

TCEP-P-07.- Solid State Polymerization of PET Pellets.

Background

The extrusion of PET produces a drop in the Viscosity of the product. As the level of IV is important the extrusion and injection process, as it affects the final manufacturing production performance. For that reason, a molecular weight IV built up process is a common practice after the flake and pellet. This is conducted by a Solid-State Polymerization (SSP) process.

This document presents a standard practice to raise IV of PET through exposure of pellets to heat and vacuum employing a rotary vacuum unit (this is a unit widely used in commercial practice). A laboratory scale rotary vacuum unit is commonly used to create quantity of 0.80 dl/g IV. A similar rotary equipment with continuous pure nitrogen flow is also appropriate and accepted.

Control and test blend flake have to be submitted to this process to understand the effect of the innovations on the final performance during the recycling process.

The rotary vacuum can be used to create the solid stating build rate samples, but other commonly used methods for solid stating PET may be used as well, as similar equipment where a continuous pure nitrogen flow and atmosphere is used.

Practice Summary

Pellets are processed in batches through a rotary vacuum SS reactor with controlled time, temperature and vacuum level (or N₂ flow). PET pellet size, initial moisture level and process conditions will influence final results.

It is recommended to conduct the Solid Stating process crystallized PET to prevent lump or fusion of amorphous pellets. Some laboratories choose to crystallize in a separate step prior to solid stating. Others may choose to solid state within the rotary vacuum unit. Either approach is acceptable.

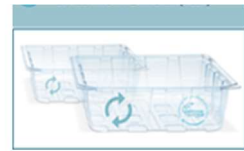
This Procedure anticipates crystallization in the rotary reactor.

Equipment Required

- Vacuum Sealed or continuous pure Nitrogen, Stainless Steel or glass Vessel,
 - heated with control temperature
 - PET sample temperature target is 210°C
- Vacuum pump or nitrogen flow
- Sample port capable of pellet sample taking without breaking vacuum

Materials and Reagent Required

- Dried glass jars and metal lids for use in sampling the solid stated resin throughout the test run
- Amorphous pellets from the control article
- Amorphous pellets made with blends of control resin with the test article.



Practice Steps

Solid stating process steps

1. Prepare the amorphous pellet blends required/desired for testing:
 - a) Screen the pellets to remove under and over-sized pellets that can impact results.
 - b) Condition pellet samples in a standard environment so that the starting moisture content of pellets is similar from one evaluation to the next.
 - c) Amorphous pellets may be crystallized prior to use or may be crystallized in the solid-state reactor as desired by the evaluator.
2. Add a given individual pellet sample to the rotary vessel at room temperature. Ensure the total weight is sufficient for the next test process (injection, extrusion,). If pellets have been previously crystallized, step 2 can be skipped. **Crystallization process:**
 - a) Place the sample in the vessel
 - b) Start rotation to avoid PET pellets from sticking and clumping
 - c) Start vacuum or nitrogen flow.
 - d) Raise temperature in a given ramp until pellet reaches 160-170°C
 - e) Pellets must be at 160-170°C for 1 hour prior to raising temperature for SSP processing
3. Raise the vessel temperature to have the PET temperature up to 210 ± 5 °C. Keep temperature constant.
4. Maintain vacuum or nitrogen flow and rotation during the whole process.
 - a) SSP time starts when article temperature reaches 190°C
 - b) Run the solid-state reactor for the desired/required time increment(s).
5. When reaction time is reached, stop heating.
6. Do not stop vacuum/nitrogen flow and rotation during the cooling process.
7. When PET temperature goes below 140°C, stop agitation and vacuum/nitrogen
8. Retain 50-100gram sample for every test specimen for further evaluations.
9. Verify any unusual sticking behavior or unusual build-up within SSP vessel.
10. The reactor is typically fitted with a sample port so that the PET pellets can be sampled from time to time to monitor IV values across the process to generate the IV build up curve.

DOCUMENT REVISION HISTORY

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