PET Flake Clumping Evaluation

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Introduction – Scope, Significance and Use

This laboratory test method can be used to evaluate whether there is low melting polymer or other components in washed and elutriated PET flake that can create clumps of PET flake under heated conditions. It is important that flakes of PET do not stick together to form hard clumps when exposed to high temperatures in driers and crystallization units. Clumps of PET can cause bridging in feed systems that prevents material flow.

There is an appendix to this test method that presents data supporting the test conditions employed.

Test Method Summary

A sample of washed and elutriated PET flake is heated in an air oven at 195° C. After cooling to room temperature, the sample is passed through a screen. Any clumps of PET flakes retained on the screen are weighed and reported as a weight percent of the sample.

Equipment Required

- Weigh scale (0.01 ± grams)
- Laboratory oven that can control temperature at 195°C

Materials Required

- Baking pan with dimensions of 23x33 cm
- Aluminum foil
- Screen or sieve with no more than a 12.5 mm opening
- Containers to transfer materials
- At least 1 kg sample of washed and elutriated PET flake

Test Method Steps

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Test steps are:

Crystallization

A crystallization step is optional but may be desirable when 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 generally benefit from this step.

1. Expose 1 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 evaluation below.

Clumping evaluation

1. Pre-heat the oven to 195° 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. Further, it is permissible to drop the baking dish pan five times from a height of 6 inches to break up soft clumps that are not the focus of the measurement.

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.

APR Guidance is that a result creating 1.0%, or less, weight loss combined from clumps that do not pass the 12.5 mm screen and loss adhered to the aluminum foil after the evaluation is preferred.

Weight loss from clumping greater than 1% leads to a result that is categorized as detrimental.

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	
2	November 1, 2022	Reduced test temperature, eliminate weighted test, added appendix

Appendix

An earlier version of this test method published in 2018 called for testing at 210° C and included an evaluation where the PET flake was weighted to simulate the static loading that PET might experience in a large hopper drier. After gaining experience with this test, users suggested that:

a. The test temperature was higher than necessary to simulate the temperature that flake is exposed to in crystallization units and driers. Recycled PET flake can be exposed to 210° C in a solid stating unit, but in all known cases, flake is agitated during solid stating to prevent agglomeration, even of natural PET by itself. Further, users expressed concern that a 210° C test temperature can disqualify material that would be desirable to retain in the PET recycling stream.
b. Experience showed that the level of clumping was about the same whether the sample was weighted with a static load, or simply contained in a shallow baking dish with no additional weight loading. And so, there was interest in determining whether the test could be simplified by eliminating the evaluation of clumping under load.

Plastics Forming Enterprises conducted an evaluation of the impact of temperature and loading on PET flake samples containing PETG in one case, crystallizing co-polyester film in a second, and PET thermoform samples in a third, and presented these results in a public report: PFE Laboratory Report #1974 PET Clumping Case Study. The illustrative results shown in the table below are taken from this report and presented with permission of PFE. In brief, results show that:

- Testing at 195° C is very effective in differentiating between PETG and co-polyesters that crystallize.
- Applying a static load to flake samples does not add value to the test procedure.
- Elutriation does remove label film content that can contribute to clumping and so it is desirable to include elutriation in preparation of PET flake employed in the clump test procedure.

Variable	Oven Temp °C	Load, lbs	Clump wt%
Control PET flake	195	20	0.00
		0	0.00
	210	20	0.00
		0	0.00
PETG label film	195	20	26.05
		0	12.75
	210	20	21.22
		0	19.24
co-PET label film MP = 212° C	195	20	0.00
		0	0.00
	210	20	0.09
		0	0.00
co-PET label film MP = 201° C	195	20	0.30
		0	0.45
	210	20	3.93
		0	3.78