

Plastics Forming Enterprises LLC



Laboratory Report #1974 PET Clumping Case Study

Applicant:

PFE

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Plastics Forming Enterprises, LLC

Plastics Consulting, Research and Engineering

Objectives:

- To evaluate the current APR Clumping Procedure.
 - Examine how different label technologies and thermoforms perform under different loads and temperatures
 - Examine how elutriation plays a role in clump percentage.

Materials:

- Control – Blend of PET 1st Heat Bottles
- Test 1 – PETG Sample with Melting Temperature of ~162°C (blended into bottles at 3% by weight)
- Test 2 – CPET Sample with Melting Temperature of ~212°C (blended into bottles at 3% by weight)
- Test 3 – CPET Sample with Melting Temperature of ~201°C (blended into bottles at 3% by weight)
- Test 4 – Thermoforms
 - Tested at 50/50 with Control Bottles
 - Tested at 100%

Equipment:

- Recycle System
 - Grinder with 3/8” Screen
 - Pilot Wash System
 - Kice Elutriator
- Test Equipment
 - Convection Oven

Case Study Purpose:

Plastics Forming Enterprises (PFE) conducted a case study for the PET clump protocol PET-S-08 to suggest to the APR PET Technical Committee to formulate an update the protocol procedure. The protocol currently states that during the clump process, the lab must preheat the oven to 210°C for clumping with ground, washed, and elutriated flake.

It was discussed to review the PET reclaimers actual processing conditions and temperatures and get industry guidance and feedback. During the review it was determined to evaluate, with data confirmation, whether the current protocol or reclaimers feedback temperature should be changed to a preheated temperature of 195°C. When the design of experiment (DOE) was created, non-elutriated and elutriated flake material was discussed as a potential additional variable as well as weighted samples.

To run this experiment there were 4 different types of material identified, each with different melting temperatures and properties. The materials were:

- Control (PET Flake)
- PETG (Test 1)- Melt Temperature 162°C
- CPET (Test 2)- Melt Temperature 212°C
- CPET (Test 3)- Melt Temperature 201°C
- Thermoform (Test 4)- Melt Temperature 246°C

These materials were selected because they are all distinctive and it was ideal to run the experiment on industry accepted materials in comparison to PETG. PETG is identified by the APR Design Guidelines as NOT preferred in this stream. The general PET guidelines were followed for the wash and elutriation process for the control and all 4 test variables. Testing was processed in various ways for the clump DOE case study procedure.

The elutriated material for control and test variables 1-3 were processed under these conditions:

- Preheated the oven at 195°C
 - No load applied
 - Applied 20 lbs.
- Preheated the oven at 210°C (1 hour crystalize)
 - No load applied
 - Applied 20 lbs.

The non-elutriated material for control and test variables 1-4 were processed under these conditions:

- Preheated the oven at 195°C
 - No load applied
 - Applied 20 lbs.
- Preheated the oven at 210°C (1 hour crystalize)
 - No load applied
 - Applied 20 lbs.

Upon investigation, PFE has concluded that it is possible to operate with a lower preheated oven temperature under certain conditions and extrapolate what could be seen at a higher temperature for industry accepting conditions. Not all the test variables met the current conditions of the protocol but as expected, PETG was effectively outside the current range and the suggested options moving forward. Regarding the difference between the 195°C and the 210°C preheating temperature, research provided confidence that the 195°C oven temperature is more comparable to industrial conditions than the 210°C oven temperature, per industry survey performed by the APR.

The industry has accepted the two tested CPET sinking labels, with full ink bleed, that are compatible with the PET materials and non-issue regarding temperature seen to be a low melt.

The study and data collected supports this protocol procedure change. This proposed change will continue the intent to increase the amount of PET being collected and processed in a commercial scale plastics recycling environment.

PFE supports the plans to make change to the APR PET-S-08 based on APR leadership to evaluate the recyclers conditions, DOE for the Case Study, and data per the case study funded and performed by PFE. The last step is to share the full study with the PET Technical Committee and vote to support the proposed change, or to continue operating with the current protocol procedure.

PFE suggests that the APR change testing conditions allowing for the testing of both elutriated and not elutriated materials unloaded at 195°C.

***When the material is elutriated, it is recommended the clump percentage is no more than 1%.
When the material is not elutriated, it is recommended the clump percentage is no more than 3%.***

Non-Elutriated Clumping Summary

Non-Elutriated			
Variable	Temperature (°C)	Load (lbs)	Clumping (%)
Control	195	20	0.00%
		0	0.00%
	210	20	0.00%
		0	0.00%
Test 1	195	20	32.67%
		0	34.45%
	210	20	37.71%
		0	27.36%
Test 2	195	20	0.00%
		0	0.00%
	210	20	1.10%
		0	0.23%
Test 3	195	20	2.91%
		0	1.65%
	210	20	7.22%
		0	28.53%
Test 4 (50%)	195	20	0.00%
		0	0.29%
	210	20	0.00%
		0	0.00%
Test 4 (100%)	195	20	0.13%
		0	0.00%
	210	20	1.18%
		0	0.00%

**Graphical representation in Appendix*

Elutriated Clumping Summary

Elutriated			
Variable	Temperature (°C)	Load (lbs)	Clumping (%)
Test 1	195	20	26.05%
		0	12.75%
	210	20	21.22%
		0	19.24%
Test 2	195	20	0.00%
		0	0.00%
	210	20	0.09%
		0	0.00%
Test 3	195	20	0.30%
		0	0.45%
	210	20	3.93%
		0	3.78%

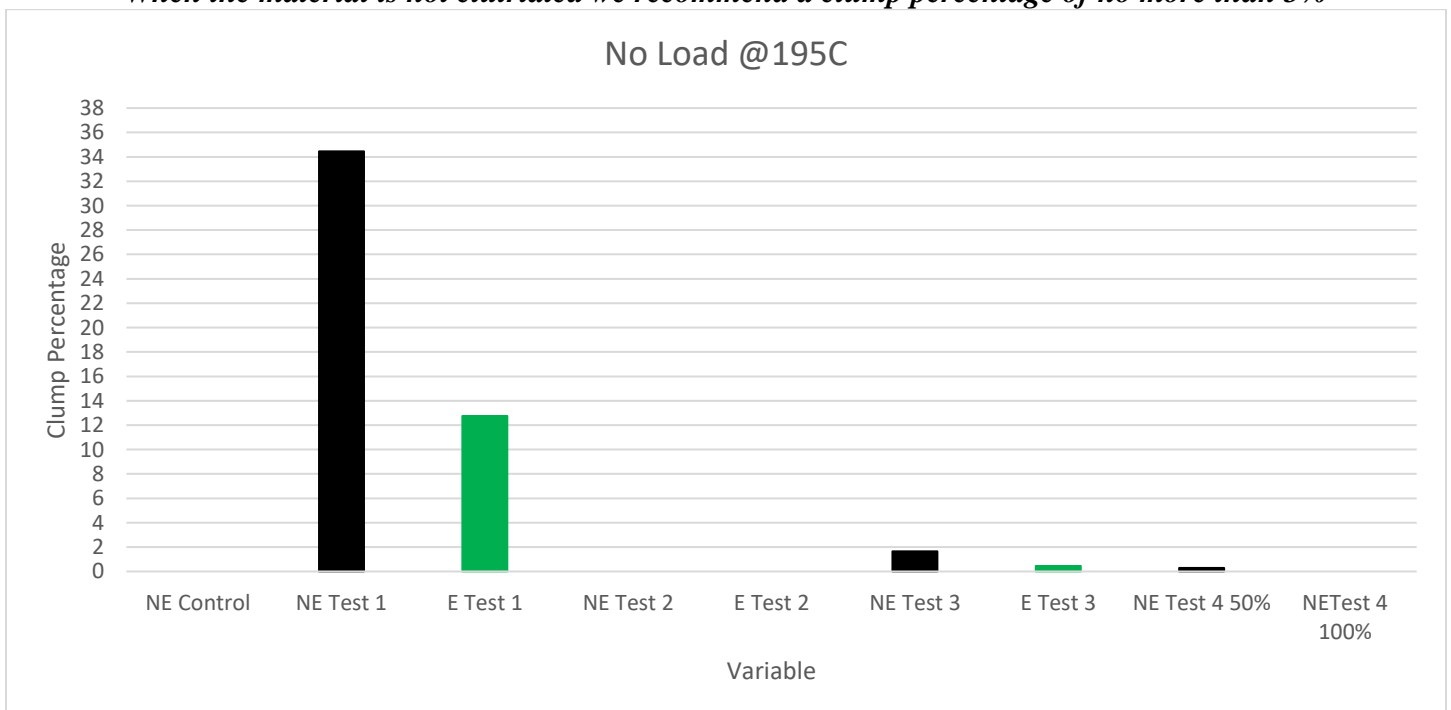
**Graphical representation in Appendix*

Conclusion

1. Control experienced no clumping under all testing conditions.
2. Test 1 did not meet any APR guidelines at any testing conditions.
 - With a melting temperature below 195°C this was expected.
3. Both Test 2 and Test 4 passed APR guidelines for all testing conditions besides the worst-case scenario (non-elutriated flake at 210°C with 20lb load).
4. Test 3 only passed the APR Guidelines when elutriated and tested at 195°C.
 - This was expected as the DSC indicated an onset melting temperature far below 195°C.
5. Elutriation and Oven Temperature showed statistically significant effects on the outcome of the clumping results.
6. Load vs. No Load showed varying effects on the clumping results and differed from material to material.

PFE suggests that the APR change testing conditions allowing for the testing of both elutriated and not elutriated materials unloaded at 195°C.

***When the material is elutriated we recommend a clump percentage of no more than 1%.
When the material is not elutriated we recommend a clump percentage of no more than 3%***





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Incoming Label Differential Scanning Calorimetry (DSC)

References:

ASTM D3418

Test Summary:

Evaluates the temperatures and enthalpies of fusion and crystallization of polymers

Procedure:

Polyolefin material is cut down to a size between 1-5 mg. The polyolefin piece is then prepped in an aluminum pan/lid container and crimped to allow complete contact between the polyolefin piece and the aluminum. The container now holding the prepped polyolefin is inserted into the DSC. A test method is chosen dependent on the customers proposal.

For ASTM HDPE-CG-01/ASTM D3418,

The DSC heated to ~30°C above the suspected temperature of fusion (melt temperature) at 10°C/min and then cooled to room temperature.

This method leaves a trace that can be assessed allowing the determination of temperatures and enthalpies of fusion and crystallization of the polymer. From these values percent crystallinity can be calculated. The traces can also allow for identification of any contaminants/barrier polymers.

Data Table:

Variable	Crystallization Temperature (°C)	Onset Melt Temperature (°C)	Melt Temperature (°C)	Primary Peak J/g
Test 1	N/A	141.92	162.59	1.644
Test 2	102.67	194.17	212.52	12.26
Test 3	113.00	181.52	201.20	10.74

APR Granulation

Test Summary:

Granulate incoming articles to the required grind size





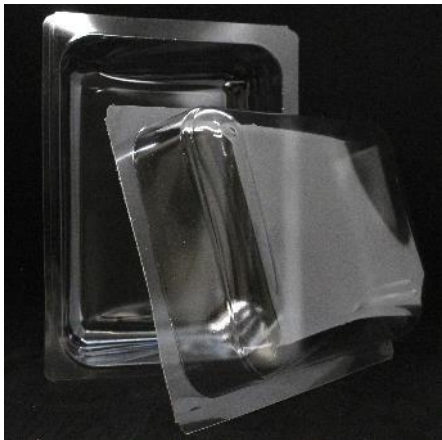

Procedure:

Granulator is cleaned of any contaminating flake from previous runs. Articles are then manually fed into a grinder with screen holes ranging from 9.5-12 mm in diameter. Ground flake is then retained and packaged for washing. The granulator is cleaned between each variable.

Grind Process:

Variable	Incoming Article	Ground Article
Control		
Test 1		

Grind Process:

Variable	Incoming Article	Ground Article
Test 2		
Test 3		
Test 4		

APR PET Wash and Sink/Float Evaluation

References:

PET-P-04

Test Summary:

Remove any contamination from PET and help in the liberation of labels, adhesives, coatings, and layers.

Procedure:

WASH:

Hot wash solution is prepared in a stainless-steel square vessel. Tap water, 1% Caustic, and 0.3% Triton X-100 or McDermid RP24 are all added to the solution. The wash is heated to 85°C and mixed with an impeller for 15 minutes at least 600 rpm. After 15 minutes, the impeller is stopped, and the mixture is removed from the heat source. The mixture sits for five minutes and floatables are skimmed off and saved. 1000mL of wash water is then retained.

RINSE:

The remaining washed PET flake sample is added to water at 45°C. The rinse is then mixed with an impeller for five minutes at least 600 rpm. After the five minutes, the container is removed from heat and allowed to sit for five minutes. Floatables are then skimmed off and saved and 1000mL of the rinse water is retained.



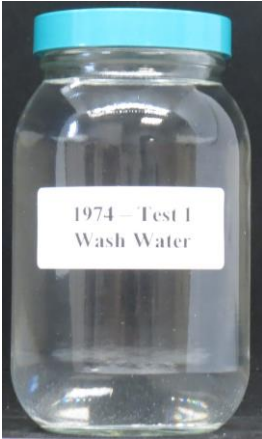

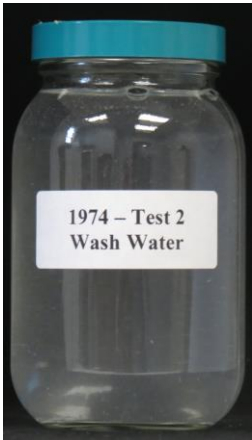

SINK/FLOAT:

A tank with clean tap water is loaded with the rinsed PET flake. The tank is agitated for five minutes and then allowed to sit for five minutes. Floatables are skimmed off and saved.

Data Table:

Run Number	Wash Temperature Setpoint (°C)	Starting Flake Weight (kg)	Total Floatables (grams)	% Total Floatables
Control	85	14	0.00	0.00%
Test 1	85	27	0.00	0.00%
Test 2	85	27	0.00	0.00%
Test 3	85	27	0.00	0.00%
Test 4	85	14	0.00	0.00%




Wash/Rinse Pictures

Variable	Settled Wash Water	Shaken Wash Water
Control		
Test 1		
Test 2		

Wash/Rinse Pictures

Variable	Settled Wash Water	Shaken Wash Water
Test 3		
Test 4		

Washed Flake/Floatable Pictures

Variable	Washed Flake	Floatables
Control		NOT APPLICABLE
Test 1		NOT APPLICABLE
Test 2		NOT APPLICABLE

Washed Flake/Floatable Pictures

Variable	Washed Flake	Floatables
Test 3		NOT APPLICABLE
Test 4		NOT APPLICABLE

APR PET Elutriation

References:

PET-P-05

Test Summary:

Remove lights (fines, labels, layers) from washed flake before extrusion







Procedure:

The elutriator is calibrated using the control flake post wash. Air-dried washed control PET flake is sent through the elutriator; the air speed is adjusted until the value for 0.5% for lights is achieved. Once the elutriator is calibrated, the rest of the control and tests are run. Air speed, heavies weight, lights weight, and lights % are recorded for each test.

Data Table:

Run Number	Air Speed (mps)	Heavies Weight (kg)	Lights Weight (grams)	% Lights Removed
Test 1	5.45	19.72	201.00	1.01%
Test 2	5.45	17.65	417.50	2.31%
Test 3	5.45	19.65	299.50	1.50%

Heavies/Lights Pictures

Variable	Heavies	Lights
Test 1		
Test 2		
Test 3		

Control Clumping Evaluation

References:

PET-S-08

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:

Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

Data Table:

Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	30	N	Y	0.00	0.00	0.00%
	30	N	N	0.00	0.00	0.00%
210	30	N	Y	0.00	0.00	0.00%
	30	N	N	0.00	0.00	0.00%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.

Control Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Non-Elutriated Loaded		NOT APPLICABLE
195°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

Control Clumping Pictures

Variable	Pans	Clumps
<p style="text-align: center;"> 210°C 30-Minute Crystallization Non-Elutriated Loaded </p>		<p style="text-align: center;">NOT APPLICABLE</p>
<p style="text-align: center;"> 210°C 30-Minute Crystallization Non-Elutriated No Load </p>		<p style="text-align: center;">NOT APPLICABLE</p>

Test 1 (PETG 162°C) Clumping Evaluation

References:

PET-S-08

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:







Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

Data Table:





Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	30	Y	Y	46.61	213.90	26.05%
	30	Y	N	21.92	105.57	12.75%
	30	N	Y	142.68	184.02	32.67%
	30	N	N	139.26	205.23	34.45%
	0	N	N	60.73	297.72	35.85%
210	30	Y	Y	59.10	153.12	21.22%
	30	Y	N	42.99	149.41	19.24%
	30	N	Y	166.19	210.88	37.71%
	30	N	N	55.61	217.98	27.36%
	0	N	N	69.48	186.41	25.59%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.







Test 1 (PETG 162°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Elutriated Loaded		
195°C 30-Minute Crystallization Elutriated No Load		
195°C 30-Minute Crystallization Non-Elutriated Loaded		





Test 1 (PETG 162°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Non-Elutriated No Load		
195°C No Crystallization Non-Elutriated No Load		

Test 1 (PETG 162°C) Clumping Pictures

Variable	Pans	Clumps
210°C 30-Minute Crystallization Elutriated Loaded		
210°C 30-Minute Crystallization Elutriated No Load		
210°C 30-Minute Crystallization Non-Elutriated Loaded		

Test 1 (PETG 162°C) Clumping Pictures

Variable	Pans	Clumps
210°C 30-Minute Crystallization Non-Elutriated No Load		
210°C No Crystallization Non-Elutriated No Load		

Test 2 (CPET 212°C) Clumping Evaluation

References:

PET-S-08

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:




Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

Data Table:

Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	60	N	N	0.00	0.00	0.00%
	30	Y	Y	0.00	0.00	0.00%
	30	Y	N	0.00	0.00	0.00%
	30	N	Y	0.00	0.00	0.00%
	30	N	N	0.00	0.00	0.00%
	0	N	N	0.00	0.00	0.00%
210	60	N	N	3.96	0.00	0.40%
	30	Y	Y	0.90	0.00	0.09%
	30	Y	N	0.00	0.00	0.00%
	30	N	Y	4.09	6.91	1.10%
	30	N	N	2.33	0.00	0.23%
	0	N	N	0.00	0.00	0.00%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.





Test 2 (CPET 212°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Elutriated Loaded		NOT APPLICABLE
195°C 30-Minute Crystallization Elutriated No Load		NOT APPLICABLE
195°C 30-Minute Crystallization Non-Elutriated Loaded		NOT APPLICABLE




Test 2 (CPETG 212°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE
195°C No Crystallization Non-Elutriated No Load		NOT APPLICABLE
195°C 60-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

Test 2 (CPET 212°C) Clumping Pictures

Variable	Pans	Clumps
210°C 30-Minute Crystallization Elutriated Loaded		NOT APPLICABLE
210°C 30-Minute Crystallization Elutriated No Load		NOT APPLICABLE
210°C 30-Minute Crystallization Non-Elutriated Loaded		

Test 2 (CPET 212°C) Clumping Pictures

Variable	Pans	Clumps
210°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE
210°C No Crystallization Non-Elutriated No Load		NOT APPLICABLE
210°C 60-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

Test 3 (CPET 201°C) Clumping Evaluation

References:

PET-S-08

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:







Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

Data Table:


Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	30	Y	Y	0.92	2.10	0.30%
	30	Y	N	0.99	3.50	0.45%
	30	N	Y	4.93	24.20	2.91%
	30	N	N	5.18	11.31	1.65%
210	30	Y	Y	9.08	30.26	3.93%
	30	Y	N	11.96	25.83	3.78%
	30	N	Y	40.65	31.50	7.22%
	30	N	N	163.45	121.81	28.53%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.







Test 3 (CPET 201°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Elutriated Loaded		
195°C 30-Minute Crystallization Elutriated No Load		
195°C 30-Minute Crystallization Non-Elutriated Loaded		

Test 3 (CPETG 201°C) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Non-Elutriated No Load		

Test 3 (CPET 201°C) Clumping Pictures

Variable	Pans	Clumps
<p>210°C 30-Minute Crystallization Elutriated Loaded</p>		
<p>210°C 30-Minute Crystallization Elutriated No Load</p>		
<p>210°C 30-Minute Crystallization Non-Elutriated Loaded</p>		

Test 3 (CPET 201°C) Clumping Pictures

Variable	Pans	Clumps
<p>210°C 30-Minute Crystallization Non-Elutriated No Load</p>		

Test 4 (Thermoform) Clumping Evaluation

References:

PET-S-08

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:

Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

50% Data Table:

Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	60	N	N	0.00	0.00	0.00%
	30	N	Y	0.00	0.00	0.00%
	30	N	N	0.45	2.44	0.29%
210	60	N	N	0.00	0.00	0.00%
	30	N	Y	0.00	0.00	0.00%
	30	N	N	0.00	0.00	0.00%

100% Data Table:

Temperature (°C)	Crystallization Time (min)	Elutriated (Y/N)	Load (lbs)	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
195	30	N	Y	0.00	1.27	0.13%
	30	N	N	0.00	0.00	0.00%
210	30	N	Y	0.17	11.62	1.18%
	30	N	N	0.00	0.00	0.00%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.

Test 4 50% (Thermoform Clumping Pictures)

Variable	Pans	Clumps
<p style="text-align: center;"> 195°C 60-Minute Crystallization Non-Elutriated No Load </p>		<p style="text-align: center;">NOT APPLICABLE</p>
<p style="text-align: center;"> 195°C 30-Minute Crystallization Non-Elutriated Loaded </p>		<p style="text-align: center;">NOT APPLICABLE</p>
<p style="text-align: center;"> 195°C 30-Minute Crystallization Non-Elutriated No Load </p>		

Test 4 50% (Thermoform) Clumping Pictures

Variable	Pans	Clumps
210°C 60-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE
210°C 30-Minute Crystallization Non-Elutriated Loaded		NOT APPLICABLE
210°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

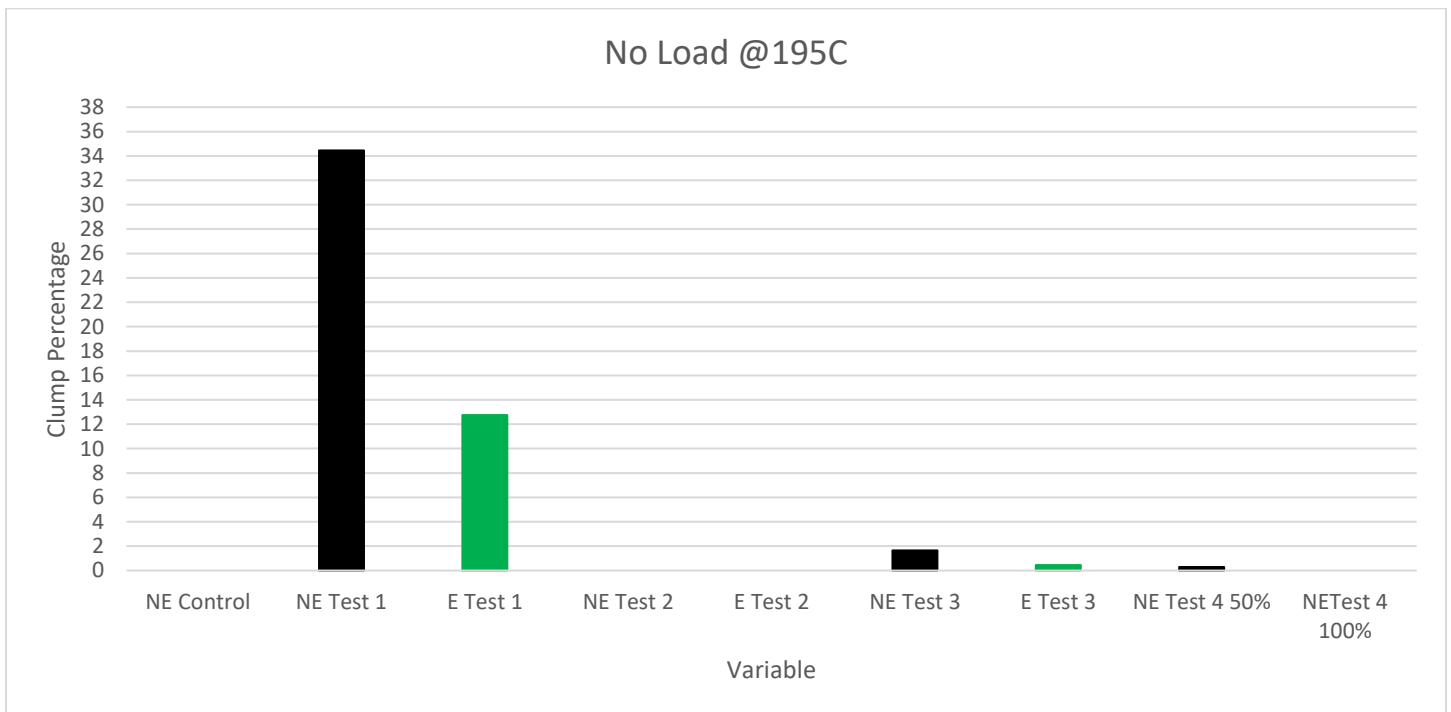
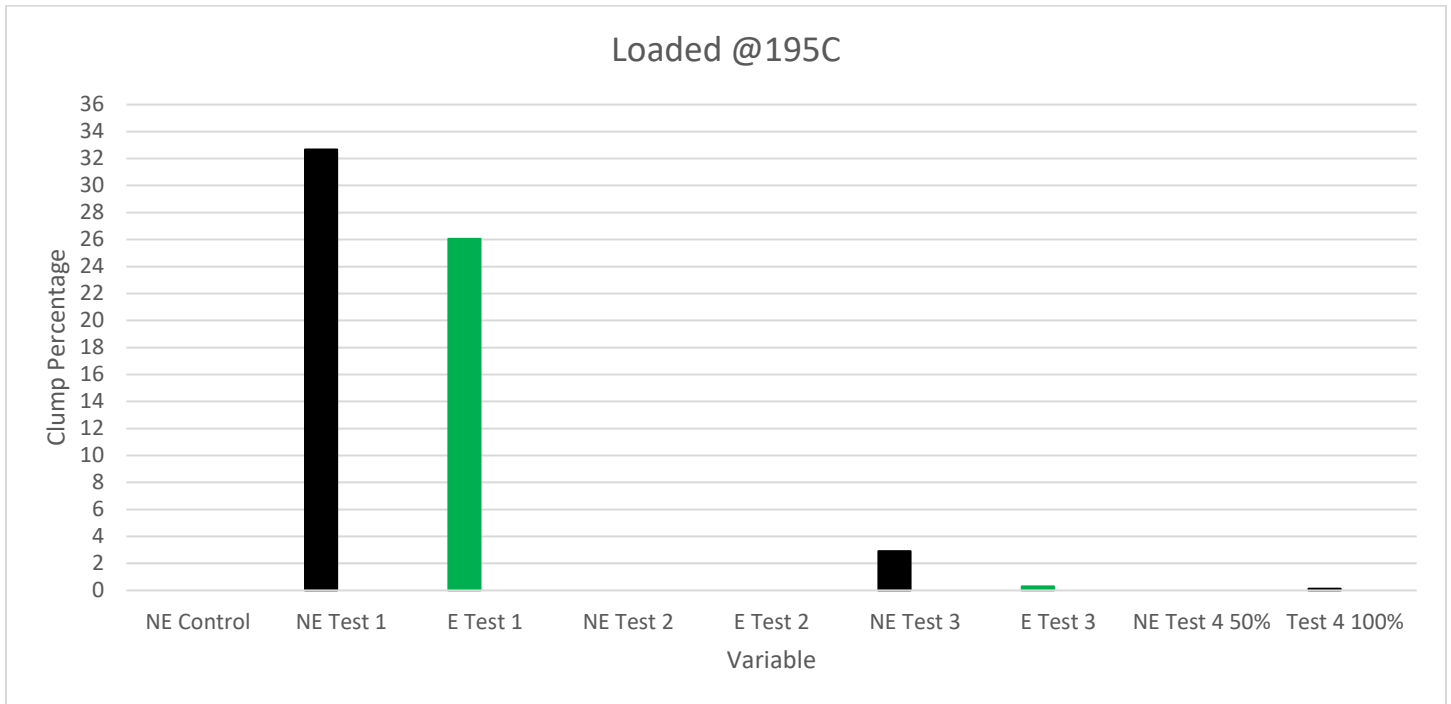
Test 4 100% (Thermoform) Clumping Pictures

Variable	Pans	Clumps
195°C 30-Minute Crystallization Non-Elutriated Loaded		NOT APPLICABLE
195°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

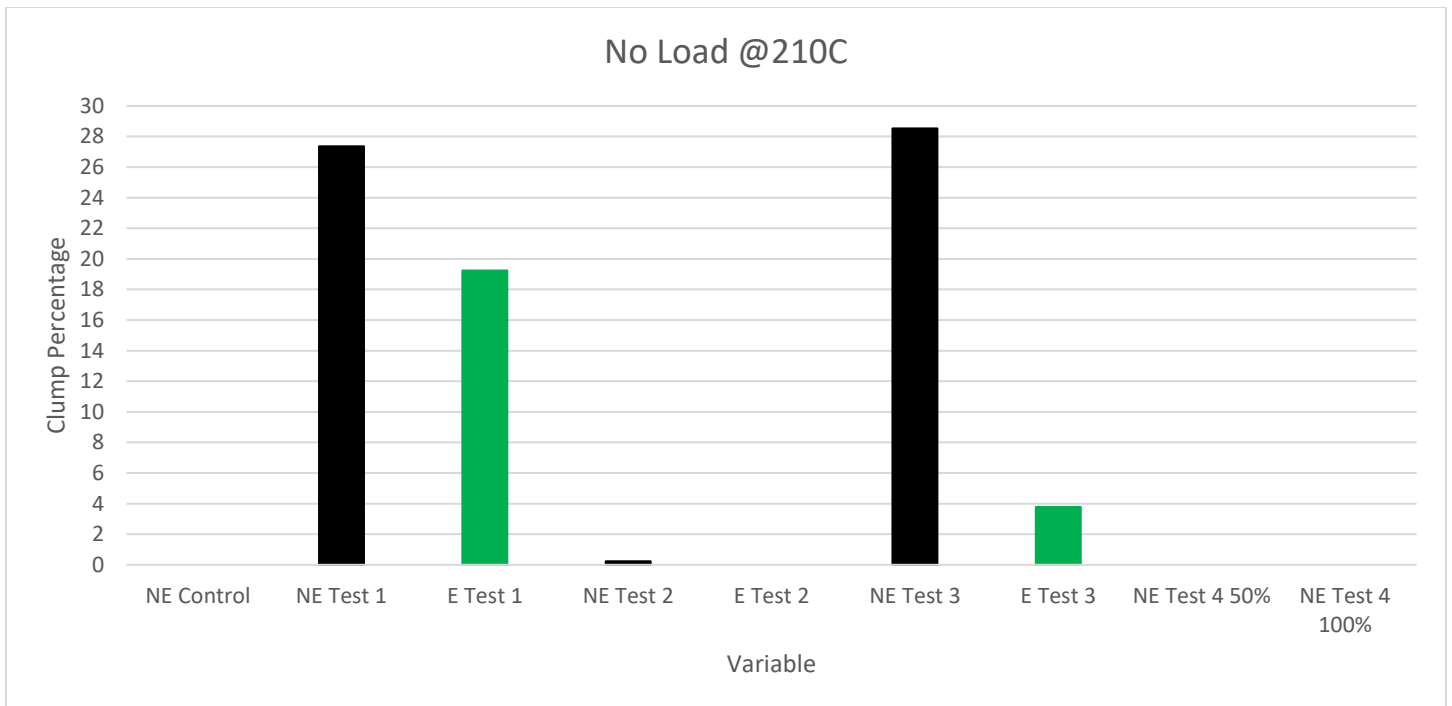
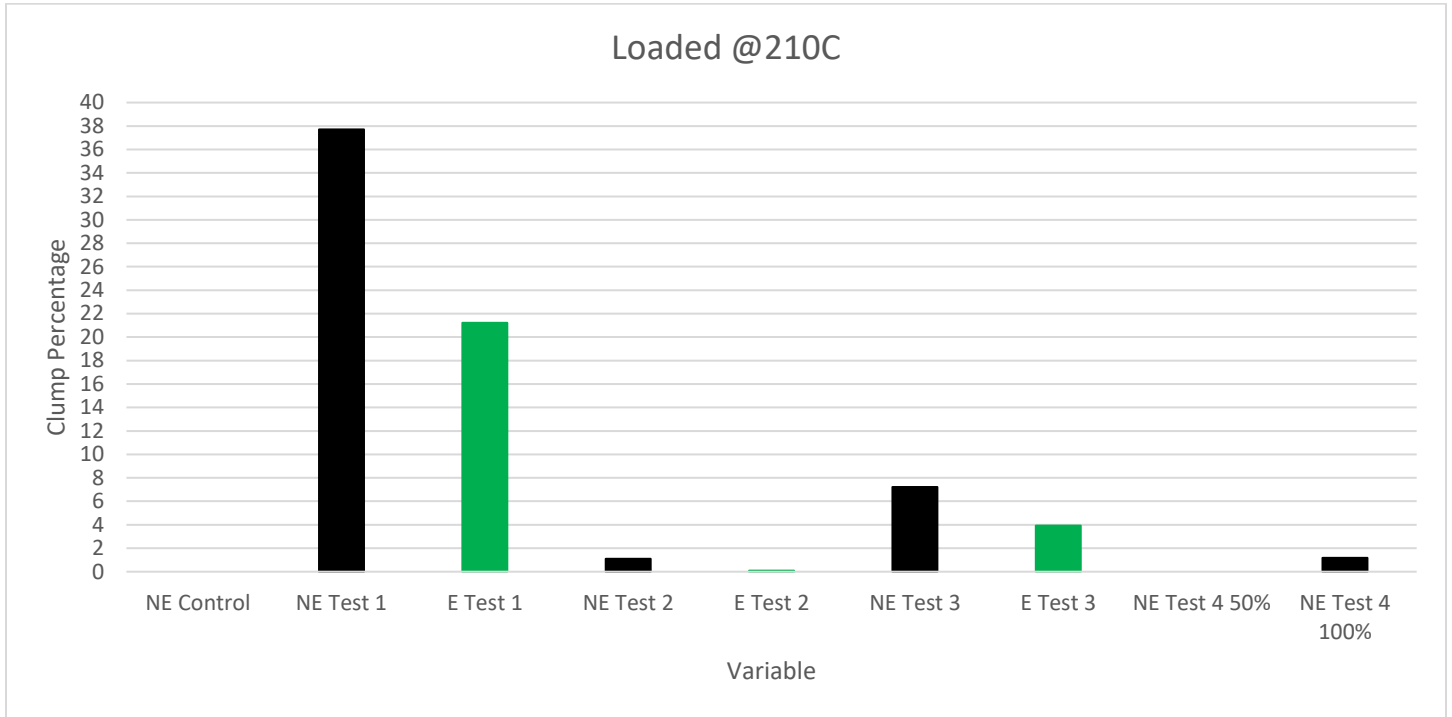
Test 4 100% (Thermoform) Clumping Pictures

Variable	Pans	Clumps
210°C 30-Minute Crystallization Non-Elutriated Loaded		
210°C 30-Minute Crystallization Non-Elutriated No Load		NOT APPLICABLE

Appendix



Appendix



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