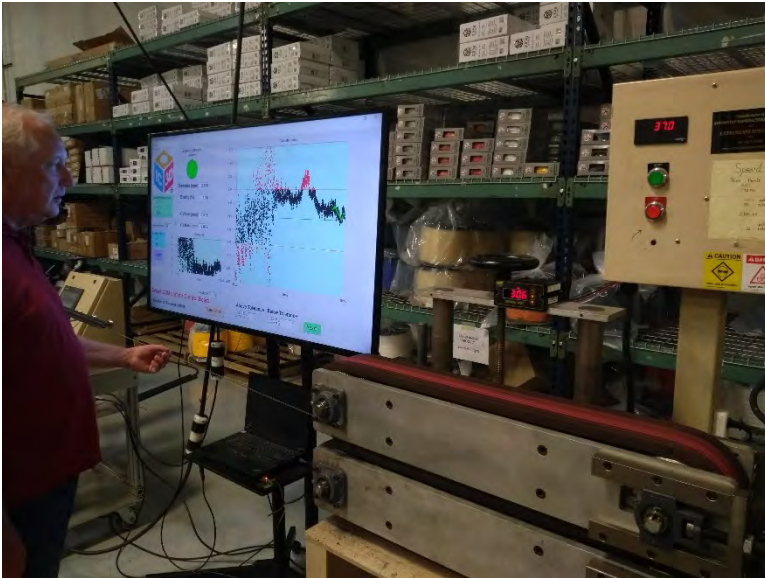


Figure 1: Initial tuning of rPET only (no Kraton)



Figure 2: Extruder settings for rPET only

Video Link to extrusion trials:

<https://photos.app.goo.gl/8MT7RuEXExc6WrWJ9>

rPET Flake w/ 10% TPE

Date Produced: 7-9-21

Material:	NLF 4 MM LNO FLAKE	Manufacturer:	Phoenix Technologies
Grade:		Supplier:	Phoenix Technologies
Lot/Batch:	?		

YIELD REPORT STARTING WEIGHT Had to dry all available >50 lbs

	1.75MM		2.85MM
Start Purge			~5 lbs
Settings Discovery			~7 lbs
Product Yield			3x 1kg spools rPET, 4x 1kg spools Kraton
Out of Spec Product			Not measured
Left over			Not measured
Total			~35 lbs used
Viscosity	Low	Draw down	High ie)
Melt Strength	Lower; acceptable	Die swell	Max allowable for stable ext.
Melting Point	Lowest allowable (450F)	Set up	Normal

Kraton additive processing guide said drying not required, but TPEs normally need to be very dry to get the best processing results. The material was dried in a dehydrator at 155F for 24 hours prior to mixing with the base rPET. Even still, filament exhibited moisture pock marking on surface most likely due to additive not being fully dry. rPET only did not have the same surface blemish issue.

THREADING/START-UP REVIEW

Material string up like normal using slow starting speed and cooling initial few inches of strand.			
Processing Parameters			
Dry Time	12+ hours prior to start	Speed	Medium
Dry Temperature	245°F	Pressure	Low
Tension and winding was easy to achieve desired results with normal set up			

QUALITY

Tolerance	A-B
Ovality	A-
Stability	B-C

PROCESS SETTINGS

	rPET	Startup	rPET w/ Kraton
Extruder	34	4	44
SCREW AMP	8.2		7.6
PULLER FPM	37.7	~20	37.7
MELT PRESS PSI	2330	?	1750
MELT TEMP	?	?	?
SCREEN PACK	20-80-60	20-80-60	20-80-60
ZONE 1 (F)	415	385	415
ZONE 2 (F)	450	420	450
ZONE 3 (F)	425	420	425
DIE (F)	365 (362 actual)	355	375 (372 actual)
WATER HEATER	165F	165F	165F
BATH	138F	138F	138F
DISTANCE FROM DIE	6"		6"

188 feet equal approximately 1 pound of material

Processing speed was approximately 13 pounds per hour

(acceptable speed for commercial process)

SPOOL NUM	WEIGHT	QUALITY	NOTES
rPET only			
1-1	~1kg	A	+/- 0.5mm and 1-2% ovality – typical specs for commercial
1-2	~1kg	A	Same as above
1-3	~1kg	A	Same as above
rPET w/ Kraton			Quality worsened w/ TPE 10% addition (+/- 0.1mm and 2% ovality)
2-1	~1kg	B	Best quality of the Kraton spools
2-2	~1kg	C	Should not jam Taz6; will jam more sensitive machines
2-3	~1kg	C	Same as above; possible intermittent overdiameter to 3.1mm
2-4	~1kg	C	Same as above; possible intermittent overdiameter to 3.1mm
TOTAL	~7kg	B	



Figure 3: Left shows overdiameters removed in post extrusion quality check – rPET alone had far less problems. Right shows closeup of extrusion contaminant from rPET regrind process –appears to be aluminum



Figure 4: Final packaged material provided to EMC2 for pickup

Extrusion Notes

Extruder settings

In my experience running PETG and ABS, I need to maintain around 3000 PSI minimum to get stable extrusion; I chose to adjust the extruder temp profile in such a manner to maximize the pressure before the die while avoiding running too cold. At first the startup temps were acceptable, but were lowered too much which led to the formation of unmelts in the extrudate – the temps of zones 1-3 were slowly raised until these fully melted.

Water bath distance

In order to stabilize the extrusion the die temp being colder and having the reverse extrusion profile led to the best results. The distance between the die and bath is an important consideration but the drooping effect is best mitigated by running the die colder. The Kraton additive further stiffened the material and decreased the droop between the die and the water bath diaphragm.

Water bath temperature

The water bath temperature with a tank temperature of around 135-140°F (138F actual). This material likes a hot bath – ovality issues will arise if the deltaT from the material temp to the water temp is too great. This is a general rule of thumb for low viscosity materials.

Die

Using a 5mm die and having significant drawdown allowed the best specs to be achieved. The cold die temp allowed for extrudate stability between die and water bath entrance. The die temp is critical and lowering to 355F caused die swell which lead to the extrudate swirling as it cooled. This led to instability in extrusion and poor specs; keeping the die just hot enough to avoid this at 365F gave great results. The setpoint for the die was 365, but the actual die temp stabilized and maintained at 362F. Die buildup on the land was a concern and further investigation of this may be necessary if the product is ran for long periods of time as it will slough off and cause intermittent single occurrence out-of-spec.

Conclusion

rPET flake material viability as 3D printing filament is good. The rPET flake as provided posed no issue to extrusion as far as feed throat bridging and material handling is concerned. The material has a lot of powder and further reduction of this powder is desirable.

Processing using a reverse extruder temp profile led to good results for diameter and ovality and stopped un-melts from disturbing the extrusion, but allowed for cooldown of the material to achieve high enough viscosity for stringing the material from the die to the entrance of the hot bath. This material likes a hot water bath (probably could go hotter up to 165F), which would allow for faster extrusion speeds. Material ran as fast as other commercial grade filament production, but with the TPE additive would not meet our filament commercialization specs- would need to achieve +/- 0.5mm diameter tolerance and eliminate extrusion instability. This should be possible with further tuning. Commercialization outlook promising.

The addition of the TPE additive worsen the specs and caused some extrusion instability compared to the rPET alone. The TPE additive is however critical to successful 3D printing characteristics. These can be further tuned out or dealt with without changing the formulation.

Printing with rPET +10% Kraton TPE

Print Settings

Extruder

Nozzle	0.5	
Extrusion Multiplier	1.05	
Extrusion Width	0.6	
Ooze Control	ON	
Retraction	ON	
Retraction Distance	2	mm
Retraction Lift	0.1	mm
Retraction Speed	30	mm/s

Layer

	0.2	mm
Infill	10	%
Top Solid Layer	5	
Bottom Solid Layer	4	
Outline Shell	1	

Temperatures

Heated Bed	80	°C
Extruder	255	°C
Cooling Fan	44	%

Speeds

Print Speed	70	mm/s
Outline Speed	75	%
Infill Speed	80	%
Support Speed	80	%
X/Y Movement	250	mm/s
Z Movement	16.7	mm/s

Printing Notes

Quite forgiving to print for rPET. The 10% TPE additive makes all the difference and helps with layer and bed adhesion once the correct extrusion temperature is achieved. 255-265C gave good results, but 250C was too cold and caused intermittent jamming. At 265C significant nozzle oozing occurred and led to print failure and buildup of blobs on the part. 80C Bed temp 80C worked well using the powder-coated magnetic bed. The TPE also reduces the part warping considerably and increases the overall strength by reducing the brittleness of the rPET.

The ‘3Dbenchy’ that was printed showed some stringing, however the material bridges quite well at 255C and 40% fan. Further recommendations for tuning would be to reduce stringing by increasing the retraction length and speed slightly. Also further tuning the fan speed %, nozzle temp, and /or print speeds can likely achieve similar results.



Figure A: Calibration 'L' bracket printed w/ no infill and single wall

The print shown in the foreground of Figure A was started at 250C nozzle temp and raised to 255C half way through the single wall part. After printing, pliers were used to test the tensile strength of the walls/layer bonding and the 250C portion failed along the layer line indicating poor layer bonding. Farther up the part the same tensile force and side bending force was applied to the 255C portion (see red circle) and the part did not fail – this test was used to determine the appropriate nozzle temp for the printing profile.

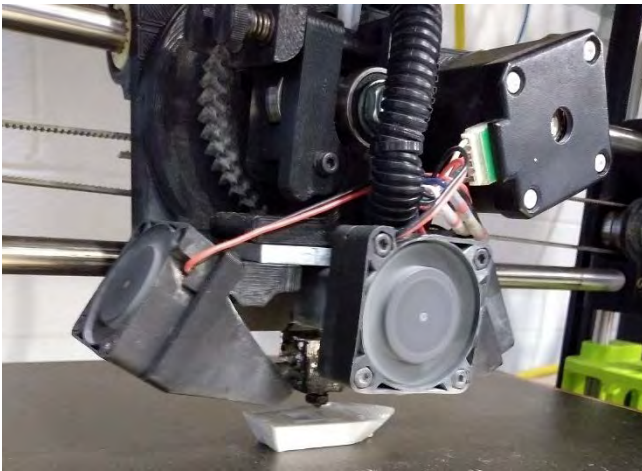


Figure B: 3DBenchy printing as test print on Taz6 w/ 0.2mm layer height and 0.5mm brass nozzle at 255C



Figure C: Benchy front view showing close-up of stringing. Filament has excellent bridging characteristics and captures detail well with given settings

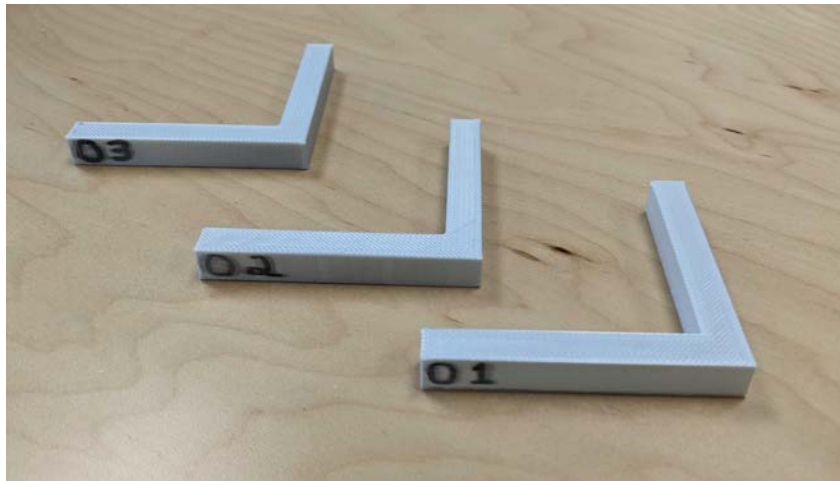
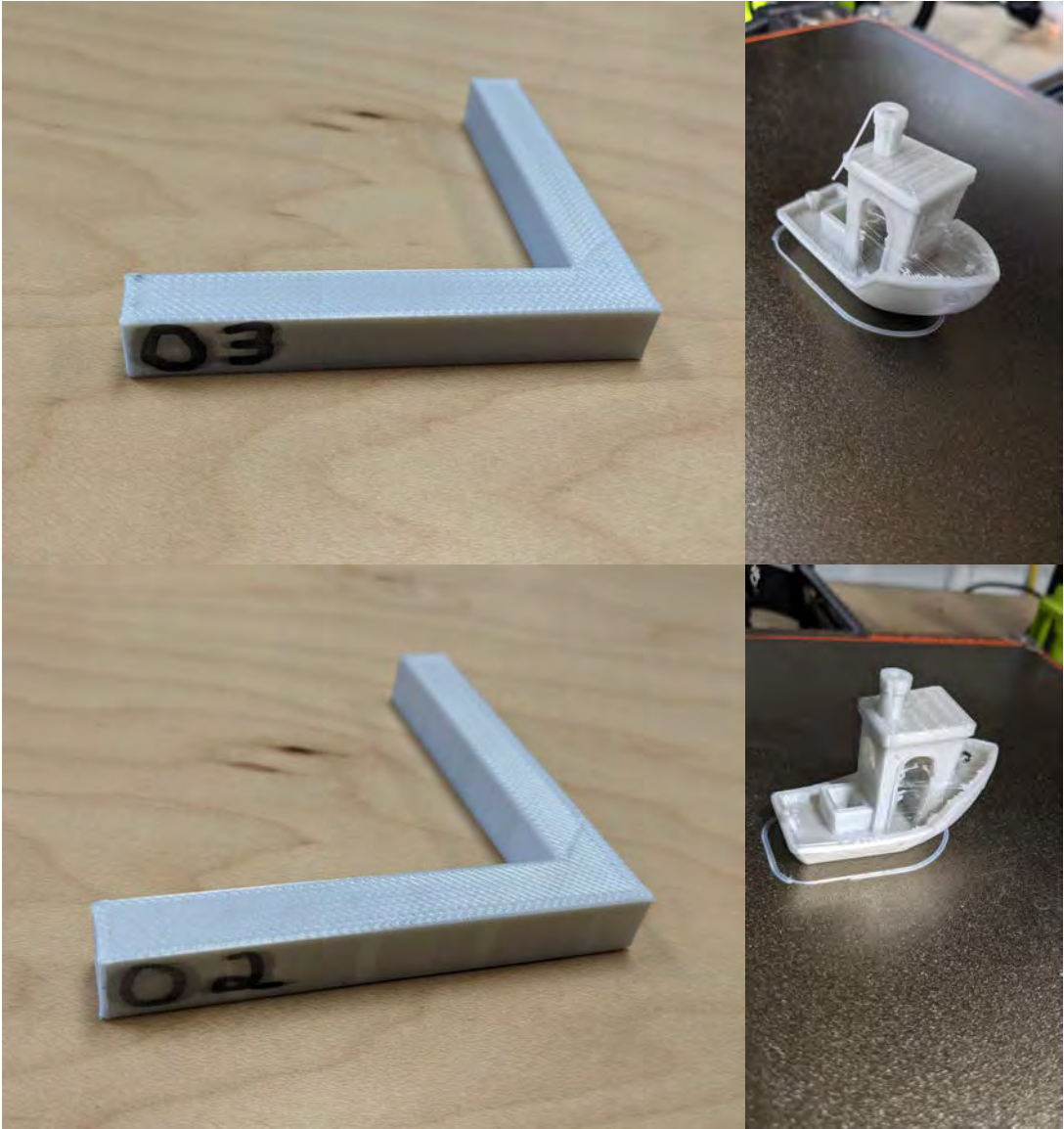
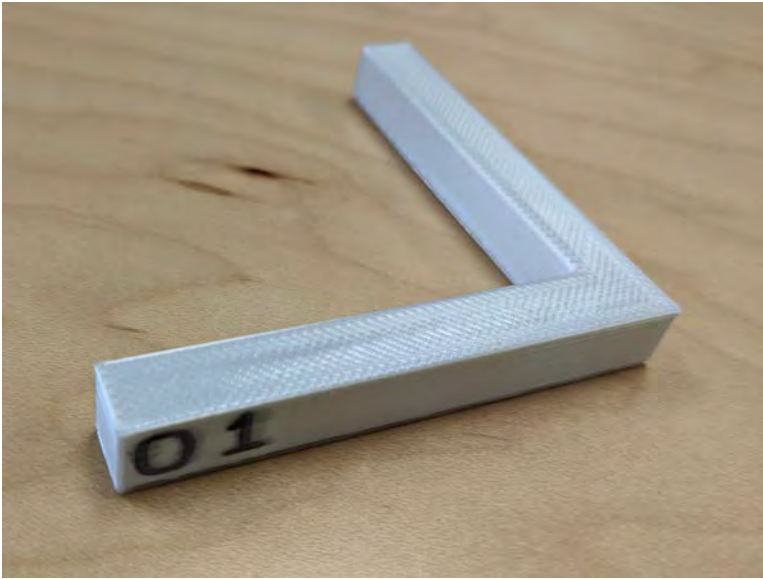


Figure D: Calibration L bracket test prints at 255C nozzle. Sample 1 is single wall w/ no infill. Sample 2 is 10% infill and Sample 3 is 40-50% infill. All printed well and showed very low warp.

Close-ups of printed parts provided to customer:





Appendix A-8

FilaMint Report on rPET 'Filament Extrusion System' for AM

**CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE
INFORMATION**

Access to Appendix A-8 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

Appendix A-9

METSS Report on rPET Plastic Lumber Flow Molding Trials



METSS COMMERCIAL RESEARCH REPORT

Report Date:	June 18, 2019
Customer:	Richard Heggs, Engineering Mechanics Corporation of Columbus (Emc2)
Reference:	Conducting Flow Molding Trials with rPET Materials; Purchase Order 19-04
METSS POC	Jonathan Macarus, Lead Project Engineer

Summary

METSS was asked by Emc2 to conduct flow intrusion molding trials using dried recycled PET pellet and flake material. Emc2 provided the PET material, flow mold, and attachment to mate the mold to the METSS' extruder. METSS conducted preliminary setup trials and a total of 5 flow intrusion molding trials. Prior to running the trials, METSS confirmed clamp connections, adjusted drying temperatures and times, determined the processing temperature range, and prioritized raw materials with Emc2.

Test Parameters

Test Dates: May 1-23, 2019

METSS Staff: Jon Macarus and Mark Docter

Emc2 Observers: Richard Heggs, Dr. Prabhat Krishnaswamy, and Cameron Orth

Materials: PET pellets and recycled flake as supplied by Emc2

Dryer: Matsui Dehumidifying Hopper Dryer DMZ-80

Drying conditions: 270 °F for 12hrs minimum

Extruder: Leistritz 34mm co-rotating twin screw, L/D ~25:1

Table 1. Extrusion Conditions

Material	Feeder Setting	PET Feed Rate		Estimated Fill Time (min)	Extruder Speed (rpm)	Amps (torque)	Temp profile
		(g/min)	(lb/min)				
PET Pellets	15%	346	0.76	2.4	200	15	270°C Flat
PET 4mm Flake	15-30%	365	0.80	2.2	200	10	270°C Flat

Table 2. Flow Mold Dimension and Estimated Fill Requirements

Flow Mold Size (in)	Volume		Material	Density (g/cc)	PET Mass	
	Cubic Inches	Cubic Centimeters (cc)			g	lb
36 x 1 x 1	36	590	PET	1.38	814	1.8

Appendix A-10

American Cierra Report on Mobile Plastic Lumber System Design

CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE INFORMATION

Access to Appendix A-10 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

Appendix A-11

ByFusion Technology Overview



BY FUSION

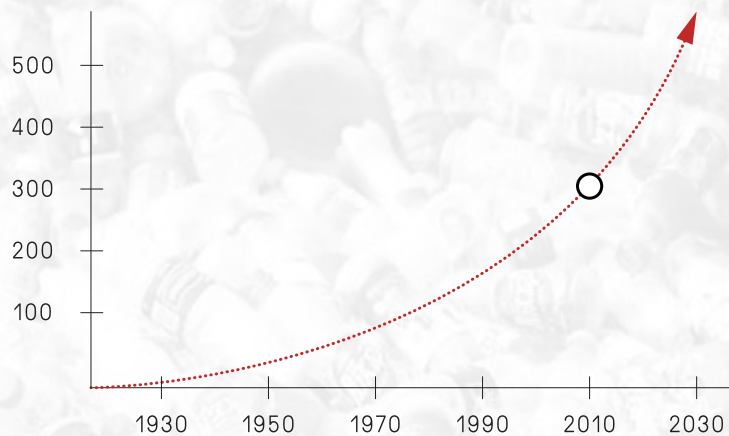
RESHAPING THE
FUTURE



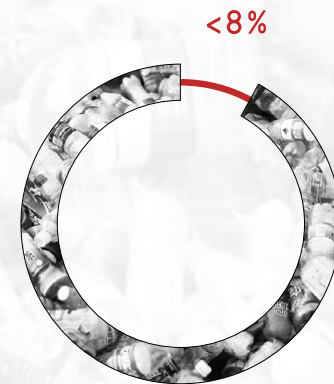
DIVE ★ AWARDS 2016



PLASTIC IS CHOKING OUR ENVIRONMENT

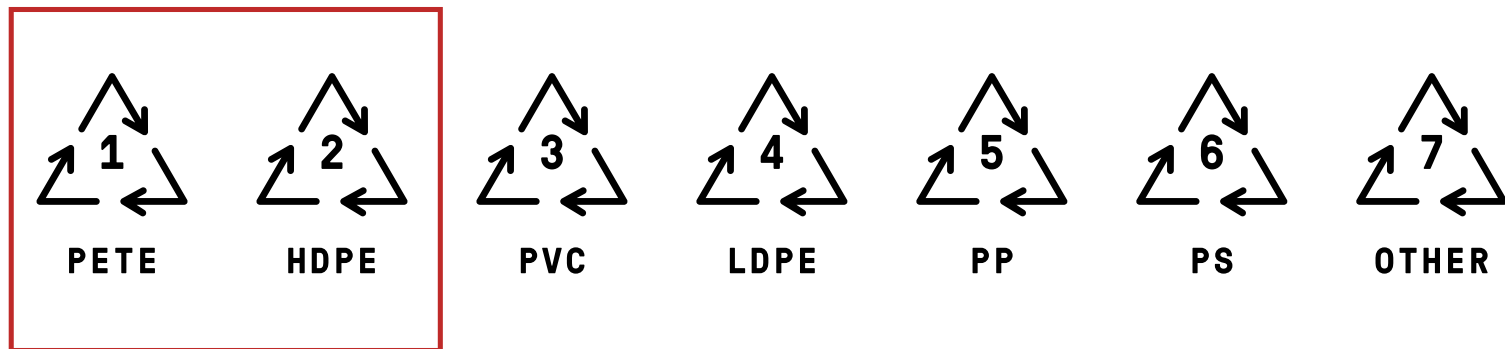


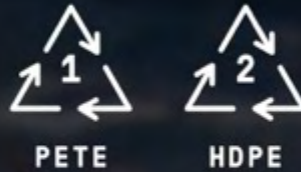
**HUMANS SPEND \$120 BILLION GENERATING
311 MILLION TONS OF PLASTIC TRASH A YEAR**



**AS OF TODAY, LESS THAN 8%
OF PLASTIC WASTE IS RECYCLED**

THERE ARE 7 TYPES OF PLASTIC WASTE





WE CURRENTLY RECYCLE TYPES 1 & 2,
WHICH ARE COMMONLY USED FOR FOOD.

WE ARE NOW STARTING TO SEE WHAT
HAPPENS TO THE OTHER 92%...





THE SOLUTION



PLASTICS NEW LIFE - BYBLOCKS™



ByFusion's Blocker takes any category of discarded plastic and turns it into ByBlocks, an advanced building material that can be used for;

- Housing
- Infrastructure projects
- Utility structures
- Landscaping and walling applications
- Warehousing
- Furniture, commercial products, wall partitions

-- Wherever **non-load bearing hollow cinder blocks** are used --



PRODUCT DIFFERENTIATORS

COMPARATIVE CHARACTERISTICS



CONCRETE BLOCK

BYBLOCK

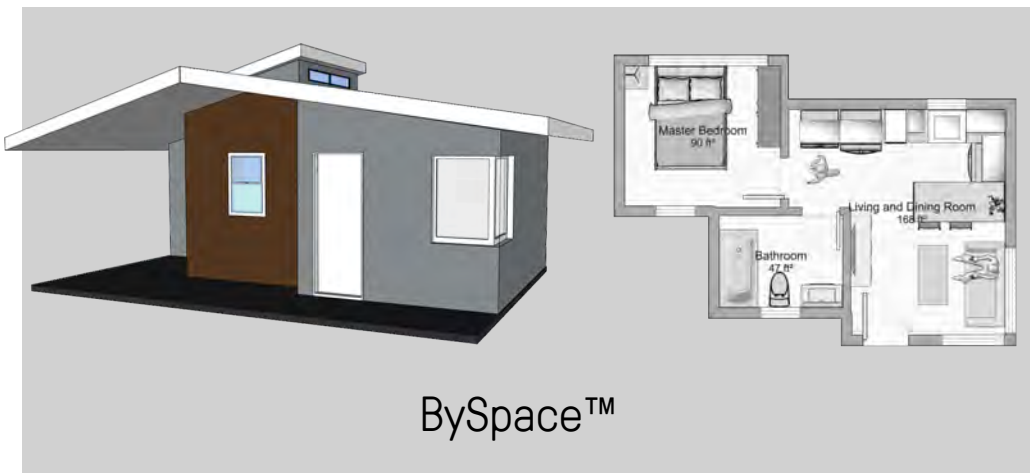
	CONCRETE BLOCK	BYBLOCK
FEEDSTOCK	Virgin Aggregate Sand, gravel, cement	Household and post-industrial plastic waste
STRUCTURAL CHARACTERISTICS	500 PSI Non-Load Bearing CMU*	500+ PSI** (target)
REINFORCEMENT	Rebar, cement	Threaded Rod
CONTRIBUTES to LEED	No	Yes
MOISTURE ABSORPTION	High	Low
THERMAL PROPERTIES	0.75 – 0.96 kJ/kg-K	1.67 kJ/kg-K
BREAKAGE	Yes	No
COST TO PRODUCE	Unknown	\$0.15 – \$0.75

*ASTM minimum requirements for single unit.

Target **non-reinforced single unit based on modulus of elasticity and tensile strength.

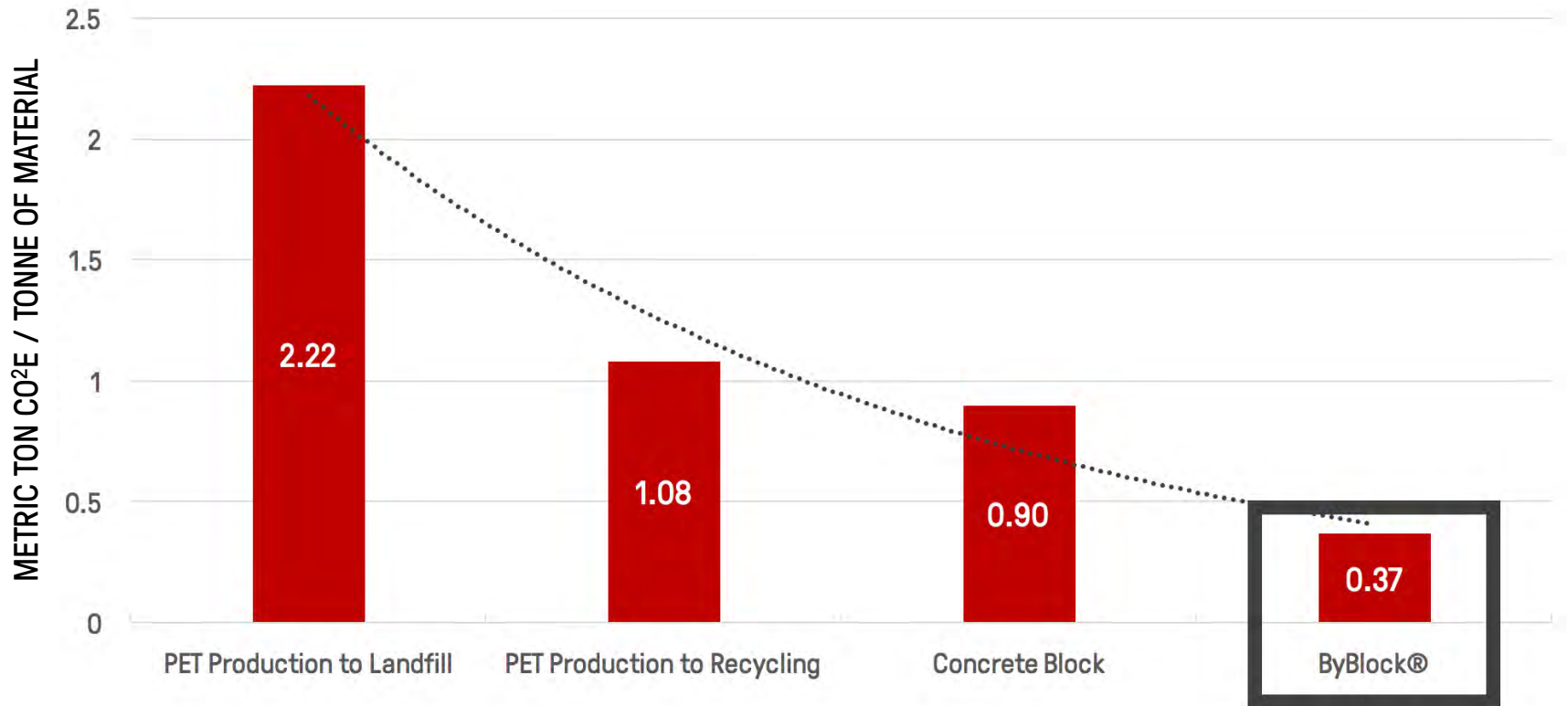
BYBLOCK PRODUCTS

PRODUCT		DESCRIPTION
MARKET READY	Standard ByBlock	8x8x16, 10kg, interlocking
	Flat ByBlock	8x8x16, 10kg, non-interlocking for top rows/wall caps
	.04T ByTable	Indoor/Outdoor side table w/ multiple finishes (Naked showing)
	.09T ByBench:	2-person bench w/ multiple finishes (Naked showing)
	.18T ByBench	4-person bench w/ multiple finishes
IN DEVELOPMENT (projects identified)	1.35T ByBox	60 sqft space w/ multiple finishes (KIT)
	3.0T ByPad	150 sqft multi-functional space w/multiple finishes (KIT)
	8.8T BySpace	300 sqft micro-housing solution – 1 bed, 1 bath, full kitchen (KIT)

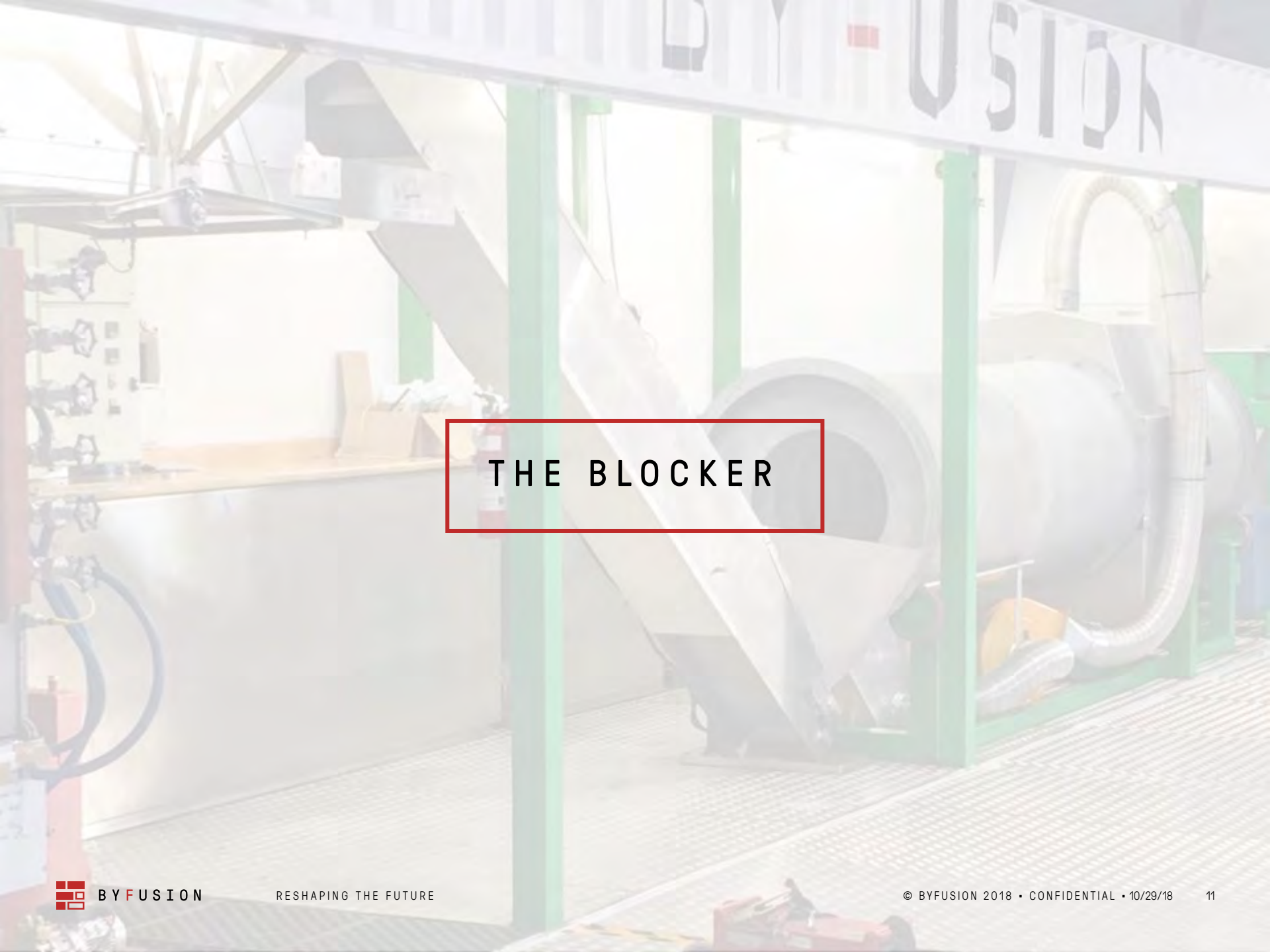


PRODUCT DIFFERENTIATORS

COMBATING GREENHOUSE GAS



Environmental Protection Industry's 2012 report entitled: Documentation for Greenhouse Gas Emission and Energy Factors Used in the Waste Reduction Model (WARM) Management Practices Chapters, Revised March.



THE BLOCKER

THE BYFUSION BLOCKER

The ByFusion Blocker is a modular tool that can link with other ByFusion Blockers and scale to meet the recycling demands of any population.



MAKING **BYBLOCKS** IS EASY

1

DISCARDED PLASTIC
IS COLLECTED



2

PLASTIC IS SHREDDED

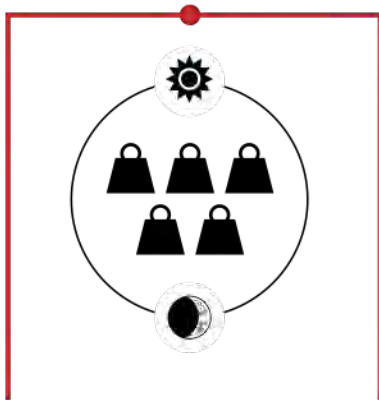


3

PLASTIC IS SUPERHEATED &
FUSED INTO BYBLOCKS



BLOCKERS ARE EASY TO SCALE



A **Community Blocker** can process up to 2-3 tons of non-sorted plastic trash per shift.



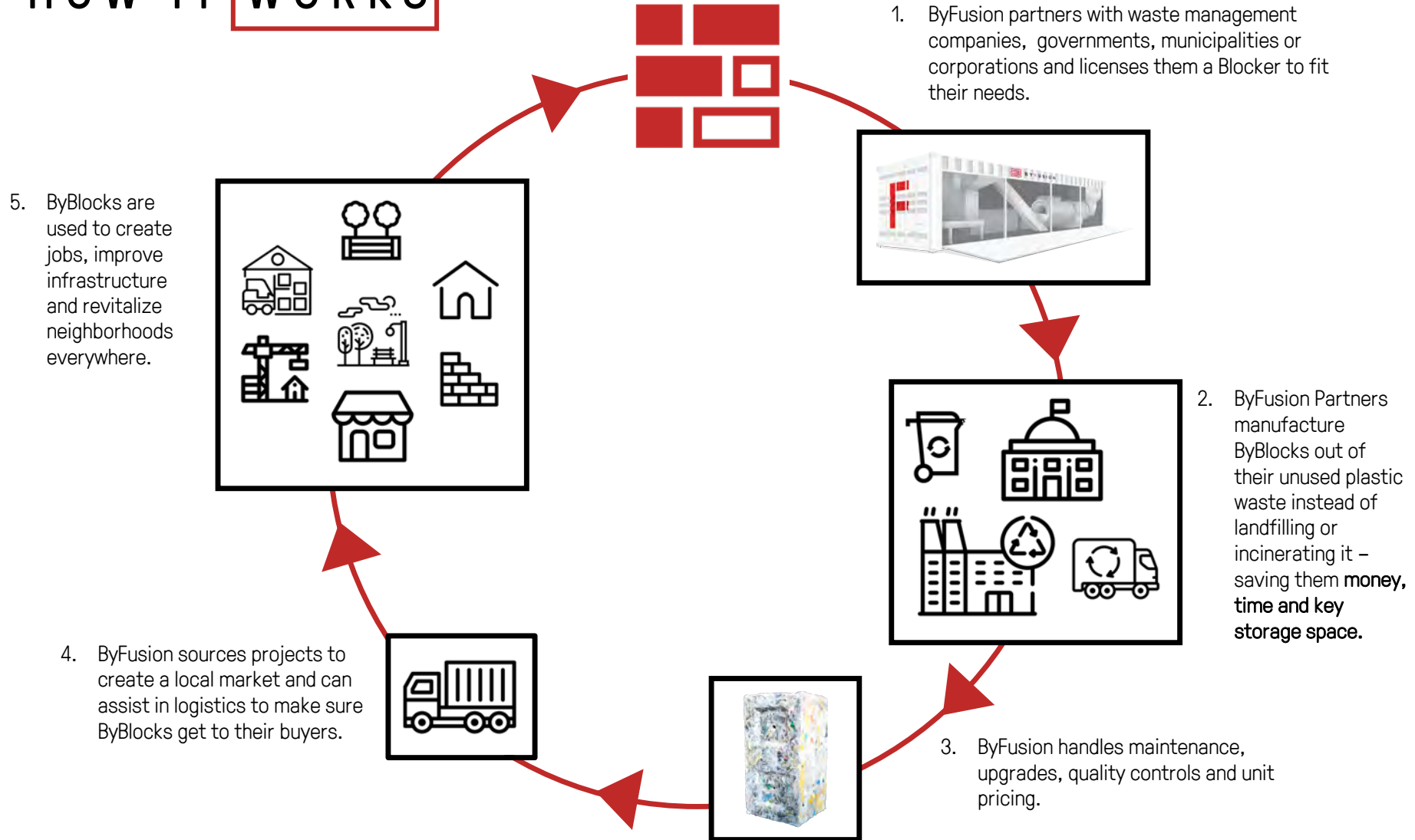
An **Industrial Blocker** system can produce enough ByBlocks per day to build a single family home.



Blockers are built to meet the plastic volume demands of MRFs, reducing operational costs and environmental impact.



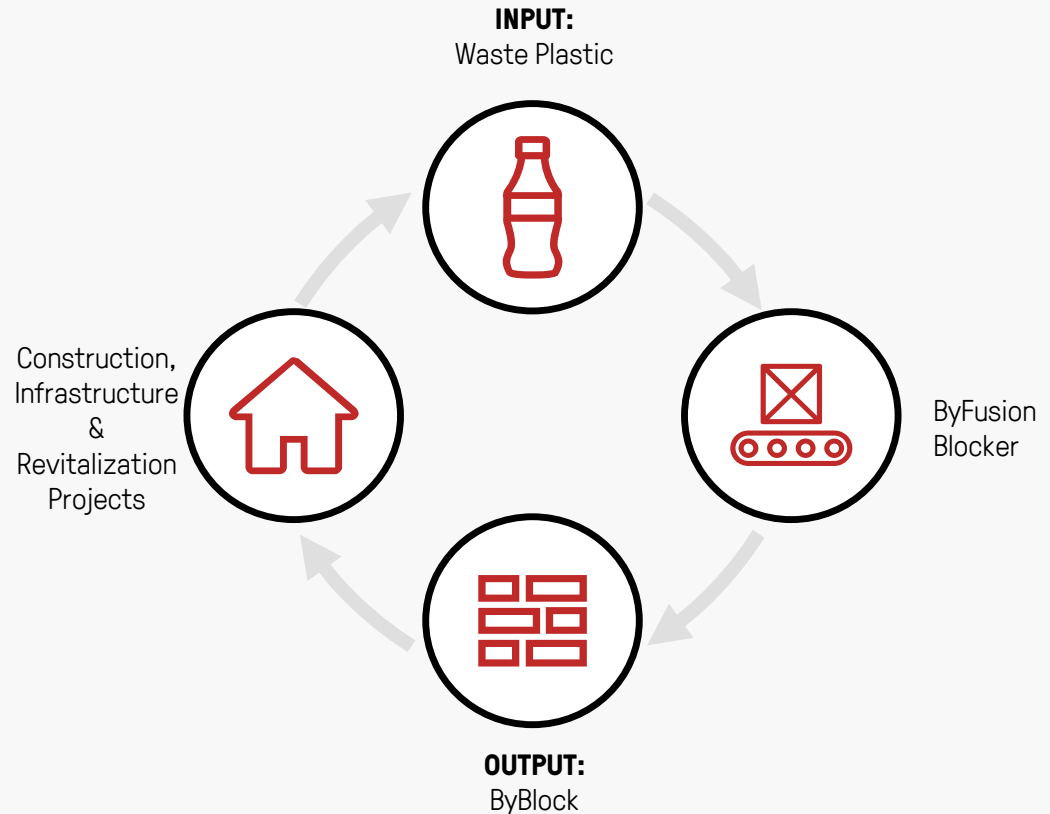
HOW IT WORKS



COMMITTED TO THE CIRCULAR ECONOMY

PLATFORM TO IGNITE CHANGE

- Clean up our planet and protect oceans, waterways and wildlife
- Provide a cost effective, 100% recycled, advanced building material
- Leverage waste responsibility
- Create jobs
- Reduce green house gas emissions
- Give back to the community
- Reduce stress on waste management infrastructure
- Save corporations and government millions of dollars



SUPPORTING THE UN'S 2030 CALL TO ACTION

ByFusion **PROUDLY** aligns with UN's goals for sustainable development to end poverty, protect the planet and promote prosperity.



BY 2034 HUMANS ARE EXPECTED TO GENERATE MORE THAN **DOUBLE THE PLASTIC** WASTE OF TODAY





THAT'S MORE THAN 622
MILLION TONS OF PLASTIC
THAT CAN END UP HERE...





OR WITH OUR HELP,
HERE



BYFUSION

RESHAPING THE FUTURE

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TOGETHER,
LET'S RESHAPE THE
FUTURE



BY FUSION



DIVE AWARDS 2016



Appendix A-12

ByBlock Compression Test Report

Test Results

On

**Compression Testing of
ByFusion's ByBlock Specimens**

Emc² Project Number 18-G232-01

To

SERDP Project Team – Project No. WP 18 - 1047

by

C. Orth



**Engineering Mechanics Corporation of Columbus
3518 Riverside Drive, Suite 202
Columbus, OH 43221
Phone/Fax (614) 459-3200/6800**

August 2019

EXECUTIVE SUMMARY

This document is Engineering Mechanics Corporation of Columbus' (Emc²'s) data record book (DRB) for the monotonic compression testing of ByFusion's ByBlock specimens. The test on the recycled plastic blocks was conducted at room temperature using the loading conditions described by ASTM Standard D6108.

1 DESCRIPTION AND OBJECTIVES

The purpose of this project was to evaluate the compressive properties of ByBlocks for ByFusion by performing compressive tests on test specimens of two different material configurations, 75% PET and 25% HDPE, and 40% PET, 25% HDPE, and 35% other filler material. This Data Record Book (DRB) report describes results of the compression tests on 6 test specimens; 75PET25HDPE01, 75PET25HDPE02, 75PET25HDPE03, 40PET25HDPE01, 40PET25HDPE02, and 40PET25HDPE03.

The test procedure was conducted according to ASTM standard D6108.

1.1 Specimen Descriptions

Table 1: Specimen Names and Material Characteristics

Specimen ID	PET Fill	HDPE Fill	Other Fill
75PET25HDPE01	75	25	0
75PET25HDPE02	75	25	0
75PET25HDPE03	75	25	0
40PET25HDPE01	40	25	35
40PET25HDPE02	40	25	35
40PET25HDPE03	40	25	35

1.2 Fixturing

A 220-kip servo hydraulic, 2-post Universal Testing Frame (UTF) controlled with an MTS FlexTest 60 digital system was used to perform the test on each of the specimens. The test fixture was designed to ensure even loading across the surfaces of the blocks to better simulate real world conditions. The fixture consisted of two flat plates in order to allow for uniform loading, two octagonal inserts, and a plate with two octagonal holes in order to load all faces. Images of one of the loading inserts and plate models are shown below in Figure 1.

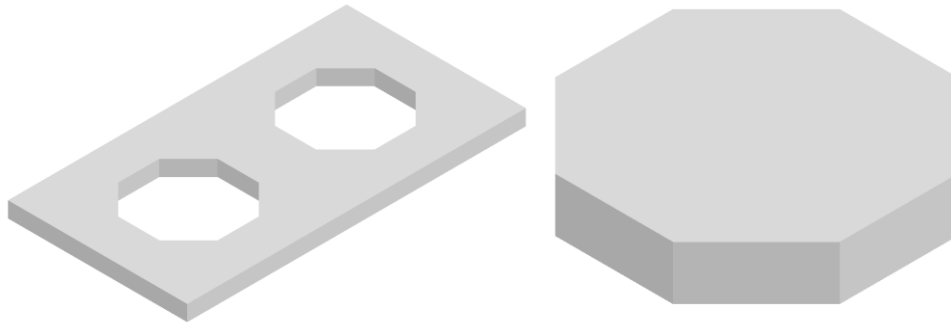


Figure 1: Fixture Images for Compression Test

2 TEST RESULTS

A summary of the test results is listed below in Table 2.

Table 2: Summary of Test Results

Specimen Name	Max Load kip	Max Stress psi	Max Strain	Load at 3% Strain kip
75PET25HDPE01	35.26	303.31	24.07%	4.46
75PET25HDPE02	45.96	395.43	22.57%	6.05
75PET25HDPE03	44.41	382.06	23.69%	6.49
40PET25HDPE01	46.53	387.3	25.71%	1.96
40PET25HDPE02	37.31	321.0	22.46%	5.68
40PET25HDPE03	55.26	475.38	23.10%	7.41

An image of the combined stress strain curves up to 8% strain is shown below in Figure 2. The graph is offset to align the Hookean region of the graph with the origin.

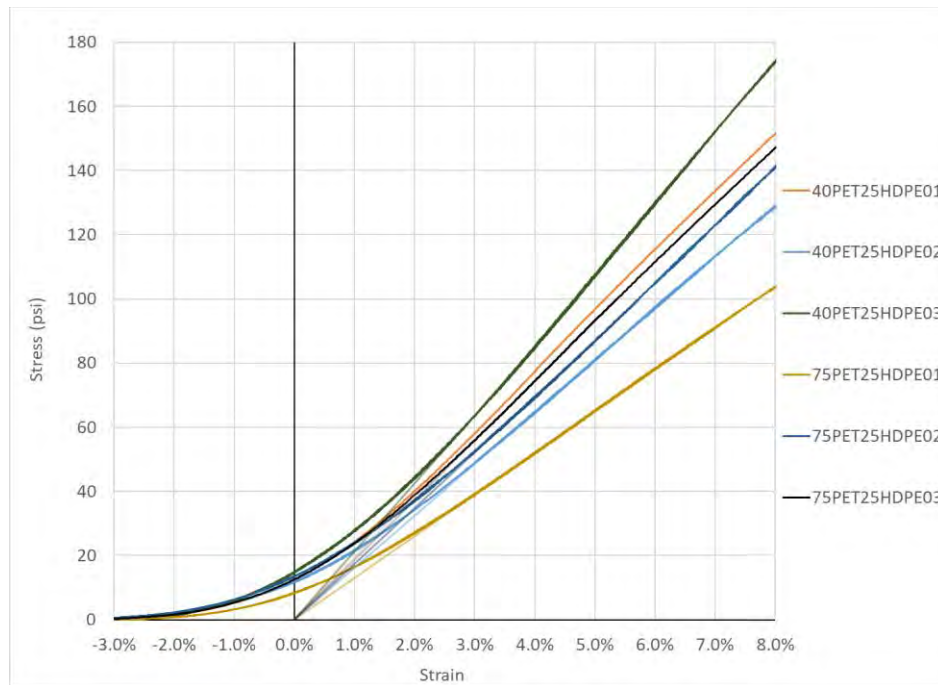


Figure 2: Stress Strain Curves of all ByBlock Tests

Figure 3 through Figure 8 are images of the load deflection curves for each of the tested blocks.

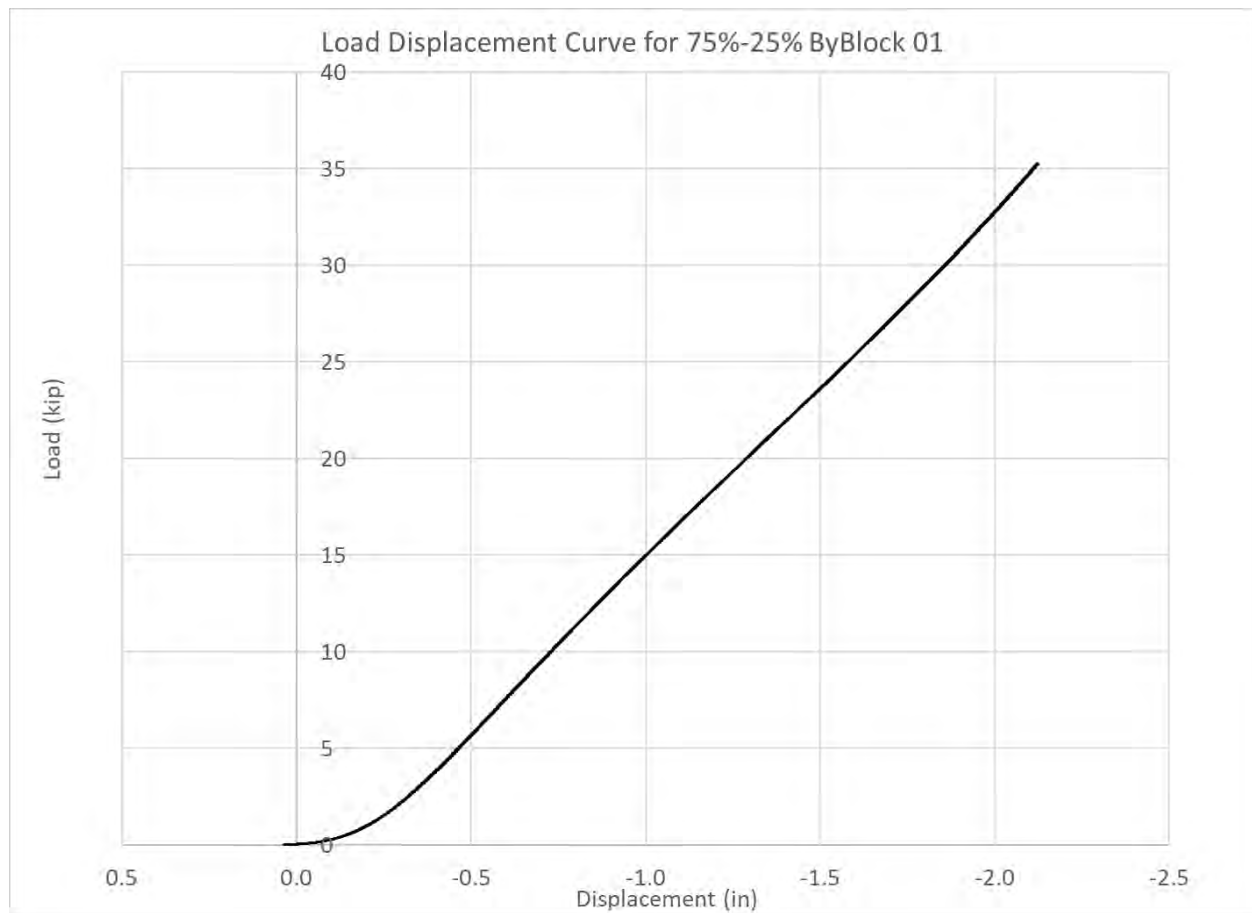


Figure 3: Load Displacement Curve for 75PET25HDPE01

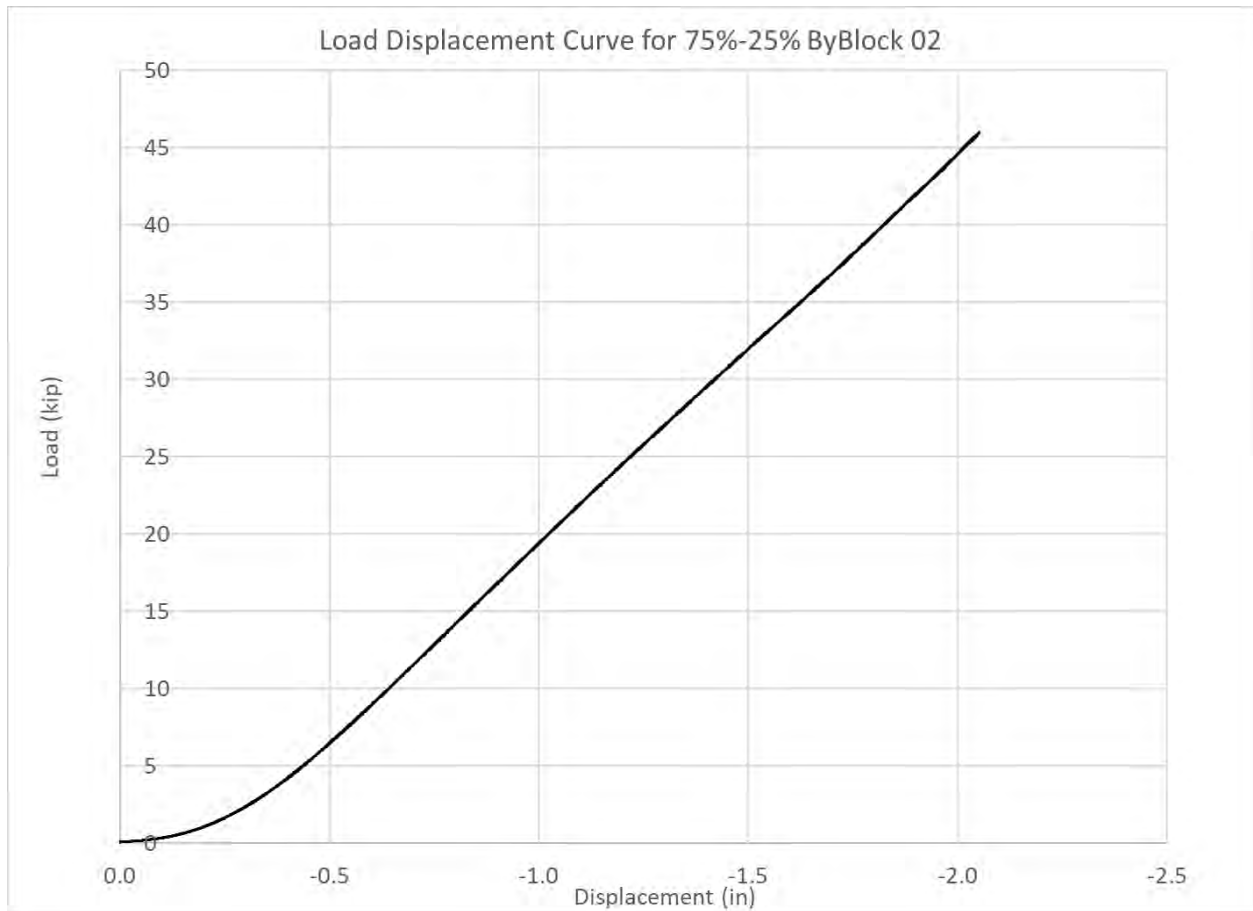


Figure 4: Load Displacement Curve for 75PET25HDPE02



Figure 5: Load Displacement Curve for 75PET25HDPE03

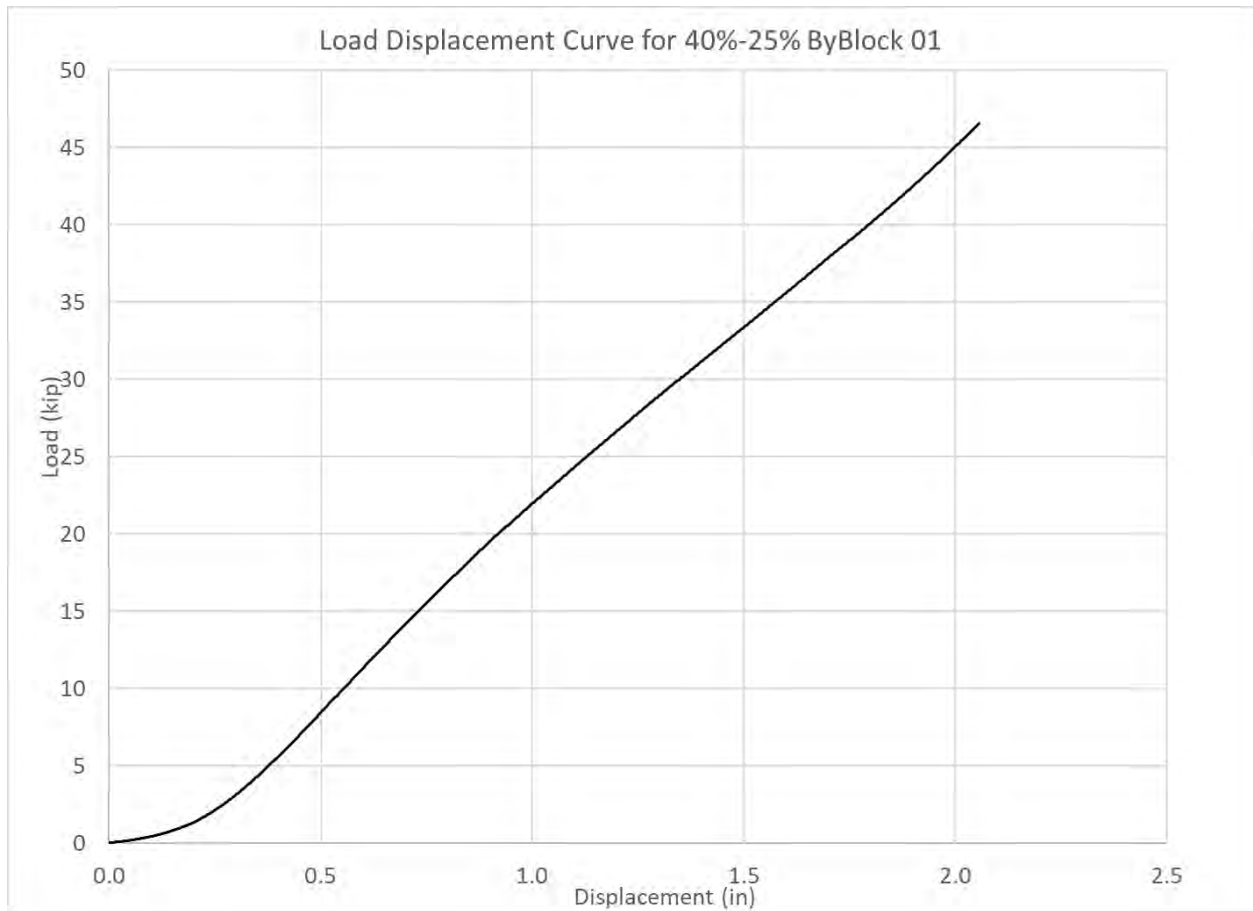


Figure 6: Load Displacement Curve for 40PET25HDPE01

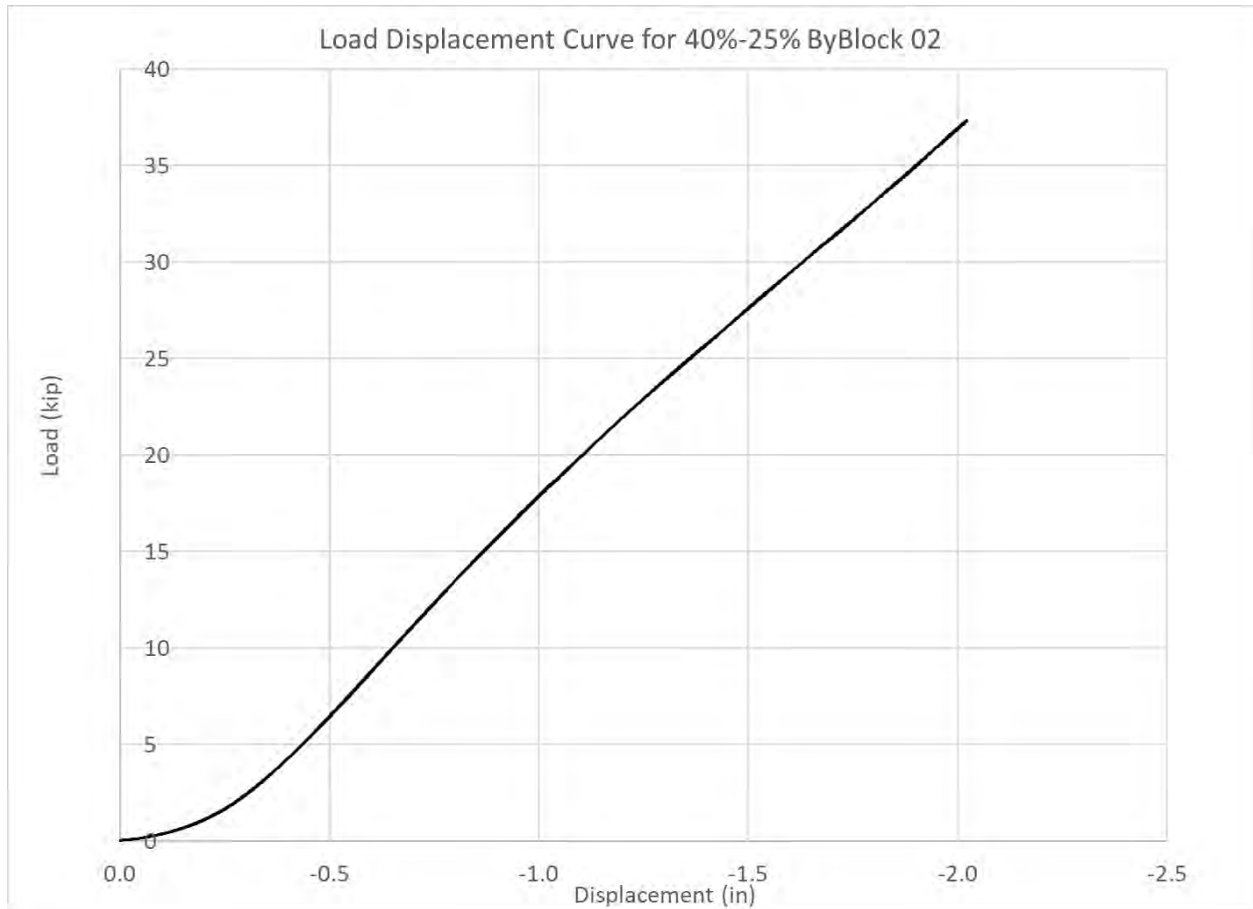


Figure 7: Load Displacement Curve for 40PET25HDPE02

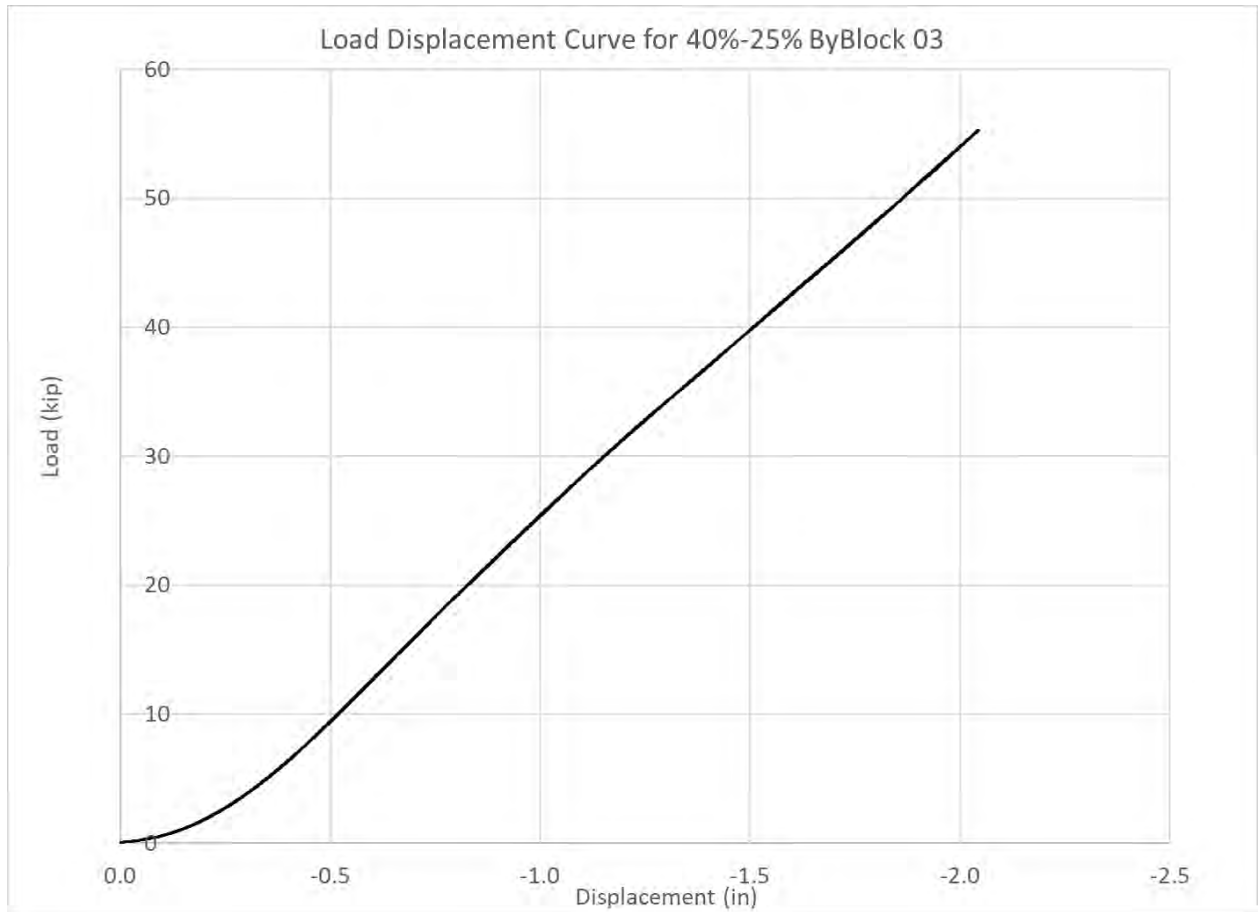


Figure 8: Load Displacement Curve for 40PET25HDPE03

Appendix A-13

ERDC-CERL Report on ByBlock Evaluation

**CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE
INFORMATION**

Access to portions of Appendix A-13 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

MEMORANDUM FOR RECORD

SUBJECT: ByBlock use case concepts

1. This memorandum discusses use case concepts for ByBlock produced by the ByFusion Blocker in support of a Strategic Environmental Research and Development Program (SERDP) project demonstration.
2. The ByFusion Blocker machine produces ByBlock from recycled plastics, including those normally considered not recyclable. ByBlocks have an overall rectangular prismatic shape with nominal exterior dimensions of 16 x 8 x 8 in. (40 x 20 x 20 cm), similar to plain-end Concrete Masonry Units (CMU). The final shape of ByBlock may include a boss and pocket for self-loading walls and/or through-hole bores for reinforcing bars, threaded rods, or cables. ByBlocks weigh approximately 22 lbs. (10 kg), roughly 30% less than typical CMU of equivalent size and design. Based on this claimed weight, each block would require approximately 800 to 1,200 empty 16.9 oz. (500 mL) water bottles, which would have contained about 130 gallons or 1,000 lbs. of water. This calculation does not consider binding agents that might be added to ByBlock during production.
3. The ByFusion Community Blocker ships in a 40-ft. ISO container and processes up to 30 tons of ByBlock per month. This equates to approximately 2,700 ByBlock units per month at 22 lbs. per block, or about 2.7 million water bottles. Drinking water consumption at large, enduring bases in hot climates can exceed 10 million bottles per month. A 10 ft. by 10 ft. section of reinforced ByBlock wall requires 112.5 ByBlock units. A 3,000 sq. ft. structure composed of these ByBlock wall sections would require a total of 2,500 ByBlock units, or about a month worth of production from a Community Blocker.
4. Contingency construction concepts that use ByBlock might have the best chance of success in temporary (0-6 month design life) or semi-permanent (6-24 month design life) horizontal infrastructure. Examples may include, but are not limited to revetments, soil and rock gabions, or personnel denial barriers such as fencing. ByBlock laid horizontally could provide effective ground cover near helipads and airfields to mitigate foreign object debris (FOD), or if structurally capable in compression, could serve as hard stand surface on minimally prepared ground. For permanent (2+ year design life) contingency construction, or for force protection structures such as fighting positions and guard shelters, ByBlock may struggle to meet or exceed blast and penetration resistance requirements of UFC 4-010-01, Minimum Antiterrorism Standards For Buildings, subject to further testing. Design engineers should consider safety and utility systems requirements in UFC 1-201-02, Assessment of Existing Facilities For Use In Military Operations, especially with respect to NFPA 1 Fire Code. Despite these challenges, Command recognizes the need for resilient materials in latrine and shower structures. ByBlock might provide viable alternatives to CMU or steel in use cases which require mold and moisture resistance.

CEERD-CFM

SUBJECT: ByBlock use case concepts

5. Cantonment construction with ByBlock faces challenges of code compliance for enclosed, inhabited structures, as is self-acknowledged in marketing messages released by ByFusion Global. CONUS demonstrations would most likely succeed by targeting “general purpose shed” storage structures (reference UFC 4-440-01, Warehouses and Storage Facilities), open-sided shelters and pavilions, or fencing. If structurally capable in shear, reinforced ByBlock retaining walls could provide a resilient alternative to wood, concrete, or steel in aggressive soils. Projects focused on LEED credits and the requirements of UFC 1-200-02, High Performance and Sustainable Building Requirements, may take interest in the environmental advantages of ByBlock, subject to appropriate life-cycle analysis.

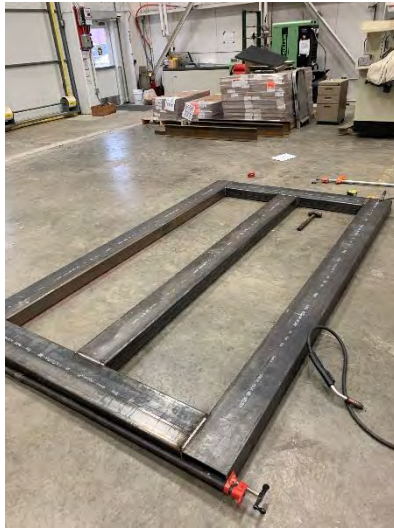
6. Construction decisions in the DoD often hinge on first costs and construction timelines despite calculable return on investment when considering externalities. Proposed designs should demonstrate a cost-competitive alternative to traditional construction methods and materials. First cost comparisons may include labor estimates. ByBlock infrastructure that reduces the time from project initiation to end-use would serve contingency construction or Humanitarian Assistance / Disaster Response (HA/DR) scenarios. Time-to-use estimates should include logistics requirements. Estimates should consider whether a Community Blocker is pre-staged at or near the point of need, or if the mission requires Community Blocker delivery and block production time. Estimates must also consider energy use and the logistics tail of JP-8 fuel for field generators to run the machine. Most DoD applications are outdoors, requiring UV-resistant coatings to protect ByBlock against solar degradation.

7. The POCs for this action are Mr. Jonathan Trovillion, US Army ERDC-CERL, 217-373-4551, jonathan.c.trovillion@usace.army.mil, or Dr. Peter Stynoski, US Army ERDC-CERL, 217-373-3484, peter.b.stynoski@usace.army.mil.

PETER B. STYNOSKI
Research Civil Engineer

The ByBox structure build commenced on 24 February 2022 at the United States Army Engineer Research and Development Center (ERDC) Construction Engineering Research Laboratory (CERL), Champaign, IL. Plans prepared and provided by Michael Kujawa of ByFusion were followed during the construction of the structure.

8"x3" steel box tubing was selected to serve as the foundation frame to enable lifting and relocation of the finished structure. Component pieces were cut to the specifications provided by ByFusion by the steel vendor and then shipped to ERDC-CERL, where it was welded together to form a rigid base for the ByBlock building. Final dimensions of the welded foundation were 70 ¹¹/₁₆ inches by 118 ¹/₈ inches.



Fork pockets that would be used to lift and relocate the structure were welded to the underside of the base. These fork pockets were constructed of C8x18.75 channel measuring 8 inches wide, 2.35 inches tall and 70 ¹¹/₁₆ inches long.

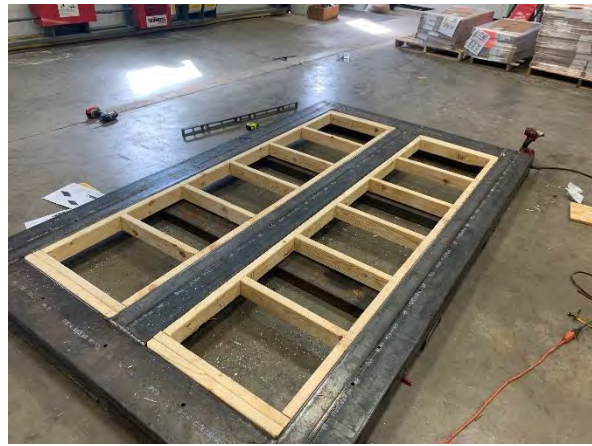


Construction of the building on this steel base required post tensioning rods that would later secure the block walls to the base. The plans called for steel flange nuts to be welded directly to the foundation to facilitate this building technique. However, the specified nuts were only about 3/8" deep and would not allow proper thread engagement by the post tensioning rods. The rods would bottom out in the nuts after they were screwed in about 3 to 4 threads. The decision was made to drill 5/8-inch holes into the top of the steel base in the design-designated pattern so that the post tensioning rods could pass through the flange nuts, and down into the 8"x3" box tubing, allowing proper thread engagement. The 5/8" flange nuts could then be welded over those holes, allowing the post tensioning rods to be threaded into the nuts at full depth.

It was at this point in the build that a discrepancy between the dimensions outlined in the drawing and the physical size of the ByBlocks supplied by ByFusion was noticed. The blocks provided were slightly smaller than those specified in the drawing, measuring 15 1/2" by 7 3/4" and not 15 3/4 x 8". Discussions with ByFusion revealed that the blocks shipped to ERDC-CERL were made in a new mold at the factory. The drawing provided for the build used the dimensions of blocks produced in the old mold. ByFusion provided an updated drawing reflecting this change, but this new drawing also altered the dimensions of the steel frame, dictating it be slightly smaller. Since the foundation frame had already been fabricated, the layout for the flange nuts needed to compensate for the discrepancy between the old and new drawings. The points at which the flange nuts were to be welded to the frame were the same relative to each other as the ones provided in the new drawing, but would no longer be the same relative to the outside edge of the frame, meaning the footprint of the final building would be approximately 1 1/2 inches smaller in both dimensions than that of the foundation.



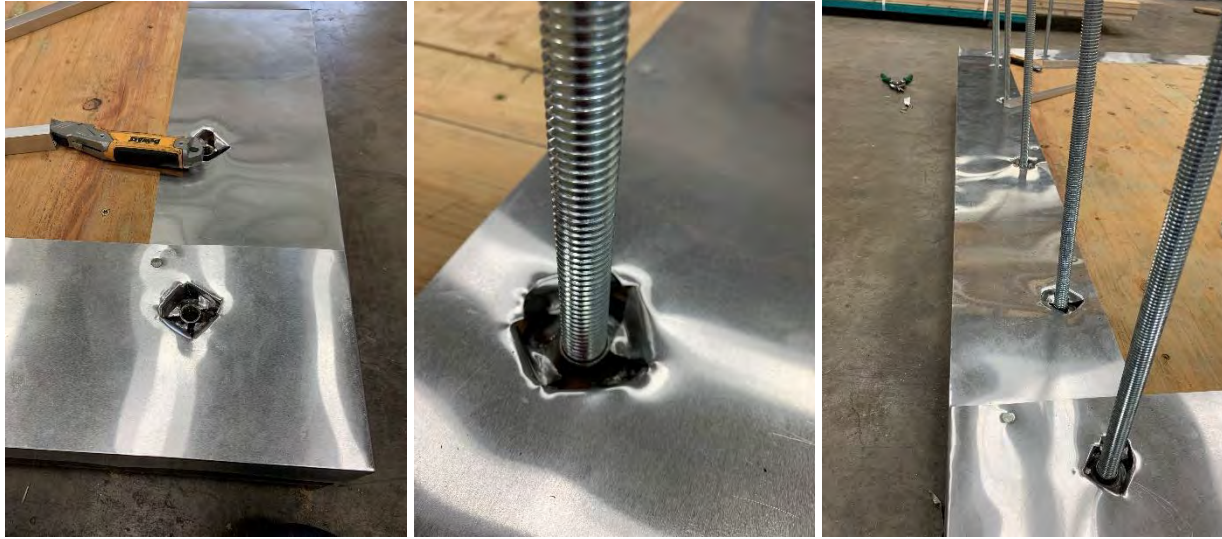
Once these holes were drilled, the flange nuts were welded, and the steel frame was complete. Wooden floor joists were added between the long members of the structure to support the floor. This was accomplished by building 2 joist assemblies constructed out of 2x4" lumber, each measuring $23 \frac{7}{16}"$ by $102 \frac{1}{16}"$. The structural drawing called for the use of Simpson StrongTie joist holders in the construction of these joist assemblies, however, these metal construction brackets were not available at the time the materials for the build were ordered from the vendor. Therefore, 3" all-purpose construction screws were used instead. The two joist assemblies were fitted into the steel base and secured with 2-inch self-tapping construction screws. Because the steel of the foundation frame was so thick, holes for these screws had to be pre-drilled.



The next step in the building process was to place the flooring on the foundation frame. The plans called for the use of 25/32" tongue and groove subflooring, but this was also unavailable though Government sources at the time materials were ordered. So, 3/4" treated plywood available onsite was substituted. Holes in the plywood had to be drilled to allow it to fit over the flange nuts welded to the the steel base. Once cut to fit, it was secured to the base by screwing it to the wood joist assemblies below.



With the flooring complete, aluminum flashing was next installed to keep rain water from entering the structure beneath the first course of block. Pre-bent flashing was not available at this time, so individual pieces of aluminum were cut from a 10 inch wide bulk roll and bent by hand on a sheetmetal brake. Each piece of flashing was limited to 4 feet long due to the limitation of the width of the brake. Once installed, the flashing covered the edges of the plywood flooring and would shed rainwater off the top of the steel foundation. Holes were cut into the flashing around the welded flange nuts and the post tensioning rods were inserted.



A heavy bead of silicone caulk was placed around the outside edge of the foundation beneath the first layer of block to keep rain water from penetrating the structure beneath the wall.



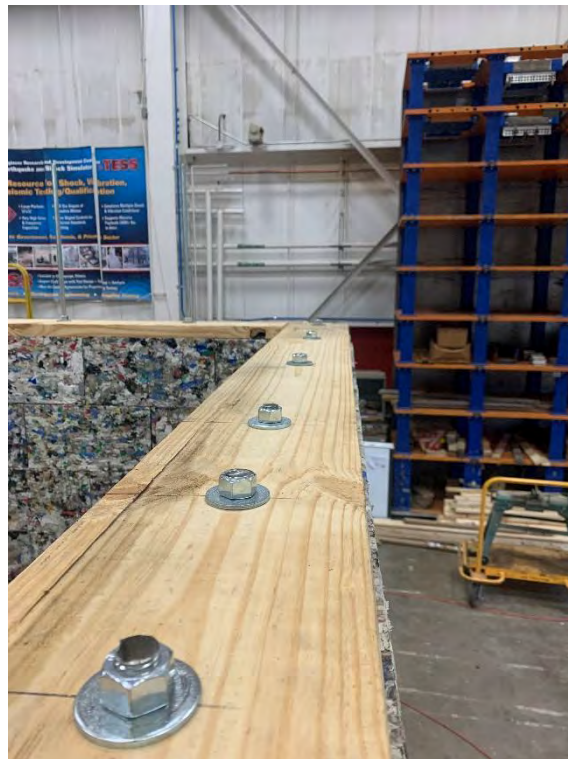
These blocks are constructed in such a way that the short bosses on the top of each block fit into a corresponding recess in the bottom of the block placed above it, effectively locking the blocks together under self-weight. There are holes in the center of these features through which the post tensioning rods pass. Blocks were lowered down over the rods in a pattern dictated by the drawing provided by ByFusion.



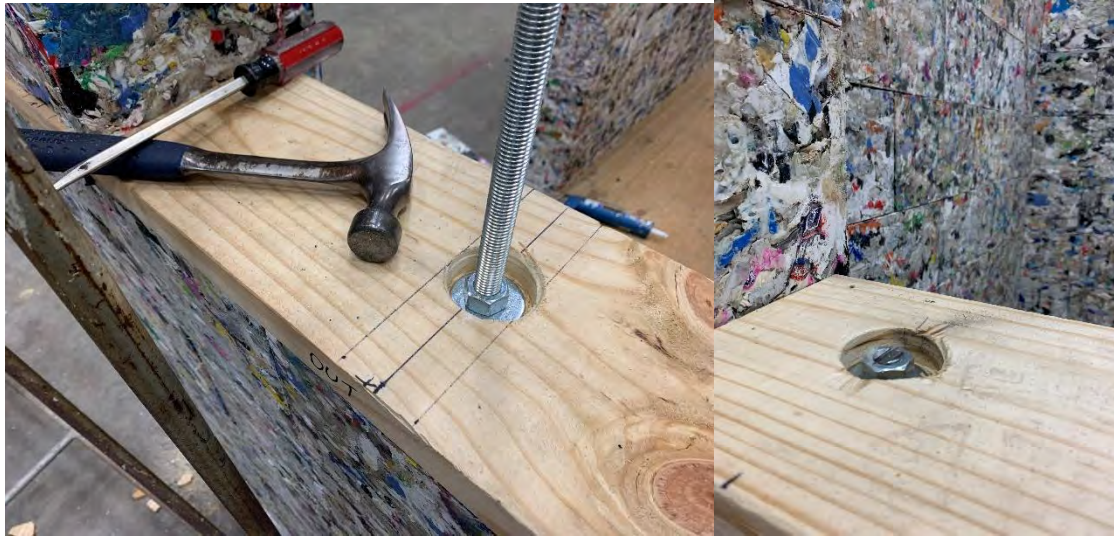
Once the stack is completed and the dimensions of the building are reached, this technology requires post tensioning to keep the walls in place. To accomplish this, 2x8 structural lumber was drilled with the same post-tension rod hole pattern as the floor, and then lowered over the rods sticking out of the top of the block walls.



Washers and nuts were then threaded onto each post tensioning rod and tightened to the point at which each wall was exactly the same height across its run, heeding a minimum torque specification provided by ByFusion. This would ensure a level surface upon which the roof would be constructed. The excess rod was then cut off.

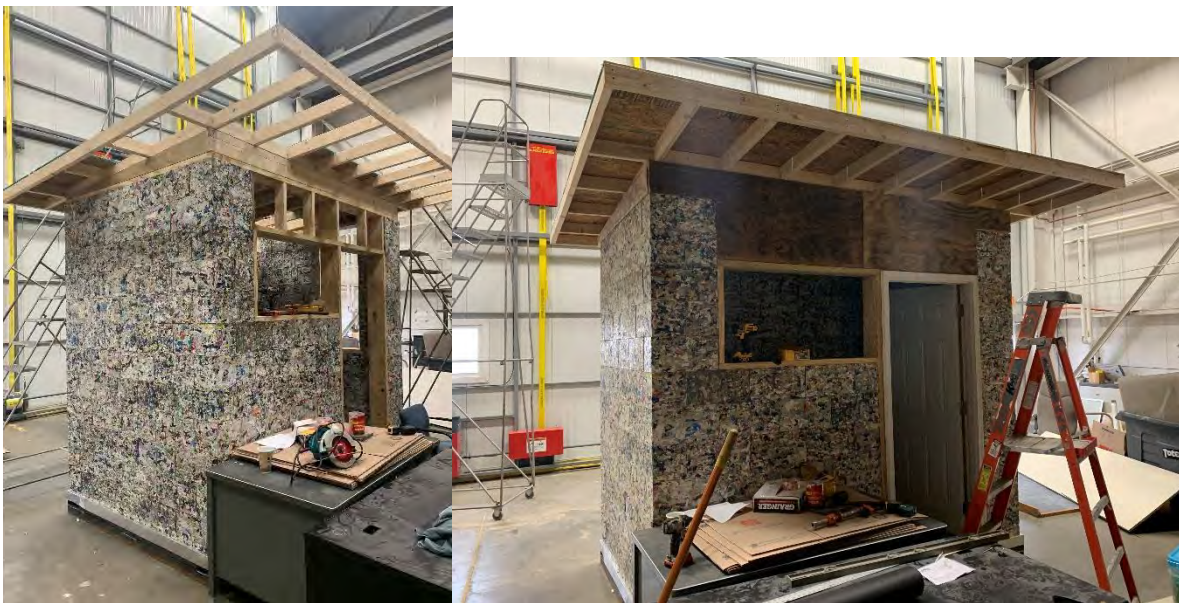


The sills of the two window openings were finished in a similar manner. Since a window would sit in these openings, the post tensioning nuts had to sit down in a counter sunk hole, produced by a spade bit, large enough to accommodate them so as not to interfere with the window installation.



Customary wood-framed building techniques were used to finish framing the walls above the window openings and the rough opening for the door.

The roof of the ByBlock shed was also finished in traditional wood-framing method. A 10'x12' roof section was built on the ground using 16" on-center construction. It was then lifted with a crane and placed on top of the ByBlock structure. However, slight changes had to be made from the drawing during the installation of the roof because the post-tensioning nuts interfered with the placement of the single-pitch low-sloped roof. The side plates that were supposed to facilitate the slope of the roof were to be cut from 2x4 lumber at an angle of 3 degrees according to the drawing. But when the roof was placed on the structure with the crane, several structural members of the roof landed directly on the nuts post-tensioning the back wall. This would not allow the roof to sit properly on the plate. So the side plates were recut from 2x8 lumber instead, allowing the down-slope portion of the side plate to sit higher, and the roof to clear the nuts at the top of the wall.



Once the roof was secured in place, It was sheeted with ½ inch Oriented Strand Board (OSB). Plans called for metal roofing to be installed over the OSB, but this material was not available though Government sources at the time of ordering. A traditional 3-tab shingle roof was installed.

The door and the two windows were installed in the rough openings. Vapor barrier and vinyl siding was used to cover the ByBlock at the recommendation of the manufacturer to protect the recycled plastics from the elements. Installing vapor barrier and siding was the same process as on a traditional lumber structure, with no special skills required, though sometimes fasteners that did not grip or did not sink required a second placement.



The total time to build the structure was approximately 8 days and required 2 moderately skilled vertical construction laborers. The total budget for the materials and supplies, excluding ByBlocks, was approximately \$5,000.



Lessons Learned:

Overall, the construction of the ByBlock building was a straightforward endeavor. However, there were a few challenges to overcome during the build, mainly related to the hybrid construction methods used, not the ByBlock technology itself.

- i. First was the problem with the spacing of the blocks and the placement of the post tensioning flange nuts. Care should be taken to ensure measurements are accurate before construction begins, or the blocks will not fit over the post tensioning rods properly and could leave sizable gaps between the blocks that would require caulking or filling to achieve adequate insulative properties for military use.



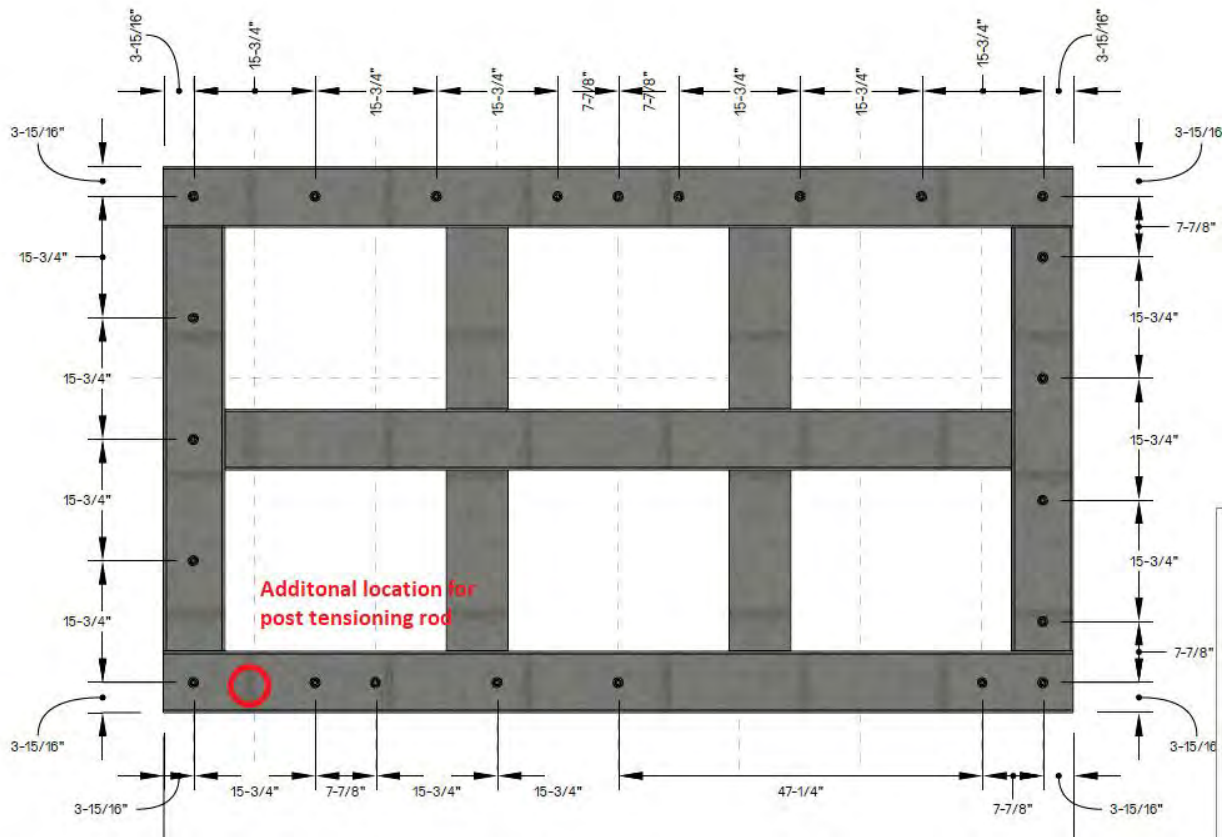
- ii. The second key issue was the post tensioning nuts at the top of the wall. Their protrusion out of the top plate must be considered when designing the roof to sit properly on the structure. Alternatively, they should be recessed into the top plate similarly to those in the window sill plates.
- iii. We encountered some inconsistencies in density from block to block, as well as voids or minor protrusions across the faces of the blocks. We noticed that when screwing the lumber to the block while framing the rough openings for the door and windows, some blocks would hold the wood with a 3 inch construction screw, but others would not. The driver would strip the screw right out of the plastic block and the screw would have to be relocated to a place that would hold it firmly. In other cases, large pieces of hard plastic, or even aluminum cans, prevented appropriate depth of penetration of the fasteners. Expect to use more screws than might otherwise be necessary when combining traditional framing techniques and materials with the ByBlock technology. Tape may be required to repair holes torn in the vapor barrier by plastic protrusions.

- iv. ByFusion prepared and shipped all of the blocks necessary to construct a building of this size. Most of the blocks were standard 15 ½ by 7 ¾ inch dimensions. Due to the shape and openings in the building, some specialty blocks were required. These included half blocks and special blocks that were set at the ends of the window sill plates that extended into the walls per California building code. The shipment to ERDC-CERL was short one half-block and one of the specialty blocks for the window sill plates. Fortunately, there was a band saw on site that was large enough to accommodate these blocks, so we were able to cut the two missing blocks out of the extra standard blocks that were supplied. Careful inventory of the required blocks should be taken at the start of the project to ensure all the necessary pieces are present at the job site. Labeling or color-coding by the manufacturer would significantly aid the inventory process.
- v. Some environmental compliance stakeholders have expressed concern about general cleanliness of used bottles, bio-contamination of the surrounding environment should the blocks be shipped globally (reference photo of insect remains), and release of microplastics to the environment surrounding an emplaced ByBox (reference photo of detritus after stacking). ByFusion already recommended resolutions to each of these concerns for future projects, including the use of drop cloths during construction, and reiterated their official, written compliance certifications against the toughest environmental restrictions in California building codes.



- vi. One final recommendation from a design perspective would be to place an additional post-tensioning rod in the corner of the building if a window is to be installed within 2 blocks of the corner, as annotated in the drawing below. Our building was small, and the window at the front was spaced only 1 and ½ blocks from the front left corner of the building. There was no second post tensioning rod placed at that corner. This made the thin column of block to the left of the window relatively weak and prone to deformation as compared to the other corners of the structure. The corner to the right of the door opening had two post tensioning rods running up through it. Even though that part of

the wall had only one block extending out to create the wall at the front of the building to the right of the doorway, it seemed more structurally sound because of the second rod. The single rod at the left corner also made it difficult to level the top of the front wall because the plate at the top of the wall had no post tensioning rod to tie the two side walls together. The plate had to be screwed to the top block in that corner with quantity 5 of 3" construction screws. The two short walls are only tied together at the top by the roof structure.



BYFUSION COMMENTS TO "LESSONS LEARNED":

- I. Rod distances are constant at $7\frac{3}{4}$ " or $15\frac{1}{2}$ " centers, depending on the required rod placement. Some minimal gaps are to be expected (1 mm - 2 mm) between ByBlock ends. Top and bottom seams will be tight from the post-tensioning. If no additional insulating panels are to be installed on the exterior and gaps are to be removed completely, a silicone or foam insulation can be applied during ByBlock installation.
- II. ByFusion has since adopted a double top plate to solve this issue with overlapping at corners. This allows for full lumber height to support the post-tensioning, while providing the additional height to achieve a flush installation of post-tensioning bearing plate/washer and nut.

Alternatively, the framing can be designed around the top end of the post-tension hardware to remove any potential interference.

- III. *General fastening to ByBlock requires a minimum of 2" thread engagement. Typically, this provides the thread length required to fasten past any slight void areas of plastic. Attempting to fasten into ByBlock at the seams should be avoided. When securing framework to ByBlock for door jambs or window bucks, a minimum #12 - #14 lag screw is recommended with a minimum 3" thread engagement.*
- IV. *ByBlock can be cut using standard cutting tools: Table Saw, reciprocating saw, Radial arm saw, chainsaw, guillotine saw. Always use PPE in accordance with safe practices when using power tools including gloves, eye and hearing protection. ByBox/ByPad "DIY Kit" projects such as this are being developed which will manage careful part identification and inventory, as well as required fasteners and assembly materials.*
- V. *ByFusion Blocker System's steam sanitizes the ByBlock as part of its process. Advancements in our fusion process has resulted in a more rigidity which translates to mimized detritus during the stacking process. As per normal health & safety recommendations, a cut station with dust collection is ideal to capture and contain any plastic material as a result of the cutting of ByBlock. This material, along with any other detritus can be returned to ByFusion and used to produce more ByBlock.*
 - A. *ByBlock holds a UL Certified Environmental Product Disclosure, meets GREENGUARD and GREENGUARD GOLD criteria for formaldehyde, total aldehydes, and CREL/TLV levels, and is CA Section 01350 Compliant. Testing followed **CDPH Standard Method v1.2**, "Standard Method for the Testing and Evaluation of Volatile Organic Chemical Emissions from Indoor Sources Using Environmental Chambers," and UL 2821, "GREENGUARD Certification Program Method for Measuring and Evaluating Chemical Emissions from Building Materials, Finishes and Furnishings Using Dynamic Environmental Chambers."*
- VI. *ByFusion has since adopted three rods per corner as part of normal design considerations. This, combined with the inclusion of double, overlapping top plates ensures all corners to be easily squared and secured without the need for unnecessary fasteners. This completed assembly also provides a rigid framework which aids to hold critical dimensions between base and wall assemblies.*

Attachment 1 – Estimates of Waste PET at FOBs
(input from US Army Corp – ERDC-CERL, Champaign, IL)

1 Camp size

What size of forward operating base would be appropriate for a PET recycling machine? Because this is specialty hardware, it's reasonable to assume that this technology would be used only in locations that are large enough to have contractor support available. The larger the camp, the higher proportion of civilians, up to 50/50 in the very largest. This means that a minimum size of camp would be battalion-sized: 1,000 personnel, or PAX (PAX means total people including military and civilians). This would be the smallest camp where contractor support would be expected. We will have to determine appropriate size of camp vs. machine throughput, i.e., given physical size constraints, what throughput would be desirable?

For comparison purposes, it would be good to scope out camps of 2000, 6000, and ten thousand as benchmarks.

2 PET Generation

2.1 PHC Guidelines

The Army Public Health Command publishes guidelines for soldier water intake (**Figure 1**). We can take these recommendations as a baseline and calculate expected bottle generation rate at different consumption rates (**Table 1**). For our purposes, work with the baseline number at 0.5 liter/hr, over 12 hours per day. This yields the number of 0.5 liter bottles per day across a camp.

Work/Rest and Water Consumption Table							
<i>Applies to average sized, heat-acclimated soldier wearing BDU, hot weather. (See TB MED 507 for further guidance.)</i>							
Easy Work		Moderate Work		Hard Work			
• Weapon Maintenance • Walking Hard Surface at 2.5 mph, < 30 lb Load • Marksmanship Training • Drill and Ceremony • Manual of Arms		• Walking Loose Sand at 2.5 mph, No Load • Walking Hard Surface at 3.5 mph, < 40 lb Load • Calisthenics • Patrolling • Individual Movement Techniques, i.e., Low Crawl or High Crawl • Defensive Position Construction		• Walking Hard Surface at 3.5 mph, ≥ 40 lb Load • Walking Loose Sand at 2.5 mph with Load • Field Assaults			
Heat Category	WBGT Index, F°	Easy Work		Moderate Work		Hard Work	
		Work/Rest (min)	Water Intake (qt/hr)	Work/Rest (min)	Water Intake (qt/hr)	Work/Rest (min)	Water Intake (qt/hr)
1	78° - 81.9°	NL	½	NL	¾	40/20 min	¾
2 (GREEN)	82° - 84.9°	NL	¾	50/10 min	¾	30/30 min	1
3 (YELLOW)	85° - 87.9°	NL	¾	40/20 min	¾	30/30 min	1
4 (RED)	88° - 89.9°	NL	¾	30/30 min	¾	20/40 min	1
5 (BLACK)	> 90°	50/10 min	1	20/40 min	1	10/50 min	1

- The work/rest times and fluid replacement volumes will sustain performance and hydration for at least 4 hrs of work in the specified heat category. Fluid needs can vary based on individual differences (± ¼ qt/hr) and exposure to full sun or full shade (± ¼ qt/hr).
- NL = no limit to work time per hr.
- Rest = minimal physical activity (sitting or standing) accomplished in shade if possible.
- **CAUTION: Hourly fluid intake should not exceed 1½ qts.**
- **Daily fluid intake should not exceed 12 qts.**
- If wearing body armor, add 5°F to WBGT index in humid climates.
- If doing Easy Work and wearing NBC (MOPP 4) clothing, add 10°F to WBGT index.
- If doing Moderate or Hard Work and wearing NBC (MOPP 4) clothing, add 20°F to WBGT index.

For additional copies, contact: U.S. Army Center for Health Promotion and Preventive Medicine, Health Information Operations Division at (800) 222-9898 or CHPPM - Health Information Operations@usag.army.mil

For electronic versions, see <http://chppm-www.apgea.army.mil/heat>. Local reproduction is authorized June 2004



CP-033-0404

Figure 1 PHC water consumption guidance

Table 1 PET generation rate, PHC

Water intake (quart/hr) per person	Water intake (Liters/hr)	Liters/day (over 12 hours)	Number of 0.5 liter bottles/day per person	weight of PET, g/day, (12.7 g/bottle) per person	Weight of PET, kg/day, 1000 pax	Generation rate, over 12 hours, kg/hr, 1000 pax
0.5	0.5	5.7	11.4	144	144	12
0.75	0.7	8.5	17.0	216	216	18
1	0.9	11.4	22.7	288	288	24

2.2 CENTCOM experience

Per contacts at CENTCOM, their experience shows bottle consumption at 11-12 0.5-liter bottles/day. **Table 2** multiplies this out to get kg/day of PET.

To make sure there's enough drinking water, the current planning factor used is 12 liter/day, in 0.5 or 1.5 liter bottles. If this quantity was actually consumed it would double the figures in **Table 2**.

Table 2 Projected generation rate

PAX	Water consumption (liter/day)	# of 1/2-liter bottles per day	Weight of PET (kg/day)
1000	6000	12000	152.4
2000	12000	24000	304.8
6000	36000	72000	914.4
10000	60000	120000	1524

3 Potential uses

Canvassing a handful of people with first-hand experience yielded the following list of potential uses for recycled PET:

- Geotextile/sheets
 - o underlayment for gravel to keep down mud and dust
 - o tarps for storage
 - o sun shades
 - o visual screening for chain-link fences
- Insulating concrete forms that could be left in place
- insulation for wall cavities
- sand bags
- HESCO-type barriers
- stackable, fillable lego-like blocks
- Dimensional lumber for misc. construction (non-structural)
- secondary containment structures for various wastes and storage tanks.

Appendix A-14

ByFusion Report on Mobile ByBlock Manufacturing System

CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE INFORMATION

Access to portions of Appendix A-14 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

**Strategic Environmental Research and Development Program (SERDP)
4800 Mark Center Drive, Suite 16F16
Alexandria, VA 22350-360,
Phone (571) 372-6565.**

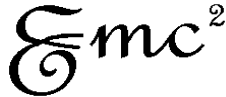
Appendix A-15

rPET Material Property Evaluation Report

**CONTAINS PROPRIETARY, CONFIDENTIAL AND BUSINESS SENSITIVE
INFORMATION**

Access to portions of Appendix A-15 may be granted by either the Principal Investigator of Project WP 18-1047 or by the program office below:

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DRAFT

Date: December 27, 2022

To: Mr. Braxton Lewis, Noblis, (Braxton.Lewis@noblis.org;
Braxton.V.Lewis.ctr@mail.mil), Project Manager

From: Dr. Prabhat Krishnaswamy (kswamy@emc-sq.com), Principal Investigator, and
Mr. Rick Heggs (rheggs@emc-sq.com), Emc²

CC: Dr. Robin Nissan, SERDP (robin.a.nissan.civ@mail.mil), COR, and Dr. Nancy
E. Ruiz CIV USN (US) (nancy.ruiz@navy.mil), COR

Subject: SERDP Project No. WP18-1047, Task 2 - Development of rPET based
Formulations, Deliverable Report on ***'Final rPET Formulation for 3D Printing
Filament in Additive Manufacturing'***

The contract for the above Project was finalized and the project initiated on June 21, 2018. Due to the COVID pandemic a no-cost extension of this project was approved until March 31, 2023. There are five major Tasks being conducted in this effort listed below:

- Task 1 - Sorting of FOB Waste Streams for rPET
- Task 2 - Development of rPET based Formulations
- Task 3 - Modification of Extrusion Processing Technologies
- Task 4 - Evaluation of rPET Products for use at FOBs
- Task 5 - Program Coordination, Reports and Deliverable

Dr. Krishnaswamy and Mr. Rick Heggs have led all the Tasks above along with Dr. Pete Stynoski of the US Army Corps of Engineers ERDC-CERL, the directly funded partner (DFP) on this project.

At the request of the Technical Review Committee during the February 2019 IPR meetings, a deliverable report titled "Interim Report on rPET Material Properties" was submitted in July 2020 and uploaded to SEMS. Attachment 1 provides this Interim Report along with all its attachments in their entirety for completeness - as these results form part of the basis of the recommendation below. Also for brevity, the results in Attachment 1 will not be repeated here and will be referenced as appropriate.

Since completing the above Interim report, it was shown (as detailed in the IPR presentations in 2021) that clean rPET flakes could be used directly to make 1.75mm and 2.85 filaments for Additive Manufacturing (AM) 3D printing applications. These filaments were made successfully at three laboratories (Filamint, OH, Conair, MI and IC3D, OH) with different equipment configurations. Details of this work will be included in the Final Project Report.

Figure 1 shows a photo of the clean rPET flake used for AM, while Figure 2 shows a spool of 2.85 mm filament made from this rPET flake.



Figure 1 – Photo of clean, 4-mm rPET white flake from PET water bottles used for AM filament production trials



Figure 2 – Photo of 2.85 mm diameter rPET filament spool made from clean flake shown in Figure 1

To achieve the above quality of 3D printing filament shown in Figure 2, it was found that the rPET material from PET water bottles (i) had to be cleaned so as to be free from labels, adhesives, and caps (usually made of PP or PE), (ii) had to undergo a size-reduction step that would result in a uniform, clean 4-mm size flake, (iii) dried to less than 0.2% moisture at 225 F for a minimum of 4 hours, prior to be used as feedstock raw material for manufacturing the filament. Once these spools of filament were made that had to be shrink-wrapped in plastic film so as to prevent any moisture uptake by the rPET filament until it is needed for 3D printing.

The next step to finalize the ideal ‘recommended’ formulation with rPET flake involved another recommendation made by the Technical Review Committee after an IPR meeting. In this activity the Committee asked us to investigate the effect of additives on rPET material properties (described in Attachment 1) and whether any improvements could be made.

Therefore, two standard additives Lotader and Kraton available to enhance ductility of polymers were investigated. The additives in various combinations and loading levels were first investigated in making 1x1 dimensional plastic lumber samples (described in QPRs from this project and to be included in the Project Final Report). Based on the results of the plastic lumber trials it was decided that 10% Kraton (FG1901) would provide the most optimum properties with regard to a combination of strength, stiffness and ductility.

Following the above results, a dry-blend of the rPET flake (see Figure 1) was prepared with 10% Kraton and used as feedstock to extrude 2.85 mm filament at IC3D in Hilliard OH under supervision of Emc² staff. For comparison, 2.85 filament spools without any additives were also extruded during these trials. Both materials worked successfully once again to make the 3D printer filament. Attachment 2 provides a copy of the report submitted by IC3D after these trials.

Once the filament was processed, standard ASTM specimens were printed as done previously using both the rPET filament with additives and without additives using 3D printers at three laboratories – IC3D, ERDC-CERL, and at Otterbein University, see samples shown in Figure 3. Also, a sample part recommended by the ERDC-CERL team members on the project, 55-gallon drum wrenches were printed at IC3D and Otterbein using the two grades of filament. Photos of the wrench samples are shown in Figure 4. This wrench was recommended as it is a frequently used item at FOBs and could be printed on site, when needed.



Figure 3 – Photos of ASTM D 790 (Flexural) specimens printed with both 2.85 mm rPET filament without additive (translucent at the bottom) and with 10% Kraton (opaque at the top)



Figure 4 – Photos of 55-gallon drum wrench printed with both 2.85 mm rPET filament without additive (translucent at the bottom) and with 10% Kraton (opaque at the top)

A second design of a much smaller drum wrench was also printed for comparison. All designs of drum wrenches using both rPET formulations were used to open and close drums (see Figure 5), and worked successfully.



Figure 5 – Photos of the smaller 55-gallon drum wrench design printed with 2.85 mm rPET filament being used to open and close the drum.

The most significant finding from the above work was that the 10% by weight Kraton additive in the clean 4-mm rPET formulation made a significant difference in both processing the filament, printing the part with any of the 3D printers at the three laboratories and the surface finish of the part (see Figure 4 opaque part is with additive).

Once the above trials were completed, standard ASTM D638 tensile properties for specimens printed at all three laboratories were determined (in addition to those reported in Attachment 1) to compare the effect of 10% Kraton additives. These data along with specific gravity and flexural properties are shown below in Table 1 along with those for other standard materials used for AM, i.e. PLA, PETG, and ABS. As highlighted in Table 1, the tensile elongation value (ductility) of

the rPET increases significantly with the use of 10% Kraton, from 1.82% to 2.66% , that is an increase of 46%. This would significantly reduce the ‘brittleness’ of rPET materials making it much more effective for use in printing at FOBs.

Table 1 – Properties of 3D printed rPET materials with and without 10% Kraton and comparison with other AM materials such as PLA, PETG, and ABS

Property	ASTM Test		Units	rPET- without Kraton	rPET-with 10% Kraton	PLA	PETG	ABS
Sp. Gravity	D792			1.149	1.344	1.177	1.247	1.006
Flexure	D790	Strength	psi	8334	7758	12616	9436	6806
	D790	Modulus	ksi	253	254	461	277	220
	D790	Yield Strain	in/in	0.054	0.0454	0.047	0.053	0.054
Tensile	D638	Strength	psi	3697	3065	5824	5958	4336
	D638	Modulus	ksi	230	164.2	401	276	262
	D638	Elongation	%	1.820	2.66	2.160	2.930	2.160

A graph of the Tensile Elongation property from the above table is also shown in Figure 6 below along with those for injection molded rPET specimens.

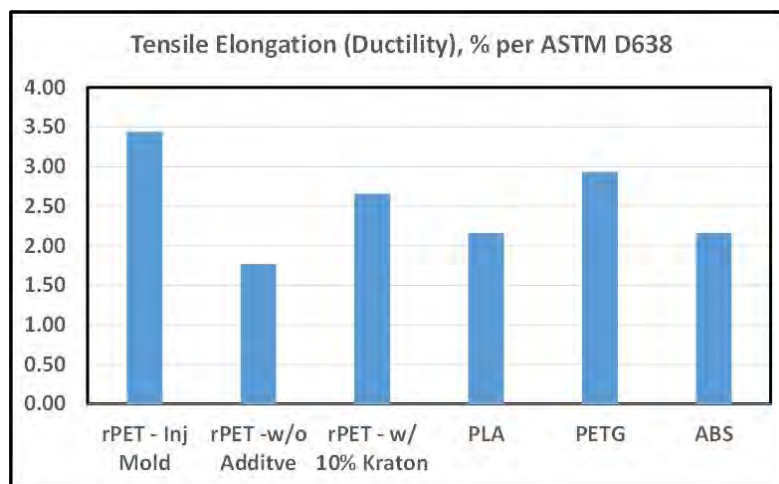


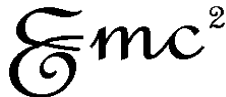
Figure 6 – Graph of Tensile Elongation (ductility) of various formulations of rPET in comparison with standard AM materials PLA, PETG, and ABS.

In conclusion, the ideal, recommended final formulation for rPET material for making 3D printing filament for AM would involve the following steps:

- Use waste PET bottles as the sole source as feedstock
- The bottles must be flattened, shredded, washed, and shredded after removal of any labels, adhesives and bottle caps.
- The shredded material needs to be size-reduced by grinding and sifting to a consistent 4-mm clean flake
- The flake is then dried at 225 F for at least 4 hours to result in than 0.2% moisture
- The clean flake is to be dry-blended with 10% Kraton FG 1901 additive

- vi. The 3D printing 2.85 mm filament is then extruded (as detailed in the final report)
- vii. The recommended setting in Attachment 2 may be used as a basis for printing high quality parts with excellent surface finish and an optimum blend of mechanical and physical properties at FOBs

ATTACHMENT - 1



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DRAFT- Revision 1

Date: July 15, 2020

To: Mr. Braxton Lewis, Noblis, (Braxton.Lewis@noblis.org;
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From: Dr. Prabhat Krishnaswamy (kswamy@emc-sq.com), Principal Investigator, and
Mr. Rick Heggs (rheggs@emc-sq.com), Emc²

CC: Dr. Robin Nissan, SERDP (robin.a.nissan.civ@mail.mil), COR, and Dr. Nancy
E. Ruiz CIV USN (US) (nancy.ruiz@navy.mil), COR

Subject: Project No. WP18-1047 – Subtask 4.2 – Interim Report on rPET Material Properties

At the February 2019 Interim Project Review (IPR) meetings, one of the recommendations from the Technical Review Committee was to require submission of an Interim Material Property Report under ‘Task 4 - Evaluation of rPET Products for use at FOBs’ of this project. Therefore, a new ‘Subtask 4.2 – Interim Report on rPET Material Properties’ was added to the work scope of this project which details the evaluation of the recycled Polyethylene Terephthalate (rPET) used in all the trials during this project.

This draft report is the deliverable that is being uploaded on SEMS on Subtask 4.2.

Subtask 4.2 – Interim Report on recycled rPET Material Properties

The contract for this effort was finalized and the project initiated on June 21, 2018. The five major Tasks planned on this project are listed below:

- Task 1 - Sorting of FOB Waste Streams for rPET
- Task 2 - Development of rPET based Formulations
- Task 3 - Modification of Extrusion Processing Technologies
- Task 4 - Evaluation of rPET Products for use at FOBs
- Task 5 - Program Coordination, Reports and Deliverable

Dr. Krishnaswamy and Mr. Rick Heggs have led all the Tasks above along with Mr. Jonathan Trovillion of the US Army Corps of Engineers ERDC-CERL, the directly funded partner (DFP) on this project.

The subtasks under the above ‘Task 4 - Evaluation of rPET Products for use at FOBs’ along with the deliverable revised dates as posted on the SEMS Project Plan are as follows:

- 4.1 Evaluate rPET material properties (Due: 4/30/2019 - **Completed**)
- 4.2 Interim report that provides a description of rPET Material Properties (Due: 12/31/19)
- 4.3 Evaluate rPET filament properties (Due: 4/30/2020)

- 4.4 Design guide for rPET 3D filament in printers (Due: 9/30/2020)
- 4.5 Evaluate rPET strapping properties (Due: 10/31/2020 - **OBE¹**)
- 4.6 Design guide for rPET strapping for packaging (Due: 1/31/2021 - **OBE**)

The details of the efforts completed under Subtask 4.2 above are Interim Report described below.

Subtask 4.2 – Interim Report on rPET Material Properties

1. Material Procurement

The focus of the overall project involves recycling of waste rPET materials available at Forward Operating Bases (FOBs) - which has been estimated to be as much as 0.5 lbs/person/day. After discussions with staff at the Army Corps it was decided that obtaining actual samples of waste PET from FOBs for conducting trials was not feasible for logistical reasons and that rPET materials from waste plastics available stateside would be a close representative of what is available at FOBs. Therefore, Emc² contacted Phoenix Technologies International, LLC of Bowling Green, OH (<https://www.phoenixtechnologies.net/>) a company a commercial recycler of waste plastics with an annual capacity of converting 80M lbs of post-consumer PET into various grades of rPET for sale. The rPET grades were purchased from Phoenix for the efforts conducted to date are listed in Table 1 below.

Table 1 – List of rPET Materials Procured from Phoenix for Various Application Trials

Vendor	Material	Emc2 - Designation	Specification / Notes	Applications
Phoenix	rPET Pellets - NLP Clear	rPET - 1	IV = 0.7; fully crystallized; ~5% amorphous	filament and plastic lumber
Phoenix	rPET Pellets - NLP-H	rPET - 2	IV = 0.75	plastic lumber
Phoenix	rPET 4mm white flake	rPET - white flake	4mm fully crystallized flake; noncommercial	filament and plastic lumber
Phoenix	rPET green flake	rPET - green flake	green clean flake post wash + sorted	plastic lumber

Figures 1 through 4 below show photographs of the four resin samples procured. As may be seen the rPET-1 and rPET-2 grades visually appear to be the same. Material Safety Data Sheet (MSDS) sheet for all rPET grades along with the Technical Specification for each of the three commercial grades (other than the non-commercial rPET-white flake) along with an injection molding processing guide are provided in Appendix 1. For reference, a technical data sheet for virgin PET material from the literature was also downloaded and is included in Appendix 2.



Figure 1- Photo of rPET-1 Resin Sample

¹ Overcome by Events (OBE).



Figure 2 – Photo of rPET-2 Resin Sample



Figure 3 – Photo of rPET-White Flake Sample



Figure 4 – Photo of rPET-Green Flake Sample

2. Material Characterization

Prior to conducting any trials with the rPET materials procured as described above, a complete characterization of properties was conducted. The first material property characterized was a complete Thermogravimetric Analysis (TGA) of both rPET-1 and rPET-2 to determine both the processing conditions and degradation temperatures for the resin. Appendix 3 provides the TGA curves and as may be noted in Figure 5 below both grades of resin are identical with regard to their TGA. That is, all grades of rPET would be expected to have properties in the same range.

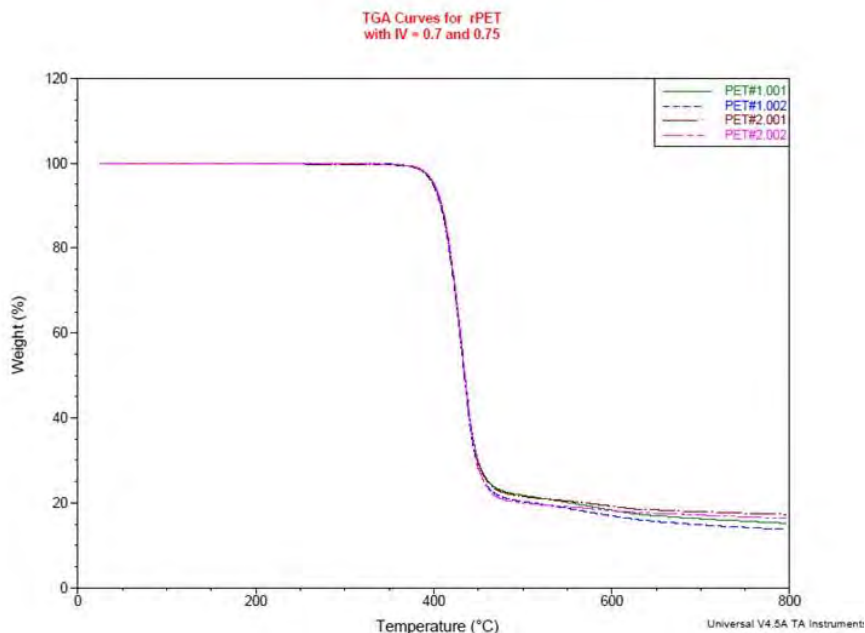


Figure 1 TGA Curves for rPET-1 and rPET-2 resin samples (two replicates each)

The next step involved evaluating standard physical, mechanical and thermal properties using ASTM test methods. Since one of the goals was to determine if rPET could be used to make filament for 3D printers, both injection molded and 3D printed (from rPET filament) ASTM specimens were used to determine material properties. Two sets of ASTM specimens were molded using the rPET materials – one at GDC in Goshen, IN and the other at The Ohio State University's Center for Design and Manufacturing Excellence (CDME) in Columbus, OH.

Appendix 4 provides the injection molding conditions used to make standard ASTM specimens at CDME (same processing conditions were used by GDC as well). Appendix 5 shows the processing conditions used to make the ASTM specimens on a Lulzbot Pro dual filament printer.

The properties evaluated, the ASTM test methods used as well as the mean properties for each case are summarized below in Table 2. All tests were conducted by an A2LA certified laboratory at Ineos (formerly Ashland Analytical Services) of Dublin OH and GDC Inc. of Goshen, IN. The detailed reports from the ASTM tests from the laboratories are provided in Appendix 6.

Table 2 – Summary of Material Property Data for Injection Molded and 3D Printed Specimens

Property	ASTM Method	Value	Value	Difference. %
Laboratory		Ineos/ GDC	Ineos	
Processing Method		Inj Molded	Lulzbot Pro	
Specific Gravity	D792	1.333	1.262	-5.33%
Tensile Strength, psi	D638	7794	4089	-47.54%
Tensile Modulus, ksi	D638	342	259	-24.27%
Tensile Elongation, %	D638	3.44	1.77	-48.55%
Heat Distortion Temperature	D648	139.46	150.26	7.74%
Flexural Strength, psi	D790	11402	5031	-55.88%
Flexural Modulus, ksi	D791	352.5	146.2	-58.52%
Flexural Strain at Yield,in/in	D792	0.0441	0.0606	37.41%
Hardness, Shore D	D2240	77	50.7	-34.16%
Izod impact - Notched, ft-lbs/in	D256	0.64	0.5	-21.88%

3. Conclusions to Date and Future Work

One of the major conclusions from the above material property evaluation is that the 3D printed specimens have lower mechanical properties (tensile, flexure, hardness and Izod) compared to the injection molded parts. This is not unexpected as the packing density of the material from injection molding is higher than that during 3D printing which is reflected in the lower specific gravity of the 3D printed parts.

However, of critical importance is the fact that the properties of the 3D printed specimens are in the general range of other engineering plastics (such as ABS, PETG, etc.) and can therefore be used for various light structural and other load bearing applications and components at Forward Operating Bases (FOBs). The values for rPET specimens from 3D printing are preliminary in nature. Additional ASTM specimens are being printed from both rPET as well as other engineering plastics listed above to confirm these material properties and also compare with those of resins such as ABS, PLA, PETG, etc. that are typically used in AM applications.

The future work planned is to evaluate other rPET properties that could be of interest for product applications at FOBs and comparisons to other engineering plastics and resins used in Additive Manufacturing. These properties will be evaluated after discussions with the team members from the US Army Corps of Engineers at ERDC-CERL and their contacts at FOBs. There are products involving three processing methods already being considered for development with rPET for FOB applications – Additive Manufacturing (AM) or 3D printed components, profile extrusions such a dimensional plastic lumber, and, ByBlocks that have the same dimensions as Concrete Masonry Units (CMU) for light outdoor construction and structures. Samples of these parts have already been made with rPET and appropriate physical, mechanical and thermal properties are being determined. When this work is completed this draft ‘*Subtask 4.2 – Interim Report on rPET Material Properties*’ will be updated and revised so as to provide rPET properties for design of components using the three processing methods being considered.



Phoenix Technologies International, LLC
1098 Fairview Ave. Bowling Green, OH 43402

SAFETY DATA SHEET

Polyethylene Terephthalate or PET Flakes

1. CHEMICAL PRODUCT/COMPANY IDENTIFICATION

PRODUCT IDENTIFIER: Polyethylene Terephthalate or PET Flakes (RPET flake, NLF™ and RPET RESIN, Lno™, NLP™, LNOCTM)

RECOMMENDED USE AND USE RESTRICTIONS: Polymer for plastics industry. Not intended for internal use or medical implantation.

MANUFACTURER / SUPPLIER:

Phoenix Technologies International LLC.
1098 Fairview Ave.

Bowling Green, OH 43402

EMERGENCY PHONE NUMBERS:

2. HAZARD(S) IDENTIFICATION

OSHA HAZARD CLASSIFICATION: COMBUSTIBLE DUST – WARNING! MAY FORM COMBUSTIBLE DUST CONCENTRATIONS IN AIR.

HAZARDS NOT OTHERWISE CLASSIFIED: CAUTION! MOLTEN MATERIAL WILL PRODUCE THERMAL BURNS. Molten polymer will adhere to skin and may cause severe burns.

Eye contact with polymer particles may cause mechanical irritation with discomfort, tearing, or temporary blurring of vision.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Material: Polyethylene Terephthalate CAS Number 25038-59-9 Variable concentration >99%

Residual additives, modifiers, colorants / impurities <1%

4. FIRST AID MEASURES

INHALATION: Polyethylene Terephthalate is not likely to be hazardous by inhalation. If exposed to fumes from overheating, move to area with fresh air. If not breathing contact emergency services.

SKIN CONTACT: Polyethylene Terephthalate is not likely to be hazardous by skin contact. If molten material contacts skin, cool immediately with cold water. Do not attempt to remove material from skin. Obtain medical treatment.

EYE CONTACT: In case of contact, immediately flush eyes with water for 15 minutes. If contact with molten material, seek medical attention immediately.

INGESTION: Polyethylene Terephthalate is not intended to be ingested as part of normal use. If ingested, consult a physician.

5. FIRE FIGHTING MEASURES

SUITABLE EXTINGUISHING MEDIA: Water, Foam, Carbon Dioxide (CO₂), or Dry Chemical.

SPECIFIC HAZARDS ARISING FROM CHEMICAL

HAZARDOUS COMBUSTION PRODUCTS: Carbon dioxide and carbon monoxide.

SPECIAL PROTECTIVE EQUIPMENT AND PRECAUTIONS FOR FIRE-FIGHTERS

Keep personnel removed and upwind of fire. Wear self-contained breathing apparatus. Wear full protective equipment.

6. ACCIDENTAL PRECAUTIONS / PROTECTIVE EQUIPMENT / EMERGENCY PROCEDURES

The interior of molten masses may remain hot for some time because of the low heat conductivity of the polymer. Use care when handling / disposing of molten masses.

Review Section 5. FIRE FIGHTING MEASURES and Section 7. PRECAUTIONS FOR SAFE HANDLING before proceeding with clean-up. Use appropriate Personal Protective Equipment during clean-up. Thermal protective equipment should be used when handling molten material (See Section 8. For further details).

METHODS AND MATERIALS FOR CONTAINMENT AND CLEANUP

Sweep up and recover, or mix material with moist absorbent and shovel into suitable chemical waste container.

7. HANDLING AND STORAGE

PRECAUTIONS FOR SAFE HANDLING: Do not breathe gases, vapors or fumes that may be evolved during processing. Caution and suitable thermal eye, face, and body personal protective equipment must be used if handling hot or molten material. Contact with molten material can cause burns, so unprotected contact with molten material must be avoided.

Polyethylene Terephthalate can cause slip hazards in multiple forms (Flake, Pellet, and Dust) and should be kept clear of all walkways. Avoid dust generation to prevent dust accumulation to minimize explosion hazard. Grinding of Polyethylene Terephthalate can create dust and potential dust explosion hazard. To reduce risk follow National Fire Protection Association's codes and standards for handling combustible dust.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE LIMITS: There are no established exposure limits for Polyethylene Terephthalate for PEL or TLV. Particles not otherwise regulated the limit is 15mg / m³ total dust 5 mg / m³ respirable fraction (PEL). For particles not otherwise specified 3mg / m³ respirable particles and 10mg / m³ for inhalable particles. All mentioned limits are 8 hour time weighted average limits.

APPROPRIATE ENGINEERING CONTROLS: Use local ventilation to control gasses, vapors and fumes from hot processing. Use static controls. Static charges can build up and ignite dust or solvent laden atmospheres. Design precautions into processes that can create dust, such as pneumatic conveying systems, grinding and other physical operations. There is the potential for a dust explosion hazard. Ensure that dust handling systems are designed in a manner to prevent the escape of dust into work areas.

INDIVIDUAL PROTECTION MEASURES / PERSONAL PROTECTIVE EQUIPMENT

EYE/FACE PROTECTION: Wear coverall chemical splash goggles when the possibility of eye or face contact from airborne material exists. Wear a face shield when working with molten material.

RESPIRATORY PROTECTION: Respirators are not needed for normal use. When airborne concentrations are expected to exceed exposure limits, a NIOSH approved respirator should be selected based on the form and concentration of the contaminant in air and in accordance with the OSHA Respiratory Protection Standard (29 CFR 1910.134).

PROTECTIVE CLOTHING: If there is potential for contact with hot/molten material, wear heat resistant impervious clothing and footwear. Special protective clothing is not needed for normal use. Gloves are recommended as good industrial practice.

RECOMMENDED DECONTAMINATION FACILITIES: Eyewash station, washing facilities.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance:	Solid, clear/colored flakes	Flammability Limits (upper/Lower):	No data available
Odor:	Odorless	Vapor Pressure:	Not Applicable
Odor Threshold:	No data available	Vapor Density:	Not Applicable
pH:	Not Applicable	Specific Gravity:	1.33 – 1.45 g/cm ³
Melting Point:	225 – 250 °C	Solubility in Water:	Insoluble
Initial Boiling Point/Range:	No data available	Partition coefficient(n-octanol/water):	No data available
Flash Point:	Not applicable, combustible solid	Auto-Ignition Temperature:	No data available
Evaporation Rate:	No data available	Decomposition Temperature:	330 °C
Flammability:	No data available	Viscosity:	No data available

10. STABILITY AND REACTIVITY

REACTIVITY: None known

CHEMICAL STABILITY: Stable at normal conditions. Polymerization will not occur

POSSIBILITY OF HAZARDOUS REACTIONS: None known

CONDITIONS TO AVOID: Temperatures above 330 °C

INCOMPATIBLE MATERIALS: Incompatible or can react with strong oxidizers.

Hazardous decomposition products: Thermal decomposition products caused by overheating polymer can include carbon monoxide, carbon dioxide, acetaldehyde and ethylene. Decomposition products (gases, vapors and / or fumes) may cause skin, eye or respiratory tract irritation, and other adverse health effects.

11. TOXICOLOGICAL INFORMATION

INFORMATION ON LIKELY ROUTES OF EXPOSURE: Polymer dust may be inhaled, and come in contact with skin and eyes. Thermal decomposition products may be inhaled.

SYMPTOMS RELATED TO PHYSICAL, CHEMICAL AND TOXICOLOGICAL CHARACTERISTICS:

Skin contact with molten material will produce thermal burns.

Eye contact with polymer dust may cause mechanical irritation with discomfort, tearing, or blurring of vision. Polyethylene terephthalate is a mild eye irritant.

Eye contact with molten material will produce thermal burns.

Decomposition products (gases, vapors and / or fumes) may cause skin, eye or respiratory tract irritation, and other adverse health effects.

ACUTE, DELAYED, AND CHRONIC EFFECTS FROM SHORT AND LONG-TERM EXPOSURE:

Polyethylene terephthalate animal testing indicates

- No carcinogenic, mutagenic, developmental or reproductive effects
- No adverse effects from short exposures by inhalation or ingestion.

Polyethylene terephthalate patch tests with humans resulted in no skin irritation or skin sensitization.

NUMERICAL MEASURES OF TOXICITY: Polyethylene Terephthalate: Oral Approximate Lethal Dose (ALD): > 10,000 mg/kg in rats

CARCINOGENICITY INFORMATION: None of the components present in this material at concentrations equal to or greater than .1% are listed by NTP, IARC, OSHA or ACGIH as carcinogenic.

12. ECOLOGICAL INFORMATION

No toxicity data is available. The product is insoluble in water.

13. DISPOSAL CONSIDERATIONS

Treatment, storage, transportation and disposal must be in accordance with applicable Federal, State / Provincial, and Local regulations.

14. TRANSPORTATION INFORMATION

Shipping information

DOT	Not regulated
TDG	Not regulated
UN Proper Shipping Name/Number	Not regulated

IMDG
IATA

Not Regulated
Not Regulated

15. REGULATORY INFORMATION

U.S. FEDERAL REGULATIONS:

- TSCA Inventory Status: In compliance with TSCA Inventory requirements for commercial purposes.
- Under RCRA, it is the responsibility of the product user to determine at the time of disposal whether a material containing this product or derived from the product should be classified as a hazardous waste (40 CFR 261.20-24).
- If discarded in its purchased form, this product does not meet the RCRA characteristic definition for ignitability, corrosivity or reactivity and is not a RCRA listed waste: however, it has not been tested by the Toxicity Characteristic Leaching Procedure (TCLP).
- SARA, Title III: This material is not known to contain extremely hazardous substances.

INTERNATIONAL REGULATIONS:

- DSL (Canadian Domestic Substances List) and CEPA (Canadian Environmental Protection Act): This product listed on the DSL or otherwise complies with CEPA new substance notifications requirements.
- No components of this product are on the Mexican Raw Materials Regulation.

16. ADDITIONAL INFORMATION

The data in this Safety Data Sheet relates only to the specific material designated herein and does not relate to use in combination with any other material or in any process.

SDS Revision Date: March 2, 2016

End of SDS



Specifications for Crystalline RPET Phoenix Process

100% Post-Consumer PET Resin Grade: NLP Clear resin

GENERAL PURPOSE GRADE

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY	0.71 ± 0.02/-0.03 dL/gm
MELTING POINT	246° ± 3° C.
CRYSTALLINITY	36.0 % ± 5.0
UNDERCRYSTALLIZED PELLETS	< 5%
CIE (L*, a*, b*) COLOR †	L* 67.0 Min a* -1.50 + 1.0 b* + 2.0 Max
PELLET SIZE	0.120" ± 0.020 MAJOR DIA.
FINES	0.08 % MAX.
FILTRATION	filtered to 60 microns
MOISTURE CONTENT	0.2 % MAX.
BULK DENSITY	50 -60 lbs/ ft3

NLP Clear rPET resin is derived from diverse feed stocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

PHOENIX TECHNOLOGIES MAKES NO WARRANTY BEYOND THE SPECIFICATIONS AND DISCLAIMS ANY WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

† 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant; Reflectance method

†† As provided by continuous melt filtration by min. 200 Mesh screen and as measured in a single dimension by Phoenix test method LAB103.



Specifications for Crystalline RPET Phoenix Process

100% Post-Consumer PET Resin Grade: NLP H Clear resin

GENERAL PURPOSE GRADE
- trial

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY	0.75 ± 0.02/-0.03 dL/gm
MELTING POINT	246° ± 3° C.
CRYSTALLINITY	36.0 % ± 5.0
UNDERCRYSTALLIZED PELLETS	< 5%
CIE (L*, a*, b*) COLOR †	L* 67.0 Min a* -1.50 + 1.0 b* + 2.0 Max
PELLET SIZE	0.120" ± 0.020 MAJOR DIA.
FINES	0.08 % MAX.
FILTRATION	filtered to 60 microns
MOISTURE CONTENT	0.2 % MAX.
BULK DENSITY	50 -60 lbs/ ft3

NLP Clear rPET resin is derived from diverse feed stocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

PHOENIX TECHNOLOGIES MAKES NO WARRANTY BEYOND THE SPECIFICATIONS AND DISCLAIMS ANY WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

† 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant; Reflectance method

†† As provided by continuous melt filtration by min. 200 Mesh screen and as measured in a single dimension by Phoenix test method LAB103.



Specifications for RPET Phoenix Curbside Flake
100% Post-Consumer PET Resin Grade: NLF-GREEN

NON FOOD GRADE

<u>ATTRIBUTE</u>	<u>SPECIFICATION</u>
INTRINSIC VISCOSITY (SOLUTION)	> 0.73 dL/gm
CIE COLOR [†]	a* -32 - 0
FLAKE SIZE	< 12mm
FINES	< 0.5% MAX.
MOISTURE CONTENT	≤ 1.0%
BULK DENSITY	≥ 20 lbs/ ft ³
PVC CONTAMINATION	< 50 ppm (lot average)
LOWMELT CONTAMINATION	< 250 ppm
NYLON CONTAMINATION	< 30 ppm
NON MELTING *	< 100 ppm
OTHER COLORS PET++	< 250ppm

No single package within a load should exceed these specifications, if the load is in a bulk truck the load average should meet these specifications

NLF grade RPET resin is derived from diverse feedstocks of post-use recycled containers, and as such, there is no representation that the plastic in any particular order will conform exactly to the Specifications given.

[†] 1976 CIE L,a,b – CIE 1931 10° Observer angle – D65 Illuminant. Reflectance method.

* non-melting to include items such as metal, glass, sand, stones, wood (any item with a melt point > 260 C)

++ applies to all other colored and opaque PET including but not limited to amber, pink, white, yellow

Appendix 1 - rPET - Injection Molding Processing Guide

Phoenix Technologies Grade RPET Resins NLP™ and LNO™P/PT

Technical Processing Information

Recommended Drying Procedures

PET is a very hygroscopic material which readily absorbs moisture when exposed to ambient air. PET which is not properly dried will result in a lower IV and loss of other important physical properties. At time of processing, the moisture in PET must be less than **50 ppm** or 0.005% by weight.

Dry thoroughly in a high heat regenerative–desiccant drying system. DO NOT remove dried material from the drying hopper or expose it to ambient conditions prior to processing. Rapid degradation will occur when hot, dry PET is exposed, even briefly, to ambient air.

Recommended Drying Parameters

Drying Time	4-5 hours
Drying air temperature	295-330° F.
Drying air dewpoint	-40° C. / F.
Drying air flow rate	1 CFM per pound per hour processed

The drying temperature used should be kept as low as possible to minimize yellowing by oxidation during this portion of the process.

Material Handling

Phoenix RPET resin is produced from Recycled PET. It shows good material flow characteristics and blends well with major virgin PET resins. For best results blending should be done just prior to drying, and conveying blended materials long distances should be avoided, but Phoenix RPET resin can generally be handled as your virgin PET resin.

Processing Parameters

Phoenix RPET resin can be processed very easily over a wide range of conditions. Typically, processing temperatures range from 530-565° F. The main focus of processing should be to minimize process temperatures and melt residence times in an effort to maintain the molecular weight of the material and minimize any yellowing of the material during processing.

- Use the lowest possible barrel-temperature profile that will produce a clear product.
- Minimize melt-residence time.
- Minimize the back pressure used in extrusion to reduce shearing of the material.
- Use the lowest possible hot runner and nozzle temperature that will produce a clear part.
- Use the lowest possible mold coolant temperature to promote part clarity.

Phoenix Technologies

Our specialization in RPET has allowed us to concentrate all of our time and effort to produce the highest quality, contaminate-free recycled resin which is very close to virgin material in quality and performance.

All of us at Phoenix Technologies want to become your supplier of recycled PET resin and are capable and willing to assist you with specific processing questions on an individual basis. Please feel free to call us at 419-353-RPET.

Material Safety Data Sheets providing safety precautions that should be observed in handling and storing the products mentioned in this publication are available and should be reviewed before handling these products.

Phoenix Technologies International LLC provides NLP™ and LNO™ grade RPET resin to a variety of customers for specific applications depending upon their needs. We are not in the business of advising customers and suppliers generally as to usage of our product, including drying and processing procedures, or to the testing or other methodology which could require modification depending upon the specific applications desired. The above information is furnished without charge as an accommodation to our customers and suppliers and is of general application only. Phoenix Technologies International LLC and its affiliates do not warrant or represent the accuracy of such information in each and every application and disclaim any and all responsibility for the results or consequences of use of such information or the use of any product, method, or apparatus referred to above. It is the responsibility of each customer or user, based on whatever testing criteria it deems appropriate, to determine the suitability of our product or methods for any particular use or purpose.

GEHR Plastics PET Polyethylene Terephthalate**Categories:** [Polymer](#); [Thermoplastic](#); [Polyester, TP](#); [Polyethylene Terephthalate \(PET\)](#)**Material Notes:** Polyethylene terephthalate shows high tensile and mechanical strength, hardness and toughness, low friction and a high dimensional stability. PET may be used at temperatures ranging from -20°C to approximately 115°C.**Properties**

- high mechanical resistance and tensile strength
- high surface strength
- easy to varnish and polish
- high dimensional stability
- high toughness
- good electrical insulating properties
- high chemical resistance
- limited dielectric properties
- sensitive to hydrolysis

Applications include bearings, pumps and parts for housings, frame components, tank lids, cogwheels, insulators for the electrotechnic, deflection rollers for the filament industry, levers, handles, and control disks.

Vendors: No vendors are listed for this material. Please [click here](#) if you are a supplier and would like information on how to add your listing to this material.

Physical Properties	Metric	English	Comments
Specific Gravity	1.39 g/cc	1.39 g/cc	ISO 1183
Moisture Absorption at Equilibrium	0.25 %	0.25 %	ISO 62
Water Absorption at Saturation	0.50 %	0.50 %	ISO 62

Mechanical Properties	Metric	English	Comments
Hardness, Rockwell M	96	96	ISO 2039
Hardness, Shore D	84	84	ISO 868
Ball Indentation Hardness	170 MPa	24700 psi	ISO 2039
Tensile Strength, Yield	90.0 MPa	13100 psi	ISO 527
Elongation at Break	15 %	15 %	ISO 527
Elongation at Yield	4.0 %	4.0 %	ISO 527
Modulus of Elasticity	3.445 GPa	499.7 ksi	ISO 527
Charpy Impact Unnotched	5.00 J/cm ²	23.8 ft-lb/in ²	ISO 179
	5.70 J/cm ²	27.1 ft-lb/in ²	round rods 220-300mm; ISO 179
	6.50 J/cm ²	30.9 ft-lb/in ²	round rods 160-200mm; ISO 179
Charpy Impact, Notched	0.200 J/cm ²	0.952 ft-lb/in ²	ISO 179

Electrical Properties	Metric	English	Comments
Volume Resistivity	3.00e+13 ohm-cm	3.00e+13 ohm-cm	IEC 60093
Surface Resistance	>= 1.00e+13 ohm	>= 1.00e+13 ohm	IEC 60093
Dielectric Constant	3.2	3.2	IEC 60250
	@Frequency 1.00e+6 Hz	@Frequency 1.00e+6 Hz	
Dielectric Strength	22.0 kV/mm	559 kV/in	IEC 60243-1
Dielectric Loss Index	0.014	0.014	IEC 60250
	@Frequency 1.00e+6 Hz	@Frequency 1.00e+6 Hz	
Comparative Tracking Index	600 V	600 V	IEC 60112

Thermal Properties	Metric	English	Comments
CTE, linear	80.0 µm/m-°C	44.4 µin/in-°F	ISO 11359

Thermal Conductivity	0.290 W/m-K	2.01 BTU-in/hr-ft ² -°F	ISO 22007-4
	@Temperature 20.0 °C	@Temperature 68.0 °F	
Melting Point	245 °C	473 °F	DIN EN ISO 3146
Maximum Service Temperature, Air	100 °C	212 °F	UL 746B
Deflection Temperature at 1.8 MPa (264 psi)	80.0 °C	176 °F	ISO 75
Minimum Service Temperature, Air	-20.0 °C	-4.00 °F	UL 746B
Glass Transition Temp, Tg	98.0 °C	208 °F	DIN EN ISO 3146
Flammability, UL94	HB	HB	UL 94
Oxygen Index	25 %	25 %	ASTM D2863

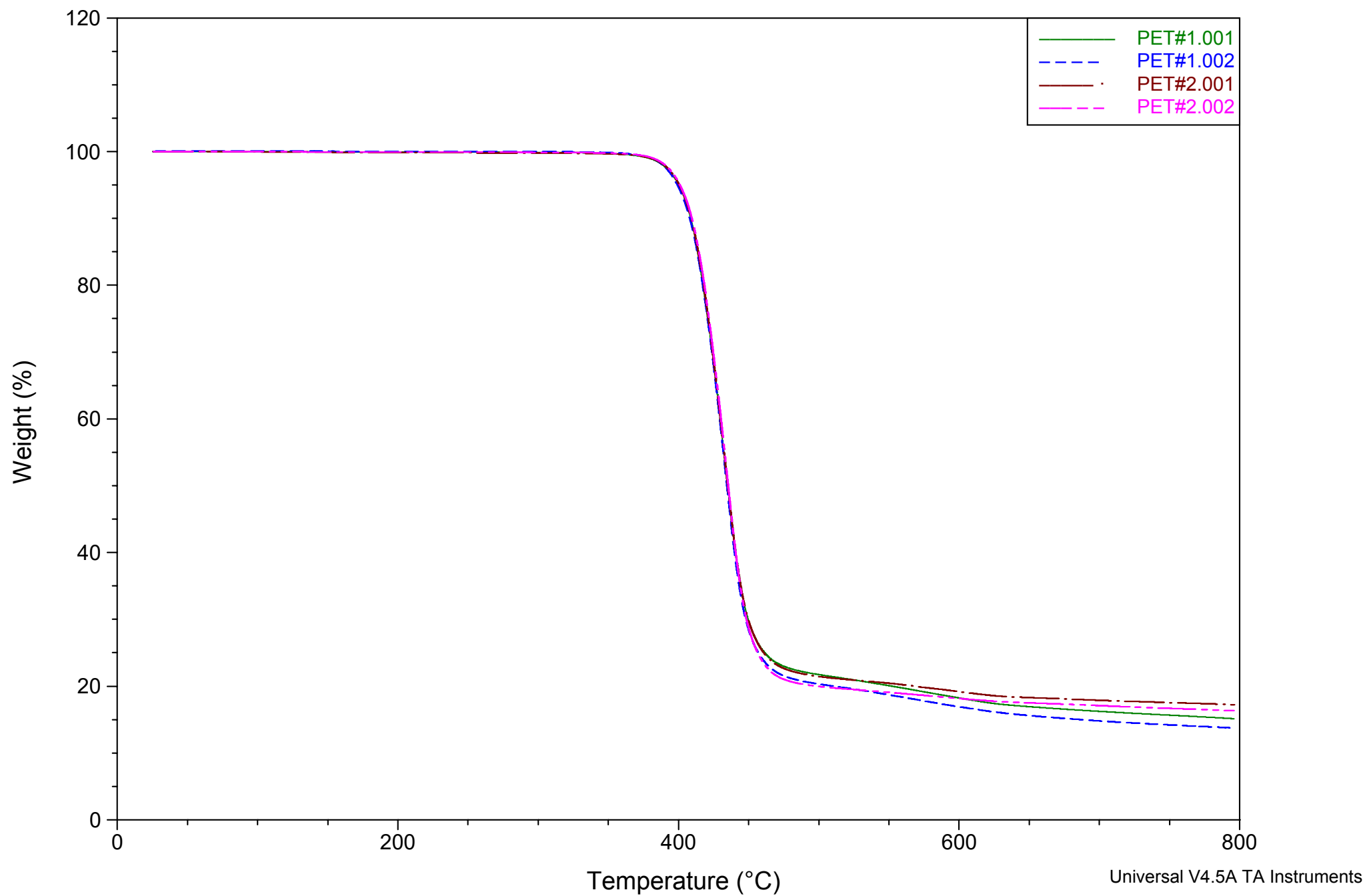
Compliance Properties	Metric	English	Comments
FDA	Yes	Yes	Physiological indifference

Descriptive Properties

Acid Resistance	yes
Aromatic Resistance	yes
Bondability	yes
CKW Resistance	limited
Color	natural
Hydrocarbonate Resistance	yes
Hydroxide Resistance	limited
Physiological indifference	yes, EEC
UV Stabilization	limited / no

Some of the values displayed above may have been converted from their original units and/or rounded in order to display the information in a consistent format. Users requiring more precise data for scientific or engineering calculations can click on the property value to see the original value as well as raw conversions to equivalent units. We advise that you only use the original value or one of its raw conversions in your calculations to minimize rounding error. We also ask that you refer to MatWeb's [terms of use](#) regarding this information. [Click here](#) to view all the property values for this datasheet as they were originally entered into MatWeb.

**TGA Curves for rPET
with IV = 0.7 and 0.75**

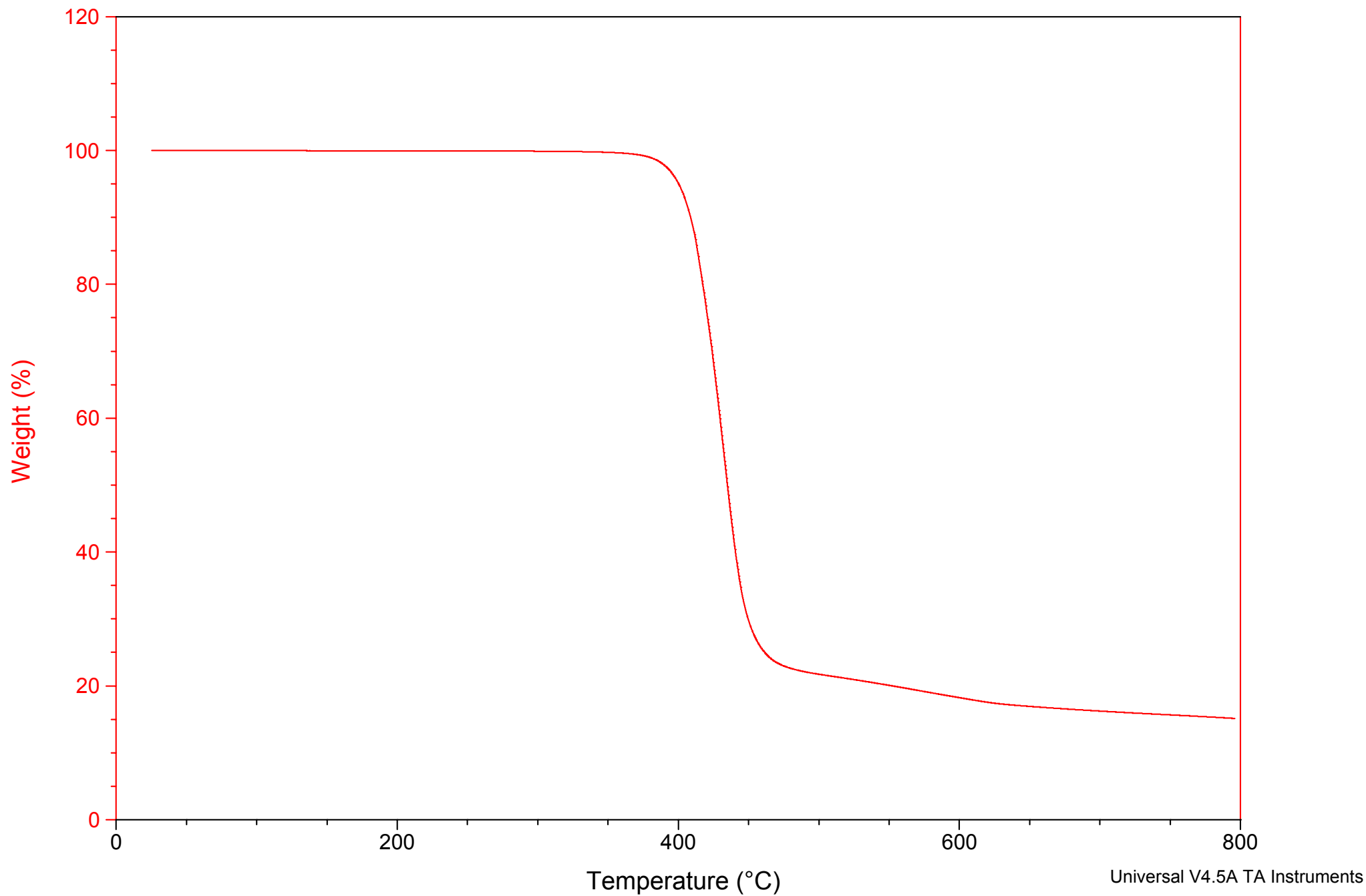


Appendix 3 - TGA for rPET-1 and rPET-2 Resins

Sample: PET #1
Size: 12.6680 mg
Method: Rubber

TGA

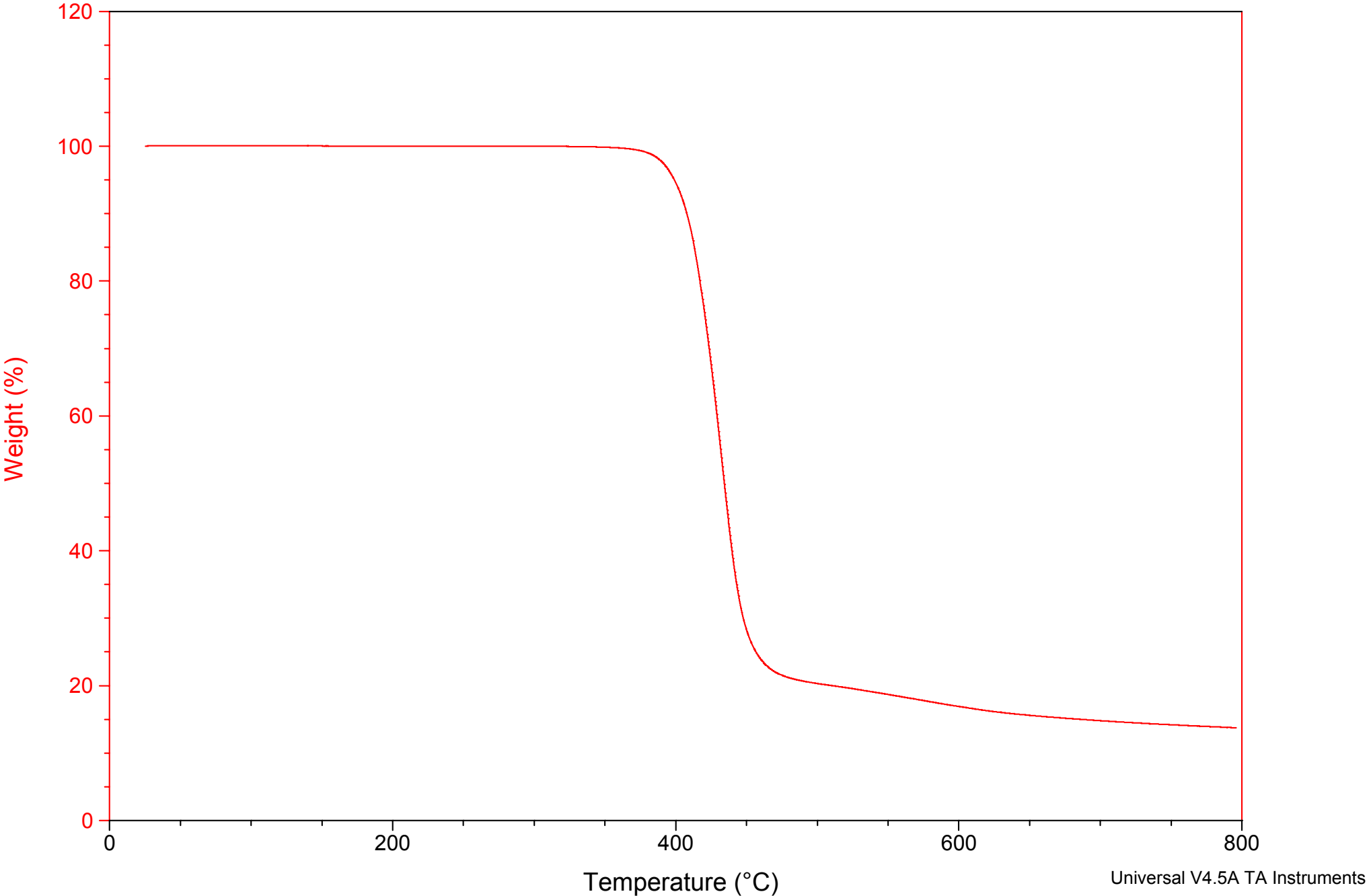
File: C:\TA\Data\TGA\ZHE L\PET#1.001
Operator: Zhe
Run Date: 07-Feb-2019 10:42
Instrument: TGA Q50 V20.13 Build 39



Sample: PET #1
Size: 13.4640 mg
Method: Rubber

TGA

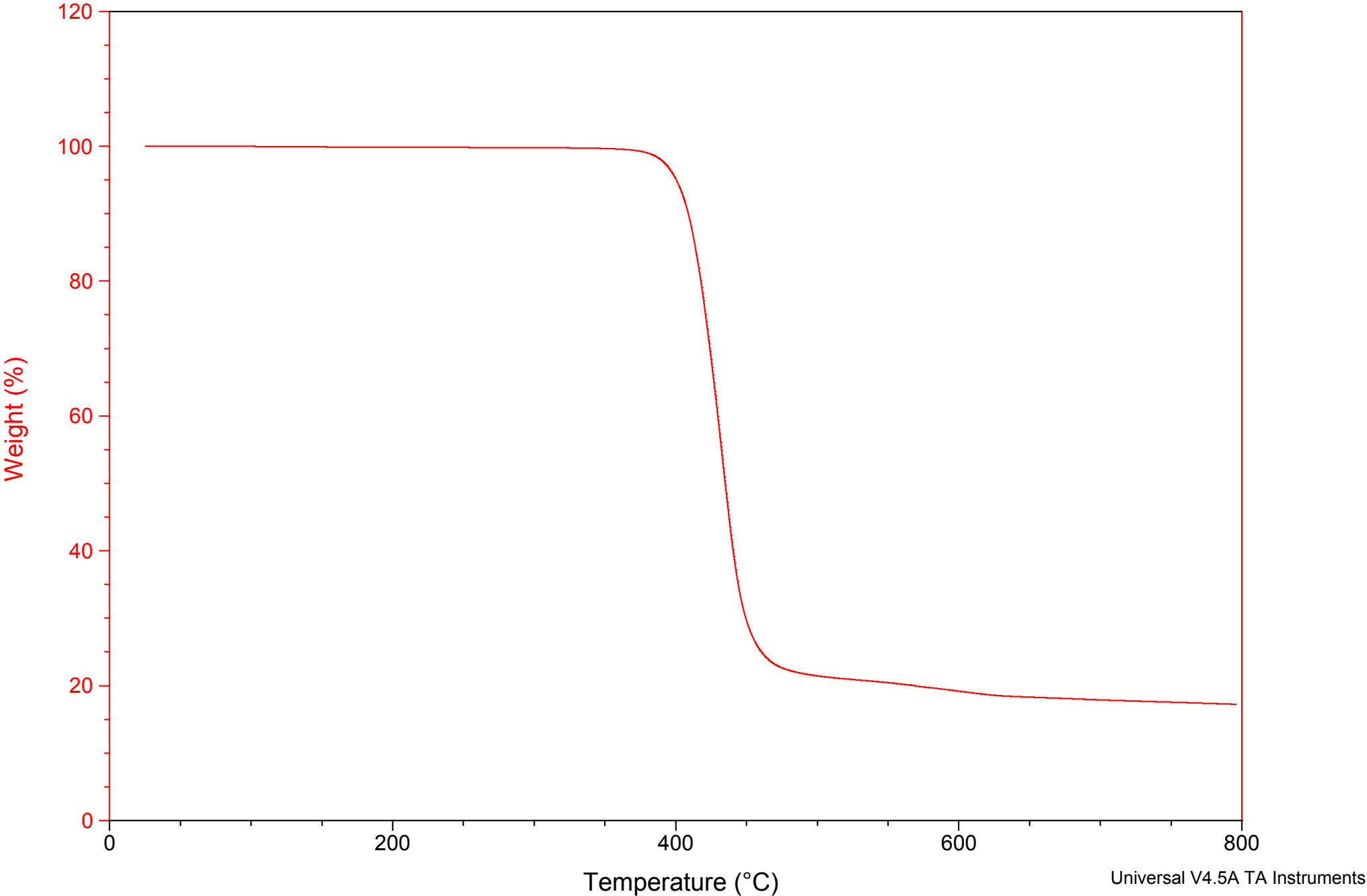
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Operator: Zhe
Run Date: 08-Feb-2019 10:58
Instrument: TGA Q50 V20.13 Build 39



Sample: PET #2
Size: 11.9120 mg
Method: Rubber

TGA

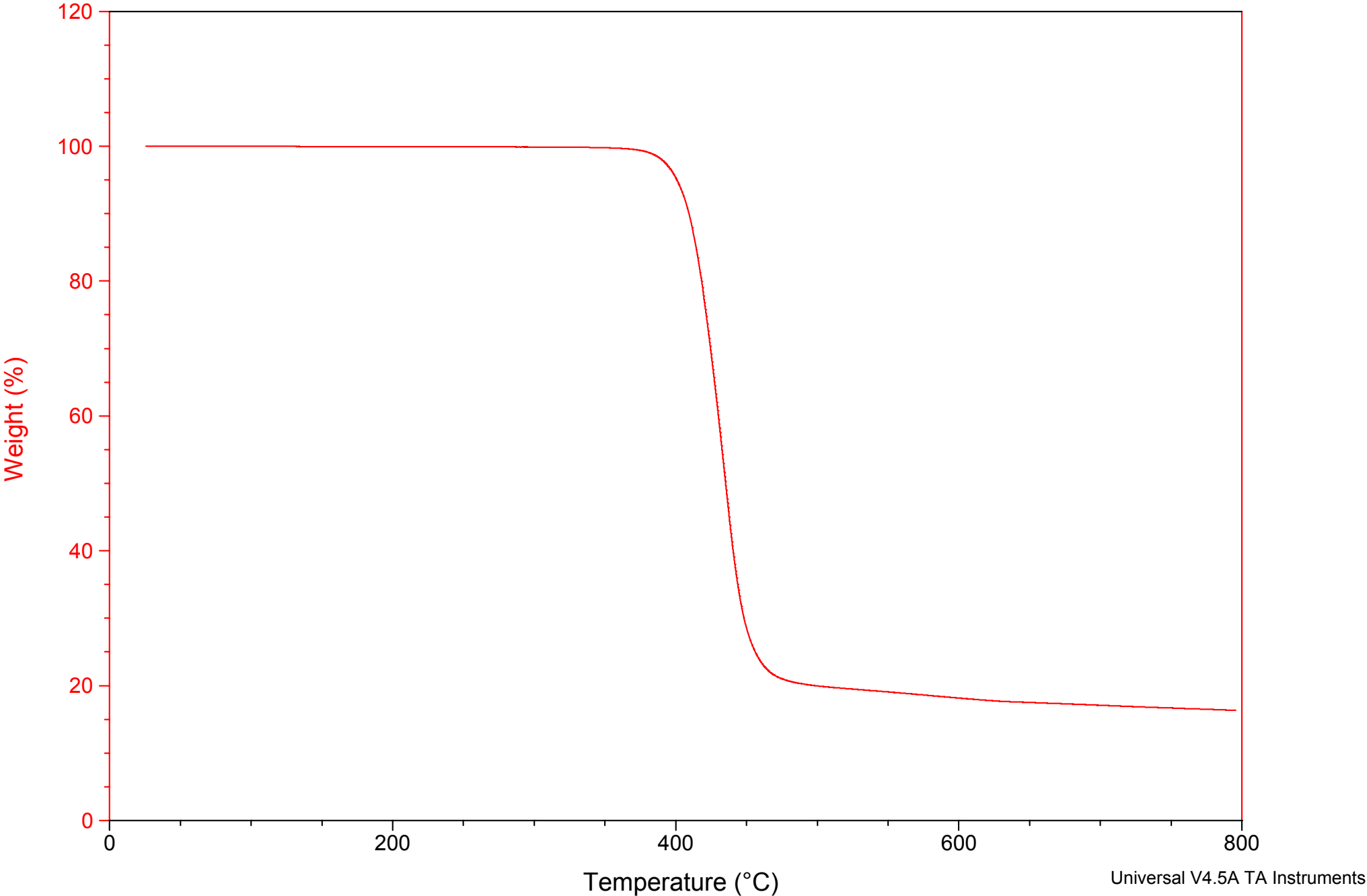
File: C:\TA\Data\TGA\ZHE L\PET#2.001
Operator: Zhe
Run Date: 07-Feb-2019 13:25
Instrument: TGA Q50 V20.13 Build 39



Sample: PET #2
Size: 13.3520 mg
Method: Rubber

TGA

File: C:\TA\Data\TGA\ZHE L\PET#2.002
Operator: Zhe
Run Date: 08-Feb-2019 13:27
Instrument: TGA Q50 V20.13 Build 39



Appendix 4 - ASTM Molding Conditions rPET cdme.osu.edu

	Parameters
Material	Recycled PET
Drying Condition	300 °F/4 hours/-40 F DF
Barrel Temp	540/540/530/520/510 °F
Mold Temp	27 °C
Filling Velocity	0.5 in/s
Packing Pressure	700 psi
Packing Time	20 sec
Cooling Time	35 sec



Molded ASTM Part using rPET



Appendix 5 – Processing Parameters for ASTM Specimens on Lulzbot 3D Printer

Izod Specimens - ASTM D256

Layer Height: 0.25 mm

Wall Thickness: 1 mm

Print speed: 45 mm/sec

Print Temperature: 270 °c

Initial Layer Temperature: 275 °c

Infill: 100%

Initial Layer Fan Speed: 0%

Fan Speed: 100%

Build Plate Adhesion: Brim – 5 mm width

Flexure Specimens ASTM D790

Layer Height: 0.25 mm

Wall Thickness: 1 mm

Print speed: 45 mm/sec

Print Temperature: 270 °c

Initial Layer Temperature: 275 °c

Infill: 100%

Initial Layer Fan Speed: 0%

Fan Speed: 100%

Build Plate Adhesion: Brim – 5 mm width

Tensile Bar ASTM D-638

Layer Height: 0.25 mm

Wall Thickness: 1 mm

Print speed: 45 mm/sec

Print Temperature: 265 °c

Initial Layer Temperature: 265 °c

Infill: 100%

Initial Layer Fan Speed: 10%

Fan Speed: 100%

Build Plate Adhesion: Brim – 5 mm width

Heat Towers Printing Conditions

Layer Height: 0.25 mm

Wall Thickness: 1 mm

Print speed: 45 mm/sec

Print Temperature: Range from 290 to 255 °c

Initial Layer Temperature: N/A

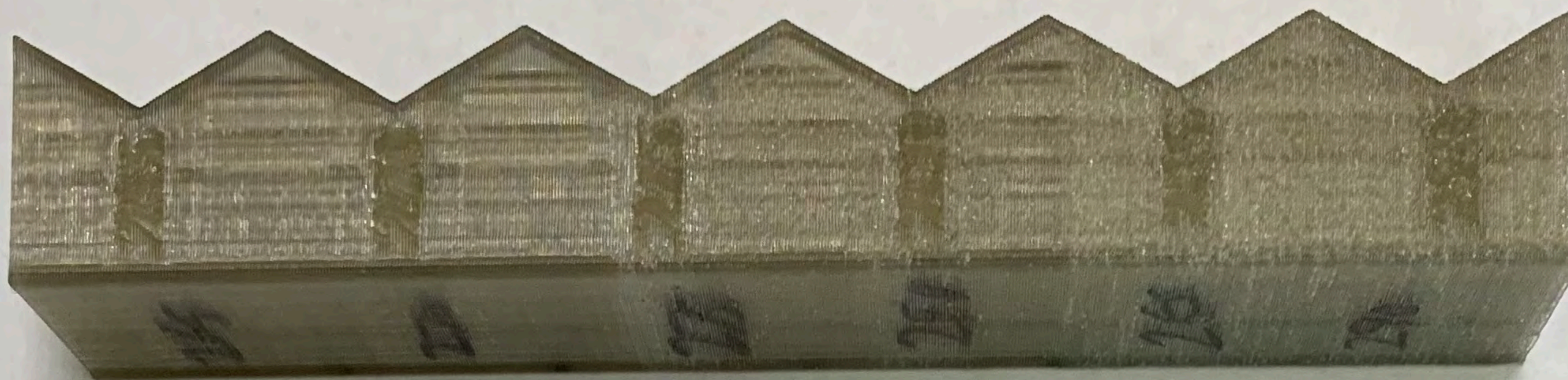
Infill: 15%

Initial Layer Fan Speed: 0%

Fan Speed: 100%

Build Plate Adhesion: Brim – 15 mm width

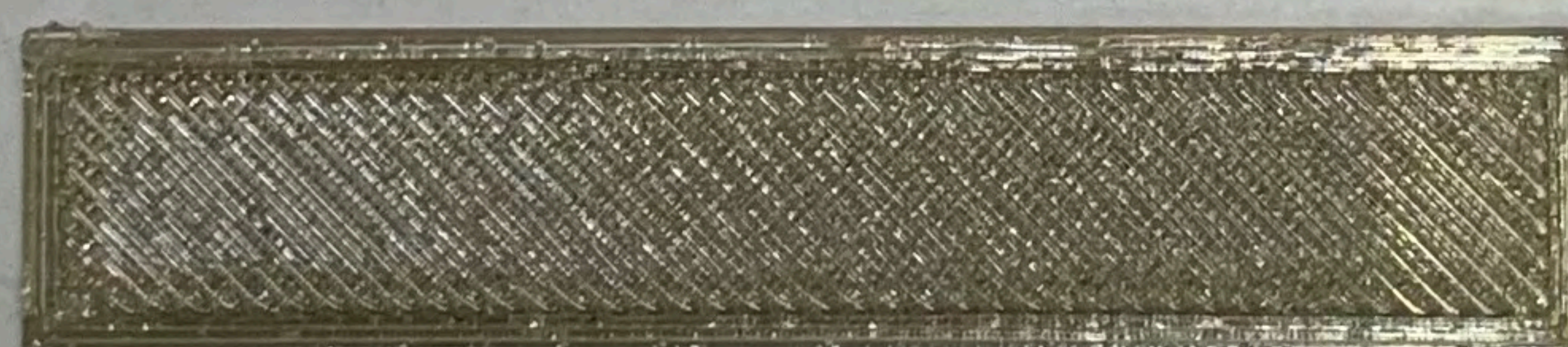
Heat Tower



Tensile Specimen (ASTM D638)



Izod Specimen (ASTM D256)



Flexure Specimen (ASTM D790)



Appendix 6 - ASTM Test Reports from Laboratories



ANALYTICAL
Services & Technology

Analysis Report

Requestor: Prabhat Krishnaswamy
Company: Engineering Mechanics Corp.
Columbus, OH 43221-1735

Our Ref: CF-0090213
Date: May 15, 2019
Primary Analyst: Garrett L. Schafer

Project: Material Testing

Copy to: File

Objective: Conduct ASTM Tests per PO #19-08
Tensile, Flex, HDT and Spec Gravity

Sample Name: **rPET-1**

PHYSICAL

Flex	Result File	0000146138.pdf
Heat Distortion Temp	Standard Testing Method	ASTM D648
Heat Distortion Temp	Heat Distortion Temperature	59.5 °C
Heat Distortion Temp	Heat Distortion Temperature	59.8 °C
Heat Distortion Temp	Mean HDT	59.7 ± 0.2 °C
Specific Gravity	Specific Gravity	1.333 ± 0.005
Tensile	Result File	0000146143.pdf

Sample Name: **rPET-2**

PHYSICAL

Flex	Result File	0000146139.pdf
Heat Distortion Temp	Standard Testing Method	ASTM D648
Heat Distortion Temp	Heat Distortion Temperature	62.2 °C
Heat Distortion Temp	Heat Distortion Temperature	61.5 °C
Heat Distortion Temp	Mean HDT	61.9 ± 0.5 °C
Specific Gravity	Specific Gravity	1.351 ± 0.004
Tensile	Result File	0000146144.pdf

Tests were performed per ISO 9001 using the following standard control methods:

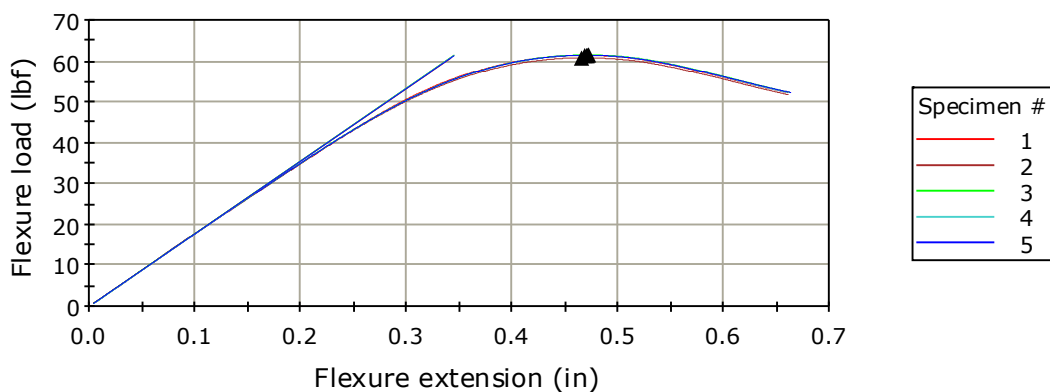
Specific Gravity

Ap-04f - Displacement

Ashland Inc.Physical Testing Lab
Instron Model 3366FLEX TEST - ENGLISH & SI UNITS
extensometer
ASTM D790 -[MAT 2203]

AF number	90213
SAMPLE ID	PET-1
Speed	0.10632 in/min
Support span	3.99000 in
Temperature	74°F
Humidity	46%
Operator name	GLS

Specimen 1 to 5



	Flexural Strength (psi)	Young's Modulus (ksi)	Strain at Yield (in/in)	SI Flexural Strength (MPa)	SI Young's Modulus (MPa)	Load at Yield (lbf)	Width (in)	Thickness (in)
1	11555	358.2	0.0439	79.67	2470	61	0.515	0.248
2	11173	346.1	0.0439	77.04	2386	61	0.520	0.250
3	11387	350.8	0.0444	78.51	2418	61	0.519	0.250
4	11409	352.5	0.0442	78.66	2430	61	0.519	0.249
5	11486	355.2	0.0441	79.19	2449	61	0.517	0.249
Mean	11402	352.5	0.0441	78.61	2431	61	0.518	0.249
Standard Deviation	144	4.6	0.0002	0.99	32	0	0.002	0.001

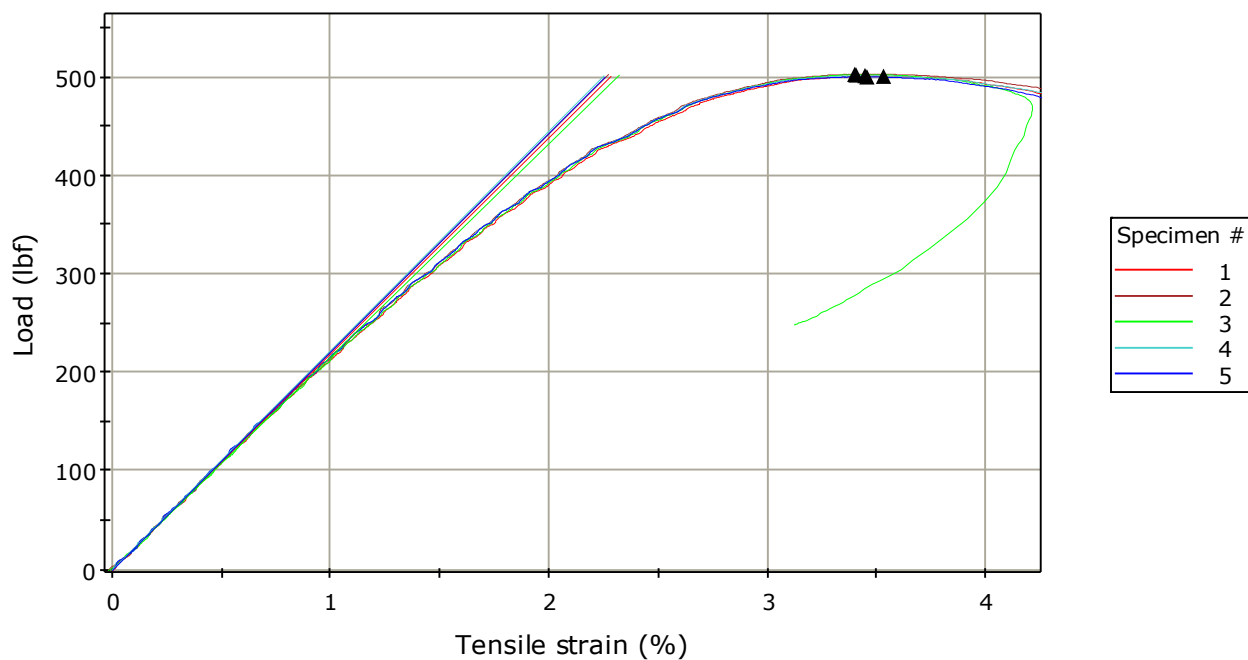
Ashland Inc.

Physical Testing Lab
Instron Model 4204 - Class A

TENSILE TEST - ENGLISH & SI UNITS
ASTM D-638 (MAT-2202)

AF number	90213
SAMPLE ID	PET-1
Crosshead speed	0.20 in/min
Temperature	72°F
Humidity	54%
Operator Name	GLS

Specimen 1 to 5

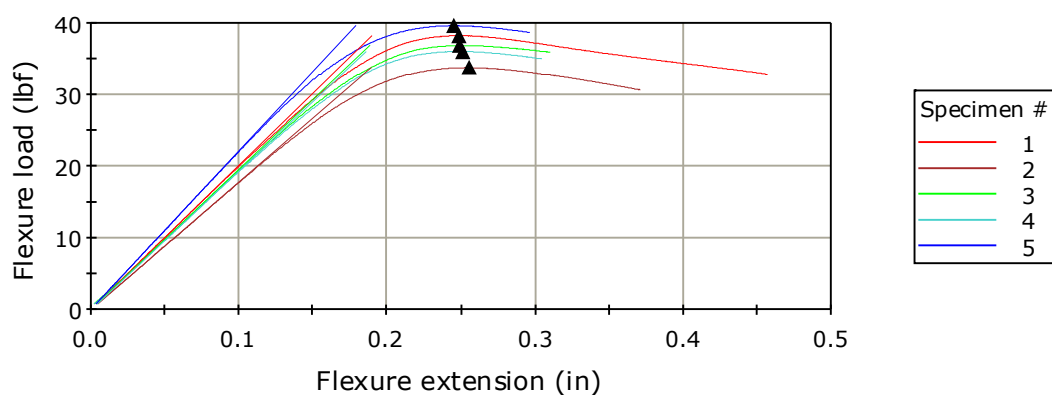


	Tensile Strength (psi)	Young's Modulus (ksi)	Elongation at Max (%)	SI Tensile Strength (MPa)	SI Young's Modulus (MPa)	Maximum Load (lbf)	Width (in)	Thickness (in)
1	7751	339.6	3.53	53.44	2341	501	0.512	0.126
2	7869	346.6	3.39	54.26	2390	503	0.511	0.125
3	7783	335.1	3.40	53.66	2310	502	0.512	0.126
4	7789	346.2	3.44	53.70	2387	501	0.513	0.125
5	7779	344.7	3.45	53.64	2377	500	0.514	0.125
Mean	7794	342.4	3.44	53.74	2361	501	0.512	0.126
Standard Deviation	44	5.0	0.05	0.31	34	1	0.001	0.000

Ashland Inc.Physical Testing Lab
Instron Model 3366FLEX TEST - ENGLISH & SI UNITS
extensometer
ASTM D790 -[MAT 2203]

AF number	90213
SAMPLE ID	PET-2
Speed	0.05690 in/min
Support span	2.13000 in
Temperature	74°F
Humidity	46%
Operator name	GLS

Specimen 1 to 5



	Flexural Strength (psi)	Young's Modulus (ksi)	Strain at Yield (in/in)	SI Flexural Strength (MPa)	SI Young's Modulus (MPa)	Load at Yield (lbf)	Width (in)	Thickness (in)
1	12658	372.4	0.0445	87.27	2567	38	0.526	0.136
2	12697	394.4	0.0434	87.54	2719	34	0.513	0.129
3	12905	390.0	0.0436	88.98	2689	37	0.520	0.132
4	12709	390.6	0.0440	87.63	2693	36	0.516	0.132
5	12577	385.9	0.0446	86.71	2661	40	0.531	0.138
Mean	12709	386.6	0.0440	87.62	2666	37	0.521	0.133
Standard Deviation	121	8.5	0.0005	0.84	59	2	0.007	0.003

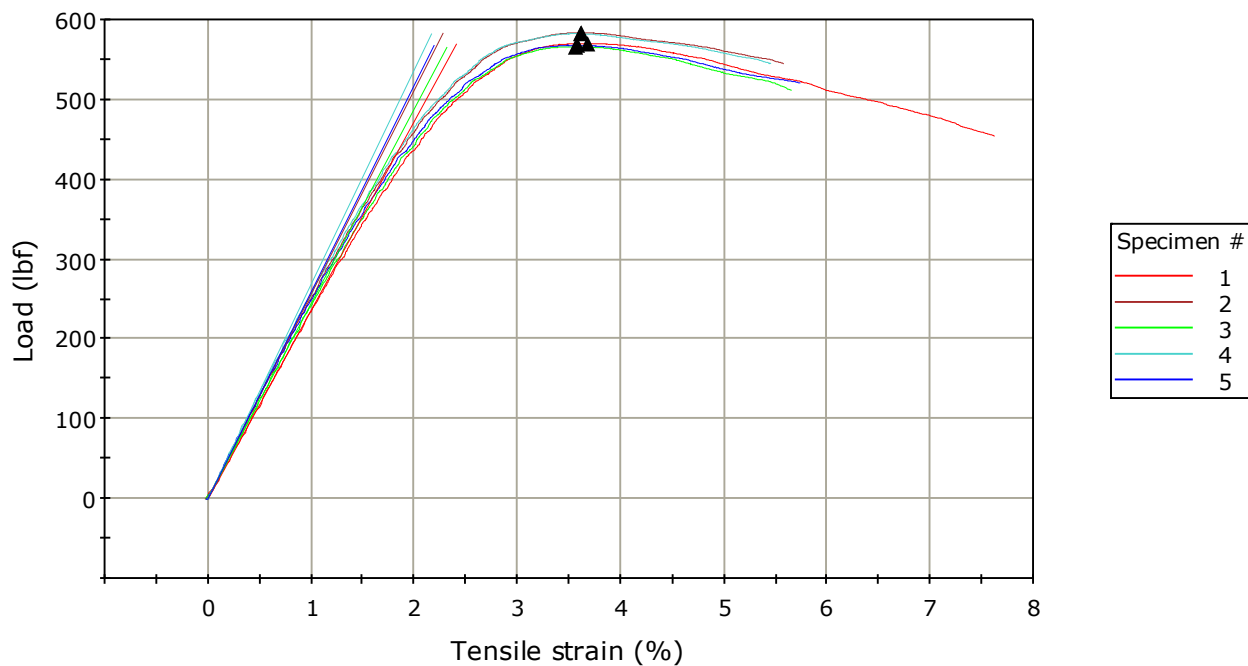
Ashland Inc.

Physical Testing Lab
Instron Model 4204 - Class A

TENSILE TEST - ENGLISH & SI UNITS
ASTM D-638 (MAT-2202)

AF number	90213
SAMPLE ID	PET-2
Crosshead speed	0.20 in/min
Temperature	72°F
Humidity	54%
Operator Name	GLS

Specimen 1 to 5



	Tensile Strength (psi)	Young's Modulus (ksi)	Elongation at Max (%)	SI Tensile Strength (MPa)	SI Young's Modulus (MPa)	Maximum Load (lbf)	Width (in)	Thickness (in)
1	8531	356.1	3.68	58.82	2455	570	0.517	0.129
2	8709	382.2	3.62	60.05	2635	583	0.514	0.130
3	8558	369.8	3.56	59.01	2550	566	0.514	0.129
4	8710	402.3	3.61	60.05	2774	582	0.515	0.130
5	8542	390.0	3.58	58.90	2689	568	0.515	0.129
Mean	8610	380.1	3.61	59.36	2621	574	0.515	0.129
Standard Deviation	91	17.9	0.04	0.63	123	8	0.001	0.001

Analysis Report

Requestor: Prabhat Krishnaswamy
Company: Engineering Mechanics Corp.
Columbus, OH 43221-1735

Our Ref: CF-0091086
Date: October 29, 2019
Primary Analyst: Thomas H. Grentzer

Project: Tensile and Izod Testing

Copy to: File

Objective: Need testing of 5 tensile specimens for PET material with ID L3D per ASTM D638.

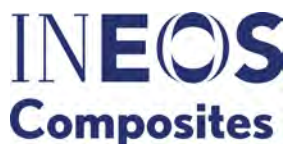
Sample Name: **L3D (PET)**

PHYSICAL

Specific Gravity	Specific Gravity	1.262 ± 0.016
Tensile	Result File	0000152626.pdf

Tests were performed per ISO 9001 using the following standard control methods:

Specific Gravity	Ap-04f - Displacement
------------------	-----------------------

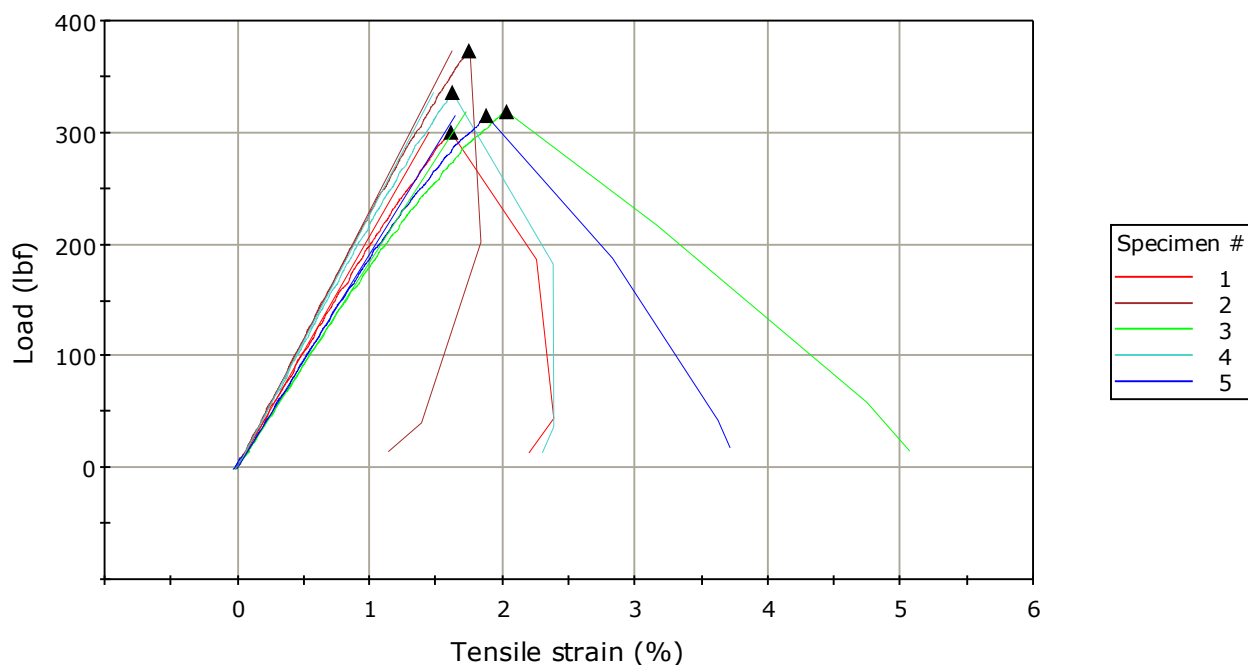


Physical Testing Lab
Instron Model 4204 - Class A

TENSILE TEST - ENGLISH & SI UNITS
ASTM D-638 (MAT-2202)

AF number	91086
SAMPLE ID	L3D PET
Crosshead speed	0.20 in/min
Temperature	72°F
Humidity	54%
Operator Name	GLS

Specimen 1 to 5



	Tensile Strength (psi)	Young's Modulus (ksi)	Elongation at Max (%)	SI Tensile Strength (MPa)	SI Young's Modulus (MPa)	Maximum Load (lbf)	Width (in)	Thickness (in)
1	3786	262.0	1.61	26.10	1807	300	0.532	0.149
2	4464	275.7	1.74	30.78	1901	373	0.535	0.156
3	4129	239.5	2.03	28.47	1651	319	0.528	0.146
4	3892	263.8	1.62	26.83	1819	336	0.545	0.158
5	4173	253.9	1.88	28.77	1751	315	0.525	0.144
Mean	4089	259.0	1.77	28.19	1786	329	0.533	0.151
Standard Deviation	265	13.4	0.18	1.82	92	28	0.008	0.006

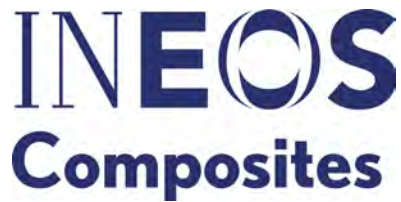
Analysis Report

Requestor:	Prabhat Krishnaswamy	Our Ref:	CF-0091297
Company:	Engineering Mechanics Corp. Columbus, OH 43221-1735	Date:	December 16, 2019
		Primary Analyst:	Thomas H. Grentzer
Project:	ASTM Testing		
Copy to:	File		
Objective:	Need testing of Flexure, Izod, HDT (DTUL) and Hardness tests on PET material with ID L3D per ASTM D790, D256, D648, and D2583. Specimens will be provided.		

Sample Name: **L3D (PET)**

PHYSICAL

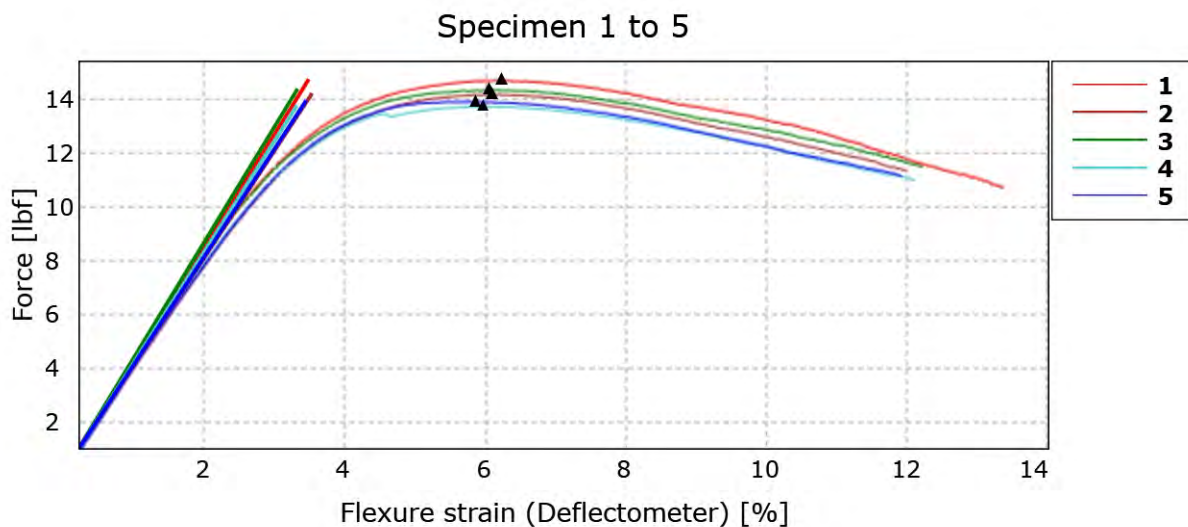
Flex	Result File	0000154364.pdf
Hardness	Standard Test Method	Shore D MAT-2258
Hardness	Shore D	50.7 ± 2.8
Heat Distortion Temp	Standard Testing Method	ASTM D648
Heat Distortion Temp	Heat Distortion Temperature	65.7 °C
Heat Distortion Temp	Heat Distortion Temperature	65.6 °C
Heat Distortion Temp	Mean HDT	65.7 ± 0.1 °C
Izod	Standard Testing Method	ASTM D256
Izod	Notched Specimen Energy	0.41 ft-lbs/in
Izod	Notched Specimen Energy	0.69 ft-lbs/in
Izod	Notched Specimen Energy	0.45 ft-lbs/in
Izod	Notched Specimen Energy	0.41 ft-lbs/in
Izod	Notched Specimen Energy	0.56 ft-lbs/in
Izod	Notched Energy	0.50 ± 0.12 ft-lbs/in
Izod	Notched SI Energy	26.91 ± 6.45 J/m



Physical Testing Lab
Instron Model 5966

FLEX TEST - ENGLISH & SI UNITS
With extensometer
ASTM D790 -[MAT 2203]

AF number	91297
SAMPLE ID	L3D PET
Rate 1	0.05859 in/min
Support span	2.20000 in
Temperature	70°F
Humidity	57%
Operator name	GLS



	Flexure Strength [psi]	Young's Modulus [ksi]	Strain at Yield [in/in]	Flexural Strength [MPa]	Young's Modulus [MPa]	Load at Yield [lbf]	Width [in]	Thickness [in]
1	5247	149.7	0.0624	36	1032	15	0.495	0.137
2	4974	139.9	0.0611	34	965	14	0.497	0.138
3	5096	152.3	0.0607	35	1050	14	0.493	0.137
4	4884	146.4	0.0599	34	1009	14	0.495	0.137
5	4951	142.8	0.0586	34	985	14	0.492	0.138
Mean	5031	146.2	0.0606	35	1008	14	0.494	0.137
Standard deviation	143	5.0	0.0014	1	34	0	0.002	0.000



ATTACHMENT - 2

IC3D Printers – Prepared by Luke DeBruin for EMC2

Date Prepared: 7-16-2021

Extrusion process pictures

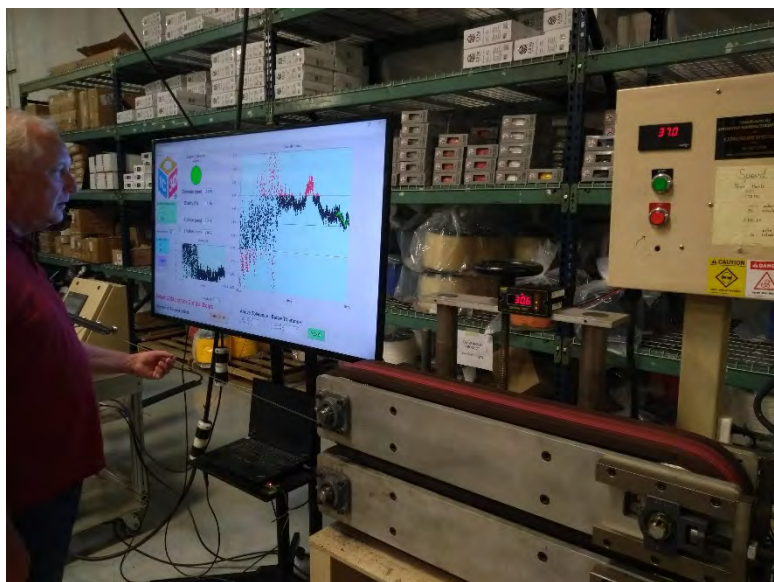


Figure 1: Initial tuning of rPET only (no Kraton)



Figure 2: Extruder settings for rPET only

Video Link to extrusion trials:

<https://photos.app.goo.gl/8MT7RuEXExc6WrWJ9>

rPET Flake w/ 10% TPE

Date Produced: 7-9-21

Material:	NLF 4 MM LNO FLAKE	Manufacturer:	Phoenix Technologies
Grade:		Supplier:	Phoenix Technologies
Lot/Batch:	?		

YIELD REPORT STARTING WEIGHT Had to dry all available >50 lbs

	1.75MM		2.85MM
Start Purge			~5 lbs
Settings Discovery			~7 lbs
Product Yield			3x 1kg spools rPET, 4x 1kg spools Kraton
Out of Spec Product			Not measured
Left over			Not measured
Total			~35 lbs used
Viscosity	Low	Draw down	High ie)
Melt Strength	Lower; acceptable	Die swell	Max allowable for stable ext.
Melting Point	Lowest allowable (450F)	Set up	Normal

Kraton additive processing guide said drying not required, but TPEs normally need to be very dry to get the best processing results. The material was dried in a dehydrator at 155F for 24 hours prior to mixing with the base rPET. Even still, filament exhibited moisture pock marking on surface most likely due to additive not being fully dry. rPET only did not have the same surface blemish issue.

THREADING/START-UP REVIEW

Material string up like normal using slow starting speed and cooling initial few inches of strand.			
Processing Parameters			
Dry Time	12+ hours prior to start	Speed	Medium
Dry Temperature	245°F	Pressure	Low
Tension and winding was easy to achieve desired results with normal set up			

QUALITY

Tolerance	A-B
Ovality	A-
Stability	B-C

PROCESS SETTINGS

	rPET	Startup	rPET w/ Kraton
Extruder	34	4	44
SCREW AMP	8.2		7.6
PULLER FPM	37.7	~20	37.7
MELT PRESS PSI	2330	?	1750
MELT TEMP	?	?	?
SCREEN PACK	20-80-60	20-80-60	20-80-60
ZONE 1 (F)	415	385	415
ZONE 2 (F)	450	420	450
ZONE 3 (F)	425	420	425
DIE (F)	365 (362 actual)	355	375 (372 actual)
WATER HEATER	165F	165F	165F
BATH	138F	138F	138F
DISTANCE FROM DIE	6"		6"

188 feet equal approximately 1 pound of material

Processing speed was approximately 13 pounds per hour

(acceptable speed for commercial process)

SPOOL NUM	WEIGHT	QUALITY	NOTES
rPET only			
1-1	~1kg	A	+/- 0.5mm and 1-2% ovality – typical specs for commercial
1-2	~1kg	A	Same as above
1-3	~1kg	A	Same as above
rPET w/ Kraton			Quality worsened w/ TPE 10% addition (+/- 0.1mm and 2% ovality)
2-1	~1kg	B	Best quality of the Kraton spools
2-2	~1kg	C	Should not jam Taz6; will jam more sensitive machines
2-3	~1kg	C	Same as above; possible intermittent overdiameter to 3.1mm
2-4	~1kg	C	Same as above; possible intermittent overdiameter to 3.1mm
TOTAL	~7kg	B	



Figure 3: Left shows overdiameters removed in post extrusion quality check – rPET alone had far less problems. Right shows closeup of extrusion contaminant from rPET regrind process –appears to be aluminum



Figure 4: Final packaged material provided to EMC2 for pickup

Extrusion Notes

Extruder settings

In my experience running PETG and ABS, I need to maintain around 3000 PSI minimum to get stable extrusion; I chose to adjust the extruder temp profile in such a manner to maximize the pressure before the die while avoiding running too cold. At first the startup temps were acceptable, but were lowered too much which led to the formation of unmelts in the extrudate – the temps of zones 1-3 were slowly raised until these fully melted.

Water bath distance

In order to stabilize the extrusion the die temp being colder and having the reverse extrusion profile led to the best results. The distance between the die and bath is an important consideration but the drooping effect is best mitigated by running the die colder. The Kraton additive further stiffened the material and decreased the droop between the die and the water bath diaphragm.

Water bath temperature

The water bath temperature with a tank temperature of around 135-140°F (138F actual). This material likes a hot bath – ovality issues will arise if the deltaT from the material temp to the water temp is too great. This is a general rule of thumb for low viscosity materials.

Die

Using a 5mm die and having significant drawdown allowed the best specs to be achieved. The cold die temp allowed for extrudate stability between die and water bath entrance. The die temp is critical and lowering to 355F caused die swell which lead to the extrudate swirling as it cooled. This led to instability in extrusion and poor specs; keeping the die just hot enough to avoid this at 365F gave great results. The setpoint for the die was 365, but the actual die temp stabilized and maintained at 362F. Die buildup on the land was a concern and further investigation of this may be necessary if the product is ran for long periods of time as it will slough off and cause intermittent single occurrence out-of-spec.

Conclusion

rPET flake material viability as 3D printing filament is good. The rPET flake as provided posed no issue to extrusion as far as feed throat bridging and material handling is concerned. The material has a lot of powder and further reduction of this powder is desirable.

Processing using a reverse extruder temp profile led to good results for diameter and ovality and stopped un-melts from disturbing the extrusion, but allowed for cooldown of the material to achieve high enough viscosity for stringing the material from the die to the entrance of the hot bath. This material likes a hot water bath (probably could go hotter up to 165F), which would allow for faster extrusion speeds. Material ran as fast as other commercial grade filament production, but with the TPE additive would not meet our filament commercialization specs- would need to achieve +/- 0.5mm diameter tolerance and eliminate extrusion instability. This should be possible with further tuning. Commercialization outlook promising.

The addition of the TPE additive worsen the specs and caused some extrusion instability compared to the rPET alone. The TPE additive is however critical to successful 3D printing characteristics. These can be further tuned out or dealt with without changing the formulation.

Printing with rPET +10% Kraton TPE

Print Settings

Extruder

Nozzle	0.5	
Extrusion Multiplier	1.05	
Extrusion Width	0.6	
Ooze Control	ON	
Retraction	ON	
Retraction Distance	2	mm
Retraction Lift	0.1	mm
Retraction Speed	30	mm/s

Layer

	0.2	mm
Infill	10	%
Top Solid Layer	5	
Bottom Solid Layer	4	
Outline Shell	1	

Temperatures

Heated Bed	80	°C
Extruder	255	°C
Cooling Fan	44	%

Speeds

Print Speed	70	mm/s
Outline Speed	75	%
Infill Speed	80	%
Support Speed	80	%
X/Y Movement	250	mm/s
Z Movement	16.7	mm/s

Printing Notes

Quite forgiving to print for rPET. The 10% TPE additive makes all the difference and helps with layer and bed adhesion once the correct extrusion temperature is achieved. 255-265C gave good results, but 250C was too cold and caused intermittent jamming. At 265C significant nozzle oozing occurred and led to print failure and buildup of blobs on the part. 80C Bed temp 80C worked well using the powder-coated magnetic bed. The TPE also reduces the part warping considerably and increases the overall strength by reducing the brittleness of the rPET.

The ‘3Dbenchy’ that was printed showed some stringing, however the material bridges quite well at 255C and 40% fan. Further recommendations for tuning would be to reduce stringing by increasing the retraction length and speed slightly. Also further tuning the fan speed %, nozzle temp, and /or print speeds can likely achieve similar results.



Figure A: Calibration 'L' bracket printed w/ no infill and single wall

The print shown in the foreground of Figure A was started at 250C nozzle temp and raised to 255C half way through the single wall part. After printing, pliers were used to test the tensile strength of the walls/layer bonding and the 250C portion failed along the layer line indicating poor layer bonding. Farther up the part the same tensile force and side bending force was applied to the 255C portion (see red circle) and the part did not fail – this test was used to determine the appropriate nozzle temp for the printing profile.

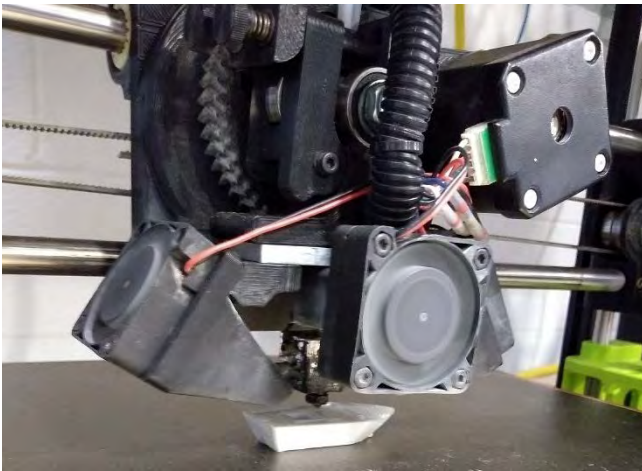


Figure B: 3DBenchy printing as test print on Taz6 w/ 0.2mm layer height and 0.5mm brass nozzle at 255C



Figure C: Benchy front view showing close-up of stringing. Filament has excellent bridging characteristics and captures detail well with given settings

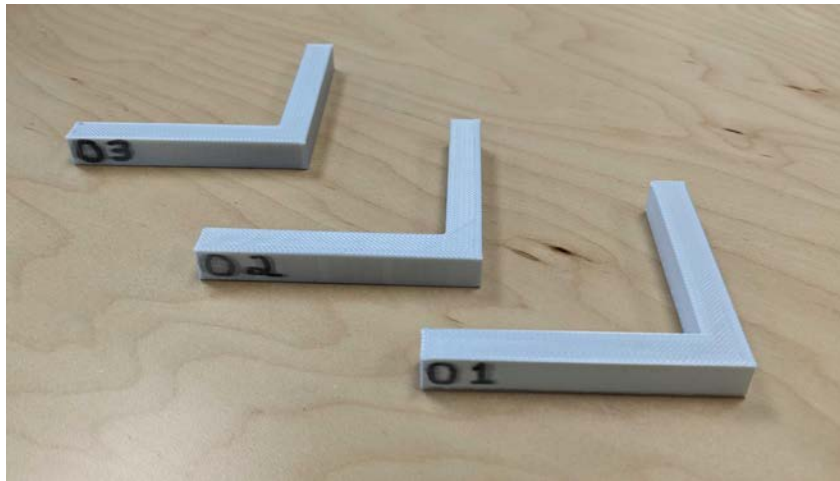
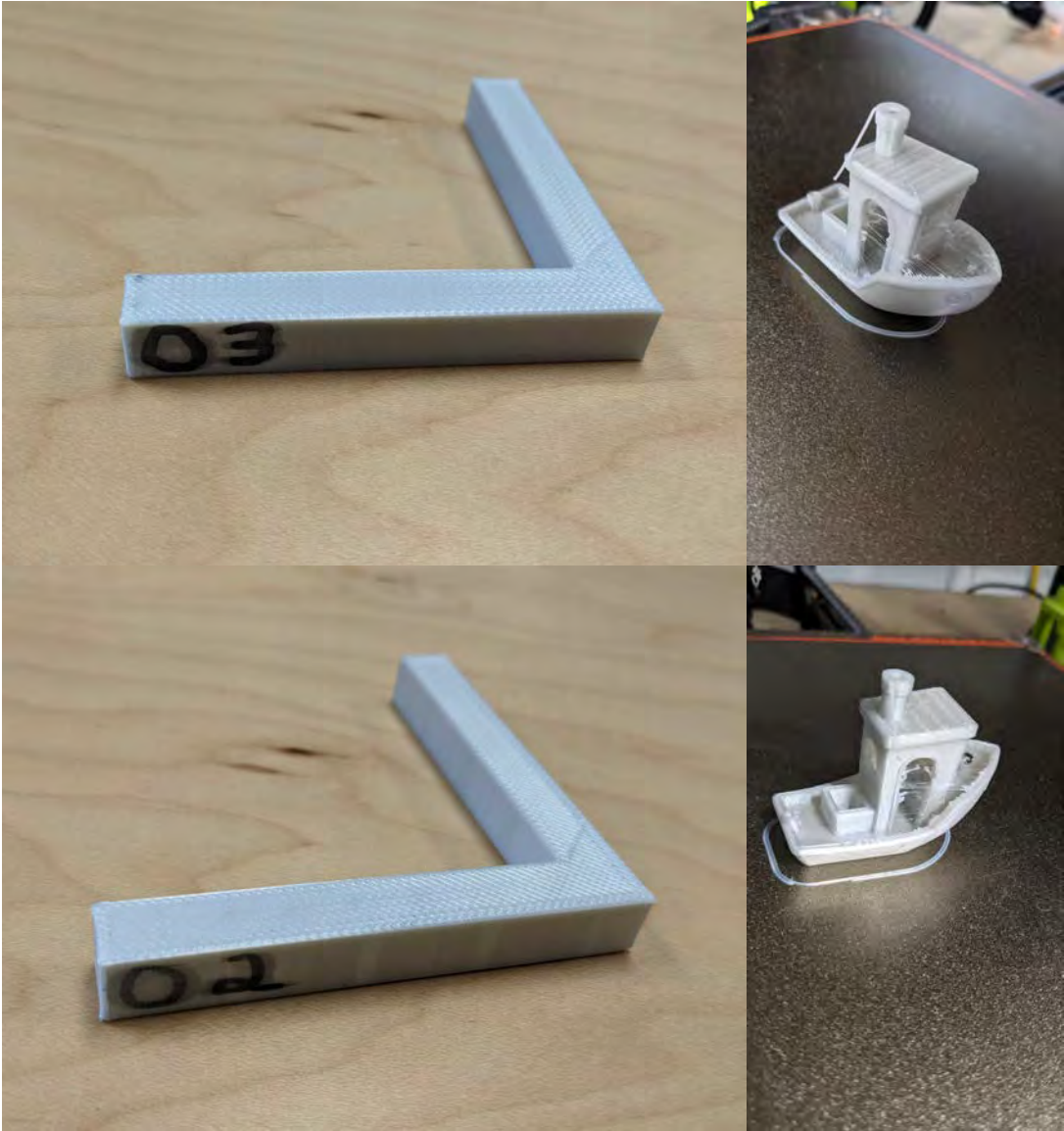
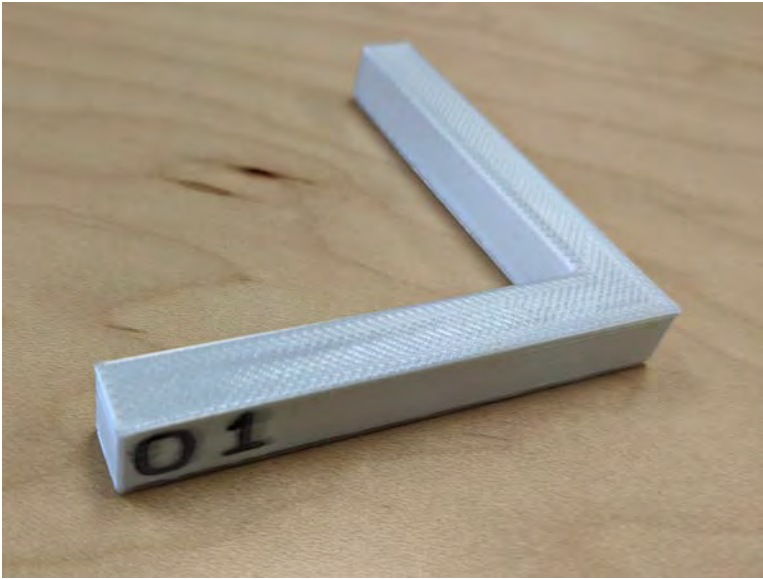


Figure D: Calibration L bracket test prints at 255C nozzle. Sample 1 is single wall w/ no infill. Sample 2 is 10% infill and Sample 3 is 40-50% infill. All printed well and showed very low warp.

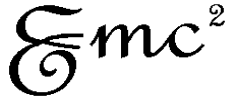
Close-ups of printed parts provided to customer:





Appendix A-16

Design Guide for rPET Additive Manufacturing



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Columbus, Ohio 43221

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DRAFT

Date: December 30, 2022

To: Mr. Braxton Lewis, Noblis, (Braxton.Lewis@noblis.org;
Braxton.V.Lewis.ctr@mail.mil), Project Manager

From: Dr. Prabhat Krishnaswamy (kswamy@emc-sq.com), Principal Investigator, and
Mr. Rick Heggs (rheggs@emc-sq.com), Emc²

CC: Dr. Robin Nissan, SERDP (robin.a.nissan.civ@mail.mil), COR, and Dr. Nancy
E. Ruiz CIV USN (US) (nancy.ruiz@navy.mil), COR

Subject: SERDP Project No. WP18-1047, Task 4 - Evaluation of rPET Products for use at
FOBs, Deliverable Report on '**Design Guide for rPET 3D Filament in Printers**'

Project Background

The contract for the above Project was finalized and the project initiated on June 21, 2018. Due to the COVID pandemic a no-cost extension of this project was approved until March 31, 2023. There are five major Tasks being conducted in this effort listed below:

- Task 1 - Sorting of FOB Waste Streams for rPET
- Task 2 - Development of rPET based Formulations
- Task 3 - Modification of Extrusion Processing Technologies
- Task 4 - Evaluation of rPET Products for use at FOBs
- Task 5 - Program Coordination, Reports and Deliverable

Dr. Krishnaswamy and Mr. Rick Heggs have led all the Tasks above along with Dr. Pete Stynoski of the US Army Corps of Engineers ERDC-CERL, the directly funded partner (DFP) on this project.

Overview of Deliverable

This Deliverable Report under Tasks 3 and 4 of the work above describes the trial runs conducted to design a 3D filament extrusion line for rPET materials obtained from waste PET water bottles at FOBs. The key constraint, in the design of such an agile, mobile manufacturing line is that the entire plant must be contained in 20-foot ISO Containers. That is, the manufacturing line can be fabricated state-side and shipped to FOBs in ISO Containers for installation and operation in remote sites using locally available feedstocks such as waste PET water bottles.

The various steps in developing a Design Guide for rPET 3D Filament for Printers involved the following:

- i. Procure rPET materials, both flake and pellets, stateside (since obtaining rPET from FOBs was deemed not feasible by the ERDC-CERL team members on this project),

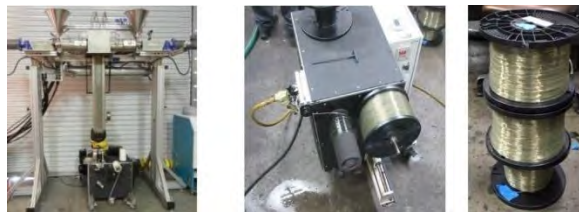
- ii. Characterize the rPET materials by determining their physical, thermal and mechanical properties,
- iii. Conduct trials to extrude 3D printer filament in both 1.75 mm and 2.85 mm diameter at several locations and varying technology using first the rPET pellets, and then with the rPET flakes,
- iv. Print calibration parts, ASTM specimens, and other sample parts/ components - similar to those suitable for FOB applications with the above rPET filament samples using various printers at different laboratories,
- v. Determine the properties of 3D printed specimens from above trials and compare them to traditional materials used for AM (such as PLA, ABS, etc.) to determine suitability for one-to-one replacement of parts needed at FOBs.

The above work covers various activities in Tasks 2 – 4 of the project all of which are inter-connected and inter-dependent. Previous deliverables on this project cover several of the items listed above and are included in their entirety in as Attachment 1 for completeness, and for brevity will not be repeated here. This Deliverable Report will specifically focus primarily on Item (iii) above along with a summary of Items (iv) and (v). The Final Project Report, currently under preparation, will cover all tasks and activities and is scheduled to be delivered and finalized by the end of the POP, March 31, 2023.

Two methods of filament production with rPET materials were investigated, Filament Spinning, and Conventional Extrusion. The trials with each of these technologies is described next.

Filament Production Trials with Fiber Spinning

Fiber spinning is a mature technology that has been used extensively in converting rPET materials into very small diameter monofilament for textile and carpet applications. Therefore, this was the first technology investigated to produce rPET filament for AM applications. Hills Inc. from West Melbourne, FL is a major worldwide supplier of equipment in this industry. After review of their capabilities, their laboratory was contracted to conduct trials for filament production, specifically because their equipment configuration is very compact and could readily be installed in a single 20-foot ISO Container for FOB applications. After some preliminary equipment and process condition issues, the trials at Hills were successful in producing both the 1.75 and 2.85mm AM filament from rPET pellets with an intrinsic viscosity of 0.7. Figure 1 below shows the results of these trials and the AM filament being spun into a spools



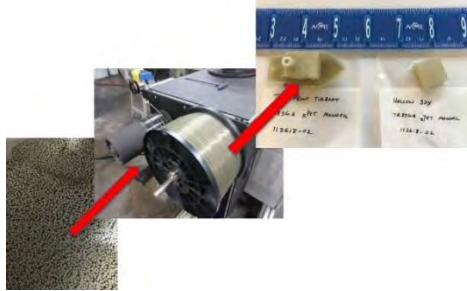


Figure 1- Compact lab equipment used for fiber spinning 3D printer filament from rPET pellets and sample calibration parts printed from filament

After being dried per the specifications (250 F for a minimum of 4 hours or equivalent), the AM filament from the fiber spinning trials were then used with several printers in various labs at Emc² and ERDC-CERL. While simple calibration items could be printed successfully, it was found that the consistency and ovality of the filament varied beyond the tolerance specifications of the 3D printers. Hence one frequent problem involved ‘clogging’ of the printer head due to excess ovality. A second set of trials were then conducted at Hills to investigate if filament ovality could be controlled to the tolerance required. These trials indicated that Hills’ equipment and technology can produce a much finer (smaller diameter) textile grade fiber consistently as ovality requirements are not stringent. However, controlling larger diameter (1.75 mm and 2.85 mm) filament ovality to the required specifications was not very reliable. After further discussion with Hills, the fiber spinning technology was not deemed feasible for FOB applications and was therefore not pursued further in the project.

Filament Production Trials with Conventional Extrusion

The next set of trials conducted involved investigating conventional filament extrusion technologies currently used extensively for AM to determine if they can be modified for installation and operation in one or possibly two 20-foot ISO containers for FOB applications. These trials were conducted successfully at three separate production / lab facilities as summarized below.

FilaMint, Chesterland, OH: After several preliminary discussions and at the recommendation of IC3D, a local manufacturer of commercial 3D filament in Central Ohio, FilaMint was selected for the initial trials. FilaMint is a small company with equipment that can easily be re-configured to suit rPET filament manufacturing. Emc² staff worked closely with FilaMint to conduct several trials modifying the extrusion conditions and processing parameters as required for rPET materials.

The results from the FilaMint trials and their reports and recommendations for the process settings are included in Attachment 2. Both 1.75mm and 2.85 mm filaments were successfully made using not only the rPET pellets used previously, but also clean 4-mm rPET flakes. This latter result is a major breakthrough in rPET filament extrusion technology as it eliminates the need for pelletizing the flake prior to filament production. Based on the success of these trials FilaMINT with Emc2’s help designed a complete 3d filament extrusion plant that could be shipped in a single 20-foot ISO container and then operated using an addition 20-foot container at the FOBs. Figure 2 shows the various pieces of equipment and their configuration for this mobile filament extrusion line for FOBs.

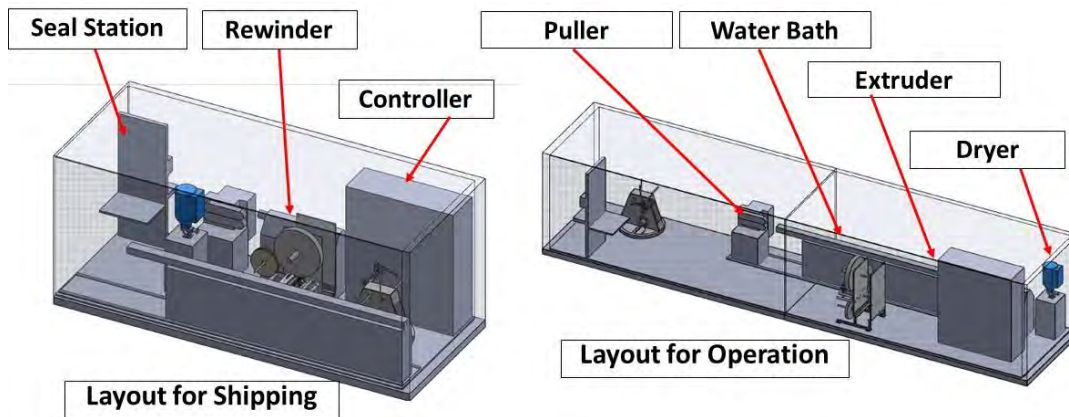


Figure 2 – Mobile 3D printer filament extrusion line in 20-foot ISO containers

An earlier deliverable from this project provided detailed information along with budgetary costs for each piece of equipment in the filament extrusion plant shown in Figure 2. For completeness, this earlier deliverable from September 2020 is included in Attachment 3.

Conair, Pinconning, MI: Conair is one of the premiere suppliers of plastics processing equipment in the USA. They are the best known producer of complete filament extrusion lines for the 3D printing industry. Based on the success of the above trials at FilaMint, a second trial was scheduled at Conair to determine if any major modifications to the system design and shown in Figure 2 was necessary for FOB applications. In any future ‘Demonstration and Validation’ phase for a follow-on ESTCP project, Conair would be more appropriate than FilaMINT for fabricating, installing and testing a filament extrusion line.

Trials were therefore conducted at Conair using both rPET pellets and clean 4-mm flake to make 2.85 mm diameter filament, see Figure 3 below. These trials were coordinated under Emc² staff supervision using the processing conditions that were deemed optimal based on the earlier effort at FilaMint described above.



Figure 3 – Photograph of rPET 3D printer filament extrusion trials at Conair

The trials at Conair established that rPET flake could successfully be used to meet commercial grade filament with < 0.05% ovality at a rate that exceeded 50 feet per minute. The test report along with Conair's commercial brochure for 3D filament production is provided in Attachment 4, as these would be useful for a follow-on ESTCP Project for Demonstration and Validation.

IC3D, Hilliard, OH: At the 2021 IPR meeting, one of the recommendations made was to investigate the use of additives to enhance the material properties of rPET parts. Specifically, since rPET can have very low ductility, additive packages could enhance the elongation to failure and also the impact strength. Therefore, a third set of 2.85 mm 3D printer filament extrusion trials were undertaken at IC3D, a commercial filament manufacturer for 3D printers, again, under the supervision of Emc² staff. The primary objective of this trial was to repeat the production of rPET filament from clean flake as well as produce filament with a 10% by weight Kraton (FG1901) additive package (detailed in Attachment 1). The choice of additive and the percent by weight loading was based on earlier trials conducted in a separate activity in the project involving rPET based plastic lumber extrusion.

The IC3D trials were again very successful in making 2.75 mm filament both with and without the additive. It was noted that the additive package enhanced both the processability, printability and the surface finish of the final part along with enhancing the ductility by 46%, as detailed in Attachment 1. The complete report from the IC3D trials is included in Attachment 5.





Figure 4 – Photo of IC3D trials to make 2.85 mm rPET 3D filament on commercial production equipment with additive (10% Kraton; opaque) and without additive (translucent)

Summary of 3D Filament Test Results

As detailed in Attachment 1, once the above trials were completed, standard ASTM specimens were printed at three laboratories (Emc², ERDC-CERL, and IC3D) to determine the physical, mechanical and thermal properties and compare the effect of 10% Kraton additives. These data are shown below in Table 1 along with those for other standard materials used for AM, i.e. PLA, PETG, and ABS. As highlighted in Table 1, the tensile elongation value (ductility) of the rPET increases significantly with the use of 10% Kraton, from 1.82% to 2.66% , that is an increase of 46%. This would significantly reduce the ‘brittleness’ of rPET materials making it much more effective for use in printing at FOBs.

Table 1 – Properties of 3D printed rPET materials with and without 10% Kraton and comparison with other AM materials such as PLA, PETG, and ABS

Property	ASTM Test		Units	rPET- without Kraton	rPET-with 10% Kraton	PLA	PETG	ABS
Sp. Gravity	D792			1.149	1.344	1.177	1.247	1.006
Flexure	D790	Strength	psi	8334	7758	12616	9436	6806
	D790	Modulus	ksi	253	254	461	277	220
	D790	Yield Strain	in/in	0.054	0.0454	0.047	0.053	0.054
Tensile	D638	Strength	psi	3697	3065	5824	5958	4336
	D638	Modulus	ksi	230	164.2	401	276	262
	D638	Elongation	%	1.820	2.66	2.160	2.930	2.160

A graph of the Tensile Elongation property from the above table is also shown in Figure 5 below along with those for injection molded rPET specimens.

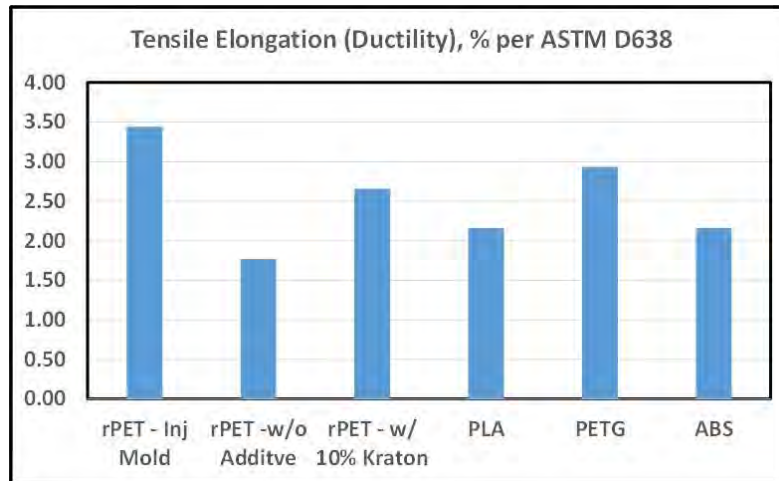


Figure 5 – Graph of Tensile Elongation (ductility) of various formulations of rPET in comparison with standard AM materials PLA, PETG, and ABS.

Concluding Remarks

Based on the above trials, it was concluded that the preliminary design for the filament extrusion line, shown in Figure 2 above and detailed in Attachment 2 would be the most optimal option for FOB operation of a mobile manufacturing for AM applications. Any further modifications to the proposed Design Guide of the 3D Filament Extrusion (in Attachment 2) would need to be undertaken during the ‘Demonstration & Validation’ ESTCP Project in consultation with Conair, the likely commercial vendor who would fabricate and build this unique system in 20-foot ISO Containers for FOB applications.

APPENDIX B LIST OF PUBLICATIONS AND PRESENTATIONS

1. Krishnaswamy, P., and others, “*System for recycling PET bottles into plastic lumber for U. S. Army’s forward operating base applications,*” American Chemical Society, 24th Annual Green Chemistry & Engineering Conference, June 2020.
2. Heggs, R, and others, “*Improving the Ductility of rPET Plastic Lumber with Impact Modifiers,*” 25th Annual Green Chemistry & Engineering Conference, June 2021.
3. Krishnaswamy, P. and others, “*Development of an Agile, Novel Expeditionary Battlefield Additive Manufacturing Plant Using Recycled and Reclaimed Thermoplastic Materials,*” Society of Plastics Engineers, Annual Technical Conference, March 2020.
4. Heggs, R. and others, “*Recycling PET into Plastic Lumber at Forward Operating Bases,*” Society of Plastics Engineers, Annual Technical Conference, March 2020.
5. Heggs, R, and others, “*Improving the Ductility of rPET for Plastic Lumber Applications,*” Society of Plastics Engineers, Annual Technical Conference, March 2022.
6. Krishnaswamy, P. and others, “*Feasibility of Using Recycled PET Water Bottle Flake as an Additive Manufacturing Feedstock at Forward Operating Bases,*” ASTM International Conference on Additive Manufacturing, November, 2022.
7. Krishnaswamy, P. “*Development of an Agile, Novel Expeditionary Battlefield Manufacturing Plant Using Recycled and Reclaimed Thermoplastic Materials – Additive Manufacturing,*” SERDP-ESTCP Symposium, December 2019.
8. Krishnaswamy, P. “*Development of an Agile, Novel Expeditionary Battlefield Manufacturing Plant Using Recycled and Reclaimed Thermoplastic Materials – AM of Recycled Polymers (rPET),*” SERDP-ESTCP Symposium, November 2021.
9. Krishnaswamy, P. and others, SERDP Project WP 18-1047 Poster on “*Development of an Agile, Novel Expeditionary Battlefield Manufacturing Plant Using Recycled and Reclaimed Thermoplastic Materials,*” at SERDP-ESTCP Symposium in 2019, 2020, 2021, and 2022.
10. Krishnaswamy, P. and others, “*Additive Manufacturing with Recycled Polyethylene Terephthalate (rPET) Bottle Flakes at Military Bases,*” Society of Plastics Engineers, Annual Technical Conference, March 2023.
11. Krishnaswamy, P. and others, “*Mobile Recycling Systems for Thermoplastics Waste at Army Bases,*” manuscript under preparation for the Journal of Society of Military Engineers (SAME), invited publication, 2023.