TCEP-P-06.- Melt filtration, Pelletizing and Crystallization.

Background

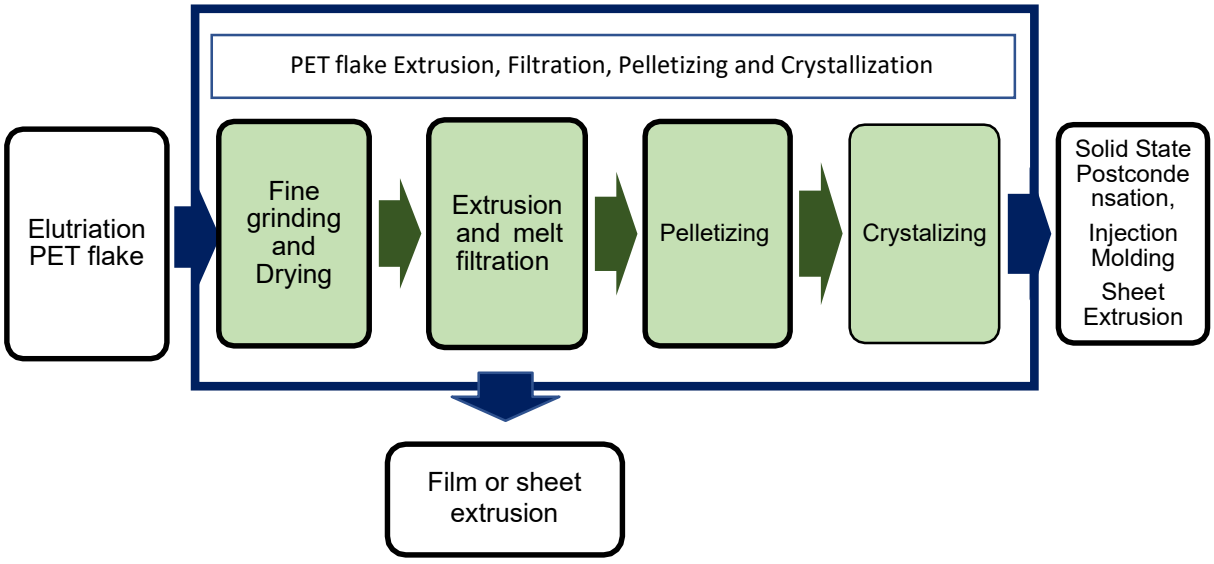
The final destination of the PET flakes is to be extruded into a new plastic specimen. When the PET flake is melted in the extruded, it needs to be filtered to remove some impurities. Then, the molted polymer can be pelletized or extruded into a sheet, fiber or other object.

This document represents the different steps of extrusion. As the different process of extrusion will be treated in other procedures, in this case it will be treated the procedure for filtration, palletization and Crystallization.

This document describes a standard practice to extrude and create strand cut PET pellets from blended PET flakes. The procedure includes the steps of finer grinding of flake, drying, extrusion, pellet forming and crystallization. At the same time, it defines required observations and samples for further testing.

Practice summary and illustration

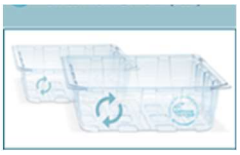
Elutriated flake is blended as required for a specific test method or Critical Guidance Protocol, and then dried in a desiccant drier. Dried flake is melted and filtered in an extruder to create strand cut pellets. Melt pressure is recorded ahead of the melt filter. Amorphous pellets can be crystalized.



Note: Melt filtration and pressure measurement is not necessary when the practice is used only to add a heat history.

Equipment required

- Dryer system:
  - Vacuum dryer
  - Temperature 160ºC
    - Capable to achieve a moisture contain <50 ppm
  - Desiccant air dryer should have a dew point below - 20ºC
- Extruder suitable for PET flake processing in the laboratory.
  - A 25 to 35 mm extruder with a 24:1 to 36:1 L/D is suitable for laboratory use.



- A twin- screw extruder may also be used
- Melt residence time in the extruder should be no more than 6 minutes.
- Requires a means for maintaining dried PET flake in a dry state while in the hopper and during extrusion.
- Equipped with a breaker plate and screen pack.
- Capable of supplying 0.38 kg/cm^{2}·hr through the screen pack.
- Employs a calibrated pressure transducer ahead of the screen and employs a data logger to record pressure values.

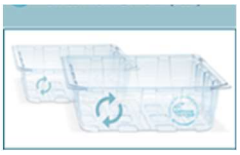
- A Strand die, water bath and pelletizer.
- Scale for measuring extruder output.

Materials required

- Containers jars or bags to hold final pellet samples
- Screen pack for the extruder - 40/250/40 mesh
- Elutriated control flake, elutriated test flake, and/or blends of flake.

Practice steps

1. Finer Grinding (optional). – Depending on the type and size of the extruder, particularly in the caseof a lab extruder, the regular flake size is too big to be used. In that case, finer flake will be required. In that case, each sample will be dry ground separately using a standard mechanical grinder, using a screen size adapted to the type of extruder. It is important cleaning the grinder every time is used to avoid cross contaminations.
2. Drying Step
    a) Each sample will be dried directly before extrusion to a moisture of less than 50 ppm in a hopper using dry hot air from a desiccant unit.
        1. The air temperature must remain between 150 and 160°C.
        2. Typical drying time at 160°C is 4-6 hours. The same conditions are used for all samples.
    b) Alternatively, a vacuum dryer can be used,
        1. Temperature between 150 and 160°C
        2. Time as necessary to achieve less than 50 ppm moisture in the PET flake
    c) Make provision to ensure the flake are kept dry during extrusion.
    d) Inspect the drying hopper after emptying for sticking flake or contamination on the sides of the hopper. Inspect the hot PET flake for the presence of any unusual fumes or odors associated with the hot flake.
    e) Record any occurrence of sticking flake, residues, fumes or odor that occurred drying the resin drying step.
3. Extrusion Steps for pelletizing (Extrusion into Sheet will be done as per TCEP-P-08)
    a) Extrude the control article flakes first:
        1. Target melt temperature of 270-280° C.
        2. The melt will be filtered with a clean 40/250/40 mesh melt filter (about 60 microns filtration)
        3. Flow rate through the filter should be at least 0.38 kg/cm^{2}·hr of mesh area.
    b) The melt is extruded through a die into strands of approx. 2.5mm diameter.
        1. The strands are rapidly cooled in water
        2. Fed into a pelletizer to form amorphous pellets.
        3. It is important to achieve similar pellet sizes for each of the test and control materials for use in solid state IV build evaluations.



4. Pellet bulk density aprox 800 Kg/m3?
5. Screening may be used to remove over and under sized pellets.

c) Start run timer

a) Record pressure values ahead of the screen pack for a 30 minute's run time.
b) Record any occurrence of unusual fumes, odor or build-up occurring at the feed throat or die exit of the extruder.
c) Observe for any hazards associated with the innovation material.
d) Save a sample of extruded pellets for IV measurement.
e) Additional samples can be retained for color and other measurements as desired.
f) Retain a 50-100-gram sample from this practice step.
g) Purge the extruder and change the screen pack between each run.
h) Store the pelletized samples in pails or bags
i) Extrude all subsequent samples at the same conditions; achieve pellets of similar size for each sample set when the pellets will be used for a solid stating evaluation

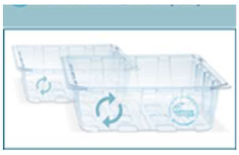
4. Pellet Crystallization. - This process is required if an injection molding test have to be conducted. The pellets need to be crystalized before been dried to avoid lumps in the dryer. In the case of solid stating, Crystallization should be considered as an option prior to SSP. Crystallization involves following steps:
   a) Each sample is homogeneously crystallized to obtain single, non-sticking PET pellets.
   b) Typical crystallization conditions are 1 hour in a pre-heated oven at 160°C.
   c) Alternatively, crystallization can be performed in a fluid bed crystallizer:
      1. 20 minutes
      2. Heated air at 175°C. (pellet temperature must be >140°C for >10 minutes but remain <170°C).
      3. The same conditions are used for all samples.
   d) After cooling break up any agglomerates to obtain single pellets.
   e) Store crystallized pellets in a sealed container or under dry conditions to maintain their moisture content <2500ppm.
   f) Record any occurrence of unusual sticking during crystallization.
   g) Retain a 50-100-gram sample from this practice step.

Steps Required to Assess Pressure Values

For the test material only:

1. Take a measure of the pressure every minute
   a) Calculate the average pressure during the 5 minutes of the extrusion (Pi)
   b) Calculate the Average pressure during the last 5 minutes of extrusion (Pf)
2. Calculate (Pf-Pi)/Pi for every sample
   a) Make a table with the calculation for every sample.
   b) If > 0,25 is an evidence of Screen pack Build Up.

DOCUMENT REVISION HISTORY



Version	Publication Date	Revision notes
V0	Sept - 21	NEW DOCUMENT
V1	April-23	Platform, document name and logo changes.