

PET Flake Clumping Evaluation

Document Code – PET-S-08

Publication or Revision Date: November 16, 2018

Introduction – Scope, Significance and Use

It is becoming more common to solid state polymerize washed PET flake prior to extrusion rather than the more traditional method of solid stating pelletized PET. In solid stating, PET flake can be exposed to heat over 190° C up to 220° C, as well as pressure in a loaded hopper. It is important that flakes of PET do not stick together to form hard clumps when exposed to high temperatures and loading in a hopper. Clumps of PET can cause bridging in feed systems that prevent material flow.

This test method can be used in the laboratory to evaluate whether there is low melting polymer or other source of contamination in washed and elutriated PET flake that can create clumps of PET flake under heated conditions.

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Test Method Summary

Washed and elutriated flake is heated in an oven at 210° C under two different conditions - one under low pressure and another with weight applied to simulate the burden in a storage hopper. Any formation of clumps of PET flake that will not pass through a screen with 12.5 mm openings is evaluated.

Equipment Required

- Weigh scale (0.01 ± grams)
- Laboratory oven that can control temperature at 210°C

Materials Required

- Baking pan with dimensions of 23x33 cm
- Baking pan with a surface area within the range of 85 to 100 cm² and depth of at least 5 cm
- Aluminum foil



- Screen or sieve with no more than a 12.5 mm opening
- Containers to transfer materials
- 9 kg of weight
- Washed and elutriated PET flake sample

Test Method Steps

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The following procedure is given for illustration. Variations in container sizes and weighting that create the same pressure can be used. Background to support the intent of the lab test:

- PET flake is exposed to approximately 210° C in commercial practice when flake is solid stated.
- The bulk density of PET flake is reported to be 300 kg/m³.
- In a hopper containing flake 3 meters in height, there is 900 kg of weight per m², or 900 kg per 10,000 cm², or 90 g/cm² of area.
- So, in the lab, assuming a 100 cm² container filled with PET flake, with an applied weight of 9 kg spread over the flake approximates the burden in the hopper.

Test steps are:

Crystallization

A crystallization step is only necessary if the starting sample is largely amorphous; such as when sheet or extrusion blow molded articles are being evaluated. Injection stretch blow molded bottles do not require this step.

1. Expose about 1.5 kg of washed and elutriated flake in an oven at 165° C for 30 minutes using the 22x33 cm baking dish to hold the sample.
2. Remove the dish from the oven and allow to cool to room temperature.
3. Any flakes that stick together at this point that can be broken apart using light hand pressure, can be separated.
4. This crystallized material is used in the evaluations below.

For low pressure evaluation:

1. Pre-heat the oven to 210° C.
2. Put 1 kg of washed, elutriated and crystallized PET flake into a 22x33 cm baking dish that is lined with aluminum foil.
3. Heat the flake for 90 minutes in the oven.
4. After 90 minutes, remove the baking pan from the oven and allow the flake to cool undisturbed to room temperature.
5. Transfer the flake contents in the baking dish to the 12.5 mm screen.
6. Shake the sieve by hand to cause single flakes to fall through. Any single flakes that are oversized and unable to pass through the sieve may be removed by hand and placed with flakes that passed through. Any agglomerated flake that breaks up during this sieving is not deemed to be a problem.
7. Weigh the agglomerates that cannot pass through the sieve and record the weight.
8. Any flake or residue that melted and stuck to the aluminum foil is weighed separately and recorded.

For evaluation under load:

1. Line the 100 cm² baking dish with aluminum foil and tare this container
2. Completely fill the lined dish with a fresh sample of PET flakes up to the top; weigh the filled container and record the weight of the flake in the container.
3. Place the 9 kg weight upon the flakes so that the weight is uniformly distributed over the flakes and not touching the side wall of the container. Then place the dish, flakes, and weight in the oven. (During the test the container wall and the weight should not come in contact or the weight support needs to be adjusted.)
4. After 90 minutes, remove the sample from the oven and allow the baking dish and contents to cool to room temperature without disturbing.
5. Transfer the flake contents in the baking dish to the 12.5 mm screen.
6. Shake the sieve by hand to cause single flakes to fall through. Any single flakes that are oversized and unable to pass through the sieve may be removed by hand and placed with flakes that passed through. Any agglomerated flake that breaks up during this sieving is not deemed to be a problem.
7. Weigh the agglomerates that cannot pass through the sieve and record the weight.
8. Any flake or residue that melted and stuck to the aluminum foil is weighed separately and recorded.

Test Assessment

The expected result for clean PET flake is that 100% of the PET flake will pass through the 12 mm screen in both evaluations.

APR Guidance is that a result creating less than 1% weight loss combined from clumps that do not pass the 12 mm screen and loss adhered to the aluminum foil in each evaluation is preferred.



DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	