Natural HDPE Flake Washing Test

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Introduction – Scope, Significance and Use

The following protocol is a screening test designed to provide a generic wash process for evaluation of the effects of container components (coatings, multilayer structures, adhesives, label substrates and bleeding labels, etc.) on recycled natural HDPE material. A "bleeding ink or label" is one with water dispersible or soluble inks that result in discolored wash water and have the potential of also staining natural HDPE flake. The method of incorporating the component with the flake for washing and the specific analysis technique will be provided in the component specific protocol.

Method Summary

Equipment and Supplies Required

- Container component for evaluation (e.g. coating, label, adhesive etc.)
- Clean, natural HDPE flakes about 3/8" nominal (500 grams/test) (depending upon particular evaluation, component may be applied to HDPE container prior to granulation)
- Preferred - Baffled beaker - stainless steel, six inches diameter by nine inches in height with four 3/4-inch baffles.
    - Alternate - similar size beaker without baffles
- A container to serve as a float/sink tank sized to hold room temperature water and flake at an 8:1 to no more than 10:1 by weight mix ratio.
- Hot plate capable maintaining the required wash temperature as required by Wash Procedure 2 in **APR-O-P-04**.
- Watch glass to cover beaker when heating
- Scale or balance capable of measuring 500 (+\- 0.5*)* grams
- Oven capable of drying flake at 60^0C
- Cookie sheet (or other suitable tray with a minimum area of 120 sq.in.)
- Cover for beaker which can accommodate thermometer and stirring impeller rod
- Overhead stirrer capable of 1,000 rpm

- Stirring impeller - pitched, 3 blades, and ideally a minimum blade length at least 0.33 of the mixing beaker diameter or width.
- Manual stirring rod
- Thermometer
- Preferred - De-watering screen- this can be a wire mesh screen or a perforated metal surface with openings greater than 1 mm in size
    - Alternate – Strainer, non-aluminum
- Distilled or deionized Water
- Graduated cylinder
- Colorimeter capable of measuring flake color in reflectance mode in L*a*b* color space (CIELAB)

Wash Chemicals List

- Materials for APR detergent wash solution
    - Triton X-100 nonionic surfactant
        - Optional alternative wash detergent: MacDermid RP24
    - Sodium hydroxide, NaOH, granules or solution

Control and Test Materials

- Control flake should not include the component (coating, label, adhesive etc.) that is being tested.
    - It is suggested that the test component being evaluated be applied to a container made from one of the control resins identified in Section O-P-01 in the **APR-O-P-00 Olefin Practices** document.
- Preparation of container test component for evaluation - this will be specific for each container component and may require affixing component to container prior to granulation.
    - Guidance for preparing the HDPE articles for evaluation can be found in **APR-O-P-02**
    - Test flake will depend upon the type of sample being evaluated (any label requiring adhesive should be adhered to the container prior to testing), Coverage area, degree of ink coverage, and test wt% (weight%) can all be increased beyond the suggested levels to create a worse case situation.
        - For coatings, an HDPE bottle will have the coating applied and cured and tested by blending at a 50:50 ratio with control flake.
        - Pressure sensitive labels should be tested at a minimum of 20% coverage area, and tested without further dilution with control flake.
        - Shrink and stretch labels can be preshrunk and tested at 3 wt% added to control flake.
        - Direct print labels should cover a minimum of 20% coverage area on the HDPE bottle and tested without further dilution with control flake.
        - Wrap labels should be tested at a 0.6 wt% level without further dilution with control flake.
        - In-mold labels should be tested at a 20% coverage area without further dilution with control flake

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Caustic Wash Procedure
(to be used for both the Control and Test Component Flake)

- Wash Procedure 2- Caustic Wash, as described in APR-O-P-04 should be used for this screening evaluation.
- Proceed to rinse, sink-float and dry recovered materials as described in APR-O-P-04.
    - Note: If the sample of flake used with this screening test is small (i.e. <500 g), a simpler rinse procedure may be employed.
        - Transfer all skimmed and de-watered floating material into a strainer.
        - Rinse the HDPE flakes while still in the strainer with running tap water while vigorously stirring the flake for 5 minutes.
        - Allow the excess rinse water to drain.
- Retain a 100-gram sample of the washed and dried flake resulting from these practice steps for color measurements.

Important Note:
It is assumed that any label or similar contamination remaining after the wash protocol has been performed and which is not adhered to HDPE flake material would be removed by a subsequent separation process and would not present a problem. Therefore, prior to performing the color measurements, any label contamination which is <u>not</u> adhered to the HDPE flake can be removed from the material. This may be accomplished~~,~~ by hand separation, or by any means sufficient to accomplish the task.

<u>HDPE Flake Color Evaluation Procedure</u>

- Using the retained 100 grams natural HDPE control and test flake samples produced from the above procedure perform color analysis, according to the machine manufacturer's instructions, using the following format:

    - Take five measurements, in reflectance on each sample. Record data as CIE L*a*b* color coordinates. Adjust the position of the sample holder prior to each measurement to expose different sample areas to measurement. The **L*** measures brightness vs. darkness of the flake, **a*** measures red/green color components and **b*** measures yellow/blue color components.

- Report all of the axis readings (such as **L***, **a***, **b***) for all five samples as well as the average and standard deviation.

- Evaluation Guidance.
  - For colored HDPE applications, changes in L*, a* and b* can be tolerated depending upon the end use of the HDPE material. However, dark colors with an L* value less than 40, can present problems during the sortation process and are not preferred at this time.

  - For natural HDPE applications, there should be minimal changes in the color coordinates. (Refer to the HDPE Critical Guidance documents for limitations on acceptable color changes.)

  - The total color difference between the control and test samples can be used to evaluate the quality of the recycled HDPE flake using the ΔE value.
    - ΔE values >3.5 would become noticeable in color shift for natural HDPE and therefore could be detrimental for use in natural (uncolored/non-tinted) applications.
    - Because additional color can develop when washed flake is subjected to a melt history during product fabrication, meeting the color requirements as outlined in either the Benchmark or Critical Guidance tests are more indicative to determine if the flake quality meets the APR definitions of Preferred or Detrimental.
  - ΔE is calculated using the following formula:

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

- Discolored water should be evaluated to see if the discoloration is acceptable for release to municipal sewer systems without further treatment. Strongly discolored water is likely to be unacceptable to many municipal wastewater treatment systems and labels which cause such discoloration should be avoided.

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	8-23-2021	New test replacing previous HDPE-S-01 and HDPE-S-02