

TCEP-P 04.- Washing and Sink/Float Separation of PET Flake

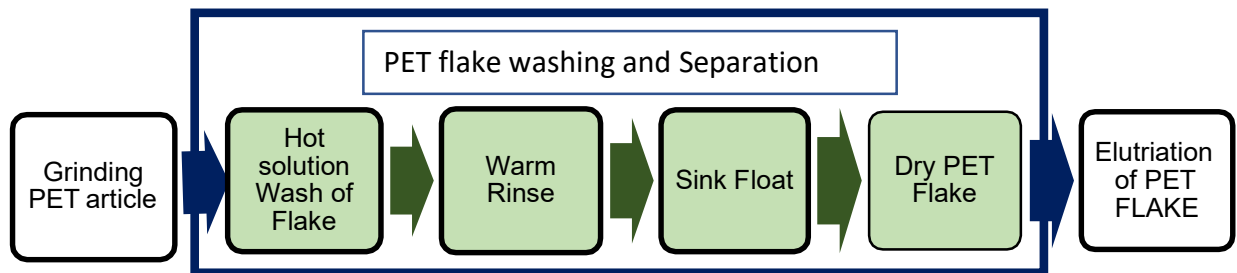
Background

The intention of the washing and sink/float of the shredded specimen is to separate the labels, adhesives, coating and other attachments from the main packaging body.

After granulation, PET flake is washed in a hot aqueous caustic and detergent solution where dirtiness and some of the attachments are liberated from the main packaging body. Light materials will float while PET flake will sink to the bottom of the equipment.

Practice summary and illustration

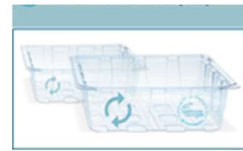
Granulated PET is washed in a hot aqueous caustic solution with a high level of agitation. Washed flake is then rinsed in warm water. Rinsed flake is exposed to a final float/sink step in tap water. Separation of any floating materials that are free to float in water are removed at each of the 3 steps. Washed flake is then dried.



A material balance evaluation must be conducted by an to verify the efficiency and losses of the washing and separation process by streams.

Equipment required

- Stainless steel mixing tank with the following specifications:
 - Size needs to allow a water: PET flake mix in a weight ratio of 4:1.
 - The volume will need to meet a recommended ratio of the water height to the tank width of 0.8 to 1.0 and not less than 0.6 or greater than 1.5.
 - A variable speed agitator with an impeller bade diameter of at least 0,33 times the diameter of the mixing tank diameter.
 - In the case that a round tank is used, it needs to have at least 3 baffles installed.
- Controlled temperature heating system. Temperature needs to be registered
- De-watering screen. This can be a wire mess screen or a perforate metal with holes with a size of less than 1mm diameter.
- Float/sink Tank:
 - To hold a water to flake ratio of no less than 8:1 and no more than 10:1 in weight
 - It will operate at room temperature.
- Lab oven and trays to dry samples



Materials and Reagents Required

- The material approved to be used as detergent wash solution:
 - Non-ionic surfactant Sodium hydroxide,
 - NaOH, granules or solution
- Flake made from control article
- Flakes made from test article

Practice Steps

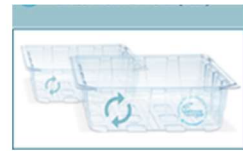
Weigh the amount of PET flake required for the evaluation as specified in the test **or Critical Guidance Protocol (TBD)**.

Hot Wash

1. Prepare the wash solution
 - a. Add tap water to the stainless wash vessel. The weight of water to be four times the weight of PET flake planned for use in wash (for example, 2000g of water vs 500 g of PET flakes).
 - b. Start heating the vessel. Verify the temperature increase and control it.
 - c. Start agitator. Verify correct operation
 - d. Add NaOH granules or concentrate solution to the water with agitation to create a 1% by weight content of NaOH.
 - e. Add surfactant to the tap water to create a 0.3% by weight content of surfactant.
2. When the wash solution reaches 70° C:
 - a. Add the PET flake
 - b. Maintain wash solution temperature at 70°C.
3. Increase the agitator speed up to the target rpm.
4. Maintain agitator for 15 minutes. Then:
 - a. Stop agitation
 - b. Stop heating
 - c. Leave the mix to sit down for five (5) minutes.
5. Skim off and save any materials floating on top of the surface.
6. Remove the liquid-flake mix over the de-watering screen and save the wash water.
7. Stir the retained wash water while taking a 1 liter sample for later evaluation.

Warm Rinse

1. Prepare the rinse equipment. It can be the same or different from the washing equipment, but have to be identical in dimensions. It has to be built in Stainless Steel
 - a. Verify that equipment is clean
 - b. Fill the vessel with Tap water. The weight (volume) of water has to be 4X the final weight of flake.
 - c. Start agitation
 - d. Start heating
2. When the rinse water reaches 45° C:
 - a. Add the PET flake
 - b. Maintain the rinse water temperature at 45° C
3. Increase the agitator speed up to the target rpm.



- a. Maintain agitation at target rpm during 5 minutes
4. After 5 minutes,
 - a. Stop the agitator.
 - b. Stop heating to the equipment.
5. Allow the product on the vessel to sit for five minutes.
6. Skim off and save any materials floating on top of the surface.
7. Pour the liquid-flake mix over the de-water screen to recover the PET flake as well as any sinking solids contained in the rinse water.

Sink Float

1. Prepare the Sink/Float equipment. It can be the same or different from the washing equipment, but have to be identical in dimensions. It has to be built in Stainless Steel
 - a. Verify that the vessel is clean.
 - b. Fill the vessel with tap water. Use at least 8X and no more than 10X the weight of PET flake.
 - c. Add the PET flake to the vessel
 - d. Start agitation in a low-speed mode.
 - e. Maintain agitation for 5 minutes.
 - f. The stop agitation
2. Allow the container to sit for five (5) minutes.
3. Skim off and save any materials floating on top of the surface.
4. Pour the liquid-flake mix over the de-water screen to recover the PET flake as well as any sinking solids contained in the rinse water.

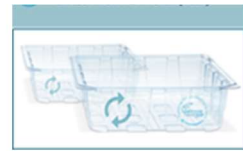
Dry by Centrifugation. -

Due to the properties of the flakes from trays are more fragile, a non-blade low speed centrifuge should be preferred.

1. Verify that the equipment is clean and free from any material from previous test to avoid cross contaminations.
2. Feed the flakes obtained from the dewatering screen
3. The maximum speed of the centrifuge will be 300rpm (confirm final acceptable speed)
4. Collect all the product at the outlet of the centrifuge
5. The product will be weighted to understand the product losses across the process.
6. Retain a sample for testing

Alternative/comparative: Oven Dry

1. Dry PET flake obtained from the de-watering screen.
 - a. The flake can be dried in air, or in a lab oven.
 - b. Control de temperature
 - i. Flake temperature during drying cannot exceed 60° C to prevent oxidation of any material in/on the flake.
 - c. The material will be considered dry when is dry at the touch and is free flowing.
2. Retain a 50-100 g sample of the flake resulting from this practice step.



Mass balance.-

- A mass balance of the washing, rinsing, floating and centrifuge will be made

Weight of sample placed in the washing	
Weigh of flakes obtained at the centrifuge	
% product lost.	

DOCUMENT REVISION HISTORY

Version	Publication Date	Revision notes
V0	Sept 21	NEW DOCUMENT
V1	April 2023	Platform, document name and logo changes.