3devo

MATERIALS MADE **SIMPLE**.

100% RECYCLED **PET** BOTTLES

Shredding & Extrusion Walkthrough

Our experience shredding and extruding Polyethylene Terephthalate (PET) bottles into 100% recycled filament. The material will be referred to "R-PET" in this report.

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The standard physical and chemical properties of commercial PET are shown in Table 1.

Density	1.41 (gmol^{-1})
Glass tansition temperature	69-115 (°C)
Melting temperature	255-265 (°C)
Water absorption (after 24 h)	0.5 (%)
Application	Liquid containers (Bottles)

1. INTRODUCTION AND CONTEXT OF THIS REPORT

This document guides the reader through the shredding process and extrusion process of **R-PET**, performed in our test lab at 3devo. It describes the experimental process that led to the optimal settings and the best product quality.

The goal of the test was to recycle PET bottles into **1.75mm filament**. Figure 1 is a picture of the original PET bottles. Please note that PET bottles are normally made from co-polymer PET because of its lower crystallinity, improved ductility, better process ability and better clarity. Therefore, this document is only an indication on one type of commercial bottles, different bottles may require different settings.

In order to achieve successful PET recycling, PET flakes should meet certain minimum requirements. Examples of the minimum requirements for the PET flakes are summarized in Table 2. The major factor affecting the suitability of PET flake for recycling is the level and nature of contaminants present in the flakes. Minimizing the amount of the contaminants leads to better R-PET quality. In this study, as we did not process used bottles, they did not need to be pre-washed; there was no stickers to remove.

Property	Value
Tm	>240 C
Water content	<0.02 wt.%
Flake size	0.4 mm < D < 4 mm

Table 2 *- Minimum requirements for PET flakes to be reprocessed*

Chapter 2 describes the shredding step prior to extrusion. Chapters 3 to 6 explain more in detail the main experimental steps of the extrusion test itself, which consisted in a series of adjustments. It is crucial to note that the extrusion test was performed on a **Precision** machine equipped with a **4mm nozzle.** Chapter 7 gives an overall conclusion regarding the recyclability of PET bottles and summarizes the entire report.

Figure 1 *- PET bottles*

2. SHREDDING

The shredding step was performed on the SHR3D IT (shown in Figure 2) equipped with a 4mm particle filter. This operation was relatievly straightforward. The bottles did not have a suitable shape to be fed easily in the shredder. After their caps were removed, the bottles were pressed by hand to be fed in shredder. Manual force-feeding, using the handle of the machine, was applied to speed up the shredding process.

The shredding process quickly gave good results. Figure 3 is a picture of the feeding step, which shows that how bottles were grabbed by the teeth of the shredder.

RESHREDDING R-PET: *This step is extremely easy and straightforward since the first regrind flows fast down the hopper of the shredder. Shredding a batch of material a second time usually only takes a few minutes. A second shredding step can be very beneficial: a more homogeneous regrind will result in a more stable flow during the extrusion step, and subsequently in a better filament quality. Figure 4 represents a difference between once shreded (right) and re-shreded PET bottles (left).*

The first sample of regrind that was obtained after a few moments of shredding. In aspect of flake size, two distinct zones were observed while showing a good homoginity in each parts. Considering that smaller flakes were appropriate for extrusion, they were seprated and in order to increase the homogeneity of the particle size, larger particles were shreded again.

Figure 2 - *Picture of the SHR3D IT (Not captured during this research experiment)*

Figure 3 - *Feeding the SHR3D IT*

Figure 4 - *Size and homogeneity comparison between once shredded (right) and twice shredded PET bottles (left)*

Figure 5 is a picture meant to compare the shape and size of the R-PET regrind to standard Polylactic Acid (PLA) pellets commonly used in extrusion for good quality results. It can be seen that the particle size distribution of the regrind is rather narrow, and that the particle size is similar or inferior to standard PLA pellets, which is a first indication that the plastic will feed and melt easily in the extruder. Figure 6 shows that the whole batch of regrind looks homogeneous and free of significant impurities. Shredding the material several times and/or installing a 3mm filter on the shredder can help the user obtain the desired quality of regrind.

Figure 5 - *Size and shape comparison between R-PET regrind (left) and standard PLA pellets (right)*

Figure 6 - *Close-up view of the regrind*

3. PREPARATION AND PRE-PROCESSING

As PET is a hygroscopic polymer and considering the fact that water reduces the molecular weight, moisture contamination should be below 0.02% (weight). Therefore, drying is regarded as an essential step in PET bottles recycling. Minimizing the moisture content of the flakes reduces the hydrolytic degradation effect and leads to higher R-PET melt strength. The **drying** was performed at **140°C for 4h.**

Before the extrusion test, the machine was purged with the following materials:

- Devoclean MidTemp to clean the barrel thoroughly
- HDPE to transition more easily to R-PET
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This purging/transitioning process was performed at 270˚C (all four heaters).

PET flakes were then introduced at 270˚C.

WARNING: *When experimenting with a new grade of plastic, it is of the utmost importance to introduce said plastic at temperatures high enough to ensure sufficient melting and to avoid the clogging of the machine.*

Figure 7 is a picture of the feeding, Figure 8 illustrates the transition from HDPE to R-PET. The transition only took a few moments, it was sudden and clearly visible.

Figure 7 - *Feeding R-PET regrind into the hopper of the extruder*

Figure 8 - *Transition from purging HDPE (white extrudate at the bottom) to R-PET (gray extrudate at the top)*

4. EXTRUSION (1): STARTING POINT AND FIRST OBSERVATIONS

The following settings were used as a starting point during the extrusion test:

Parameter	H4	H3	H2	H1	Screw speed	Fan speed
Set value	270 °C	270 °C	270 °C	270 °C	5.0 RPM	50%

WHY 270°C? As mentioned earlier, it is wiser to start at a temperature that is too high, to avoid the clogging of the machine. For most grades of PET, 270°C is on the high end of the thermal window of operation (255-270°C).

WHY 5.0RPM AND 50% FAN SPEED? These values are very often appropriate values to start experimenting with a new material. In order to extrude stable filament of 1.75mm thickness, the best rotation speed is usually found between 3.0 and 7.0 RPM, which is why the starting value of 5.0RPM is always a good start. As far as the fan speed is concerned, it is harder to define an ideal percentage that works by default, because this parameter can vary a lot; it is good to start with a medium value and be ready to make quick adjustments.

THE FIRST RESULTS: The transition from HDPE was smooth and fast, the flow did not seem to be much disturbed. The flow appeared to be rather stable but the extrudate was too liquid and the extruded PET was swirling a lot. There are several possible explanations, while it might be due to high temperature and high fan speed, the low melt strength of R-PET flakes is the main problem. It was hard to pull it properly because it was wobbly and flattened by the puller. In addition, the extrudate contained bubbles as if air had been trapped inside the melt.

RAT HOLING: *Another phenomenon was observed, this time affecting the feeding. This phenomenon is known as rat holing: it generally affects powders and regrinds, it occurs when the formulation forms cohesive structures in the hopper, which do not flow properly down in the throat and therefore diminish the flow. This can clearly be seen in Figure 9. Carefully using a stick every few minutes to break the structure, or a continuously stirring device, can improve the feeding.*

Figure 9 - *Rat holing: the material forms a cohesive structure against the walls of the hopper, making the feeding inconsistent*

5. EXTRUSION (2): ADJUSTMENT STEPS

The objective was to find the optimal extrusion settings with the help of the filament sensor. The main issue was that the extrudate was too hot, resulting in the flattening of the filament by the puller wheels. Figure 10 is a picture taken during the adjustment phase, before the filament was of sufficient quality to be spooled.

FILAMENT FAN SPEED: Because the extrudate was swirling a lot and was too solid when reaching the puller, the correct approach was to decrease the fan cooling percentage. Several options were tested **between 0% and 50%.**

SCREW RPM: In order to achieve a steady flow and maintain just enough pressure inside the barrel, the solution was to decrease the screw speed slightly, to **3.6 RPM.** This way, the rotation speed of the screw was fluctuating less and the extrudate was given additional time to be cooled down by itself rather than using high fan speeds.

TEMPERATURES: All temperatures were decreased gradually down to 265°C, point at which the analysis showed an increase in the RPM fluctuation which suggested that the material was not being melted easily enough. It was found that a “low-high-low bell” curve was the optimal temperature profile: the material seemed to melt neither too soon nor too late inside the barrel, this is the optimal situation to obtain a stable flow.

THE IMPORTANCE OF DRYING: *It is known that R-PET are troublesome to process when they have not been dried prior to the extrusion. The experiment confirmed that moisture can have a very negative effect on the extrusion process and the quality of the filament (see Chapter 6 for illustrations). Drying the material before extruding it results in a smoother surface. It is also important to know that moisture can also cause the presence of “void” inside the filament : moisture trapped in the material forms bubbles at the core of the filament, the shrinkage is then uneven, the surface becomes rough, and the filament cannot be printed since matter is “missing” inside it.*

Figure 10 - *Filament flowing freely during the adjustment phase*

6. EXTRUSION (3): SPOOLING

The following settings were used as a starting point during the extrusion test:

Parameter	H4	H3	H2	H1	Screw speed	Fan speed
Set value	265 °C	267 °C	267 °C	265 °C	3.6 RPM	15%

A spool was successfully manufactured using these settings. Figure 11 is the graphical representation of the datalog which corresponds to the produced spool. The spooling process happened between 5800 (s) to 8600 (S). It shows that the filament thickness was not very stable during the entire spooling process, but almost well kept within the usual industry tolerance (1.75±0.05mm). Figure 12 is a picture of a spool which were obtained at 3devo.

Finally, to prove the printing capability of this spool, we printed a small bottle using produced R-PET filament. The 3D printed bottle is shown in Figure 13.

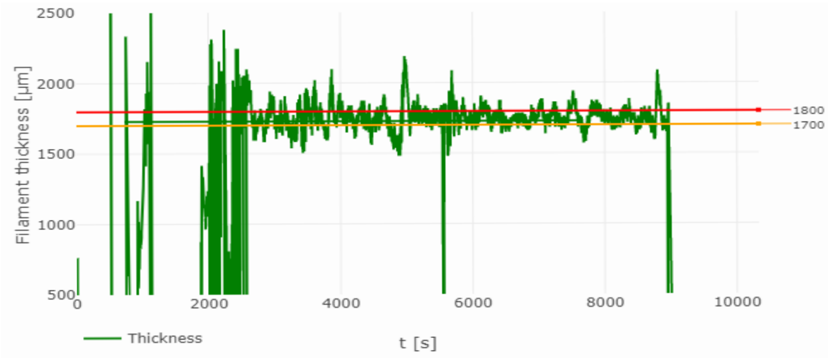

***Figure 11** - Datalg: filament thickness (set value: 1.75mm, red line: 1.80mm, yellow line: 1.70mm)*

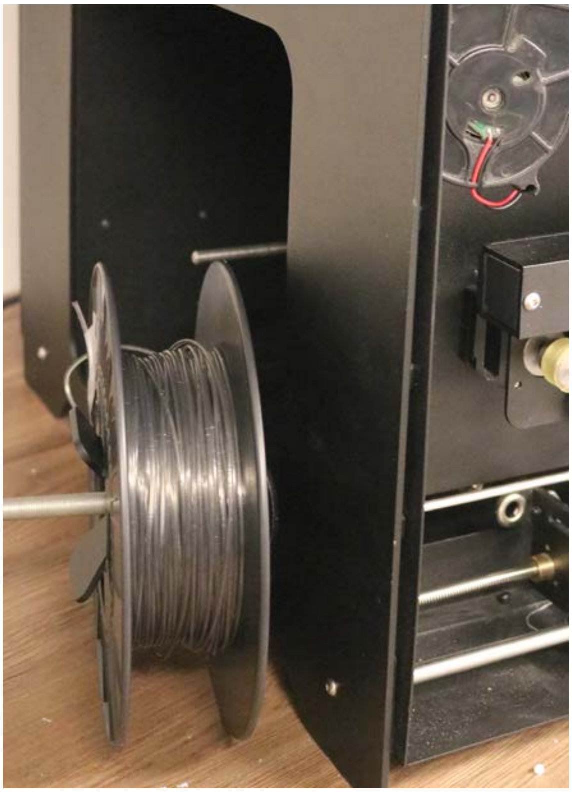

***Figure 12** - Spool of R-PET*

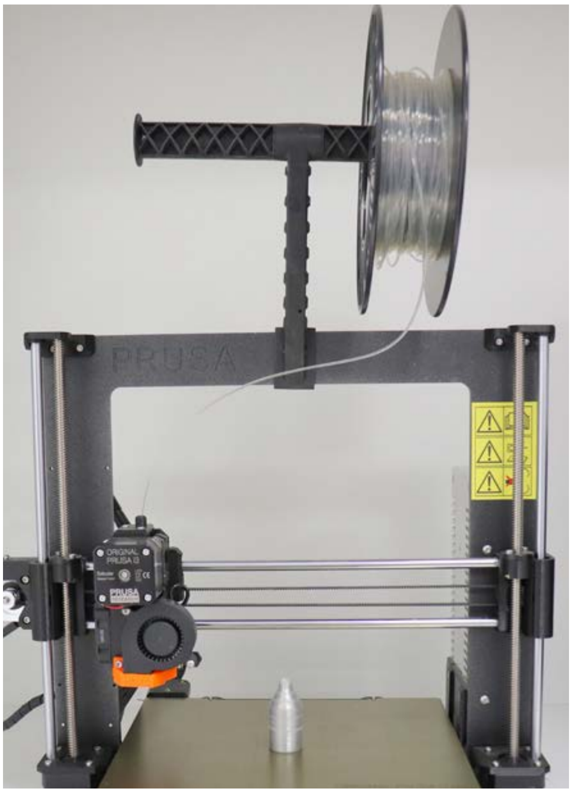

***Figure 13** - 3D printed bottle using R-PET filament*

Figure 14 - *Final 3D print with 100% PET filament*

7. CONCLUSION AND RECOMMENDATIONS

This extrusion experiment was extremely positive. Indeed, 1.75mm filament of quite acceptable quality was obtained using a Precision equipped with a 4mm nozzle. Although, it was shown that 3d printing with some high quality parts of the filament is possible, it should be noticed that the material cannot be shredded and extruded very easily, and that the resulting filament's thickness can still be improved to meet the industry standard (+/- 50 microns). The fact that the material could be processed at relatively low temperatures made the launching of the process a fast operation.

REPORT SUMMARY:

TO DOs:

- Keep temperatures around 165°C for optimal quality
- Dry the material at 140°C for at least 4h in a dryer or an oven
- Purge thoroughly after extrusion using Devoclean MidTemp (at the processing temperatures)
- Transition back to R-PET with HDPE first. This is not mandatory but will save some R-PET

WARNINGS:

- Use the shredder and the extruder with great care: blades and heat are involved
- Watch out for impurities (dust mostly) during shredding and storage, impurities will lower the quality of the final product
- It might be necessary to adjust the fan speed depending on the room conditions
- Rat holing must be taken care of regularly by stirring in the hopper manually or with the help of a continuously stirring device

Parameter	H4	H3	H2	H1	Screw speed	Fan speed
Set value	265 °C	267 °C	267 °C	265 °C	3.6 RPM	15%