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PE Film Standard Laboratory Processing Practices

Introduction

This document presents Standard Laboratory Practices recommended by the APR to prepare PE film and flexible products for use in tests that evaluate the compatibility of the materials with commercial PE film recycling processes. These practices are referenced and employed in various APR Test Methods and Guidance Protocols. Not all test protocols will require every practice described here.

The general term PE film and flexible product is used so that these practices can apply to any film or flexible package made from PE resin in the form of either cast or blown extrusion. Testing can be employed with a PE film or flexible product alone, or to evaluate the impact of design features such as closures, attachments, labels, or additives, for example, on recycling. These practices can also be used to evaluate PE virgin resin pellets incorporating innovations or new design features.

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Selection of Control Film or Control Resin, FPE-P-01

Background

Recycle testing typically involves comparing the performance of a plastic product containing a design feature or innovation to that of a control product that does not employ the design feature or innovation. This practice provides the steps used to select a control film or virgin resin and produce any necessary test materials suitable for control comparison.

There are hundreds of PE resins available in the North American market that can be used to make flexible film packaging articles. These differ in melt flow rate, density, and other physical and chemical properties. Additionally, the recycling stream in which innovative film and flexible packaging must be compatible is comprised of thousands of different package formats made from the hundreds of PE resins available.

Thus, the selection of a small number of commercial virgin control resins for use in comparing and blending with the Innovation test article is not practical. Instead, a film control article is used for testing. Virgin resins may be used as controls only when the innovation to be tested is submitted in pellet or resin form as well.

The incoming starting PE control film or resin representing the ultimate package construction to be tested is comprised of the test package or resin without the design feature or innovation. The control structure should be as close to the innovation structure as possible. The same base resin must be used for both the innovation and the control. The control base resin must fall within the MFR range for blown films. *The Control will be an APR-Preferred structure, comprised of a minimum of 90% PE and copolymers by weight of the total packaging structure as defined in the Design Guide.* Printing, overlacquers and/or adhesives are not allowed on the control film, even if pre-tested. Printing, overlacquers and adhesives are currently classified as Detrimental to Recycling per the Design Guide, and Detrimental materials should not be used in controls.

Two options are available:

- Option #1 – The preferred option for a control is to use a 100% PE film article or resin similar to the Innovative package in properties and function, and known to be recyclable without controversy. The test article and control are as simple as possible and avoid inclusions unrelated to the feature to be tested.
- Option #2 – A second option for a control is to use a PE film article consisting of the same materials as the innovative package, known to be recyclable without controversy, with the only difference being the specific ingredient/feature being evaluated is not present in the control.

If Option #2 is chosen, the proposed control for Critical Guidance Recognition applications must be approved by the APR Technical Director prior to testing.

If the innovation to be tested is an ink, pigment, or adhesively applied label, Practice FP-P-02 should be used to prepare the items.

Preparation of Printed PE Film & Flexible Products for Evaluation, FPE-P-02

Background

- This practice lists the steps taken to prepare a film or flexible package test article for evaluation of direct printed ink on film surface or reverse printing, and non-paper pressure sensitive labels composed of adhesive, substrate and inks (paper pressure-sensitive labels on film packages are not in scope for testing with APR protocols).

Evaluations can involve the “intended” print or label or a “generic” print or label.

- An "intended" product is prepared as it will be sold in the market using the specific materials and design that will be marketed. Typically, intended products are ready for production or commercialization. For an “intended” product, the coating weight of inks must be reported.

- A "generic" product is one used in the laboratory to represent a variety of market products and represents a facsimile of what will be commercialized. For example, the ink colors and composition are represented but the label is not the branded label. In these cases, the labeled PE test articles should be tested with 100% surface area ink coverage, (white, black, B, Y, R) at minimum 2 lbs/rm solids coating weight AND minimum 3% ratio ink solids/film basis weight.
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Preparation of test article with direct printing or pressure sensitive label printing

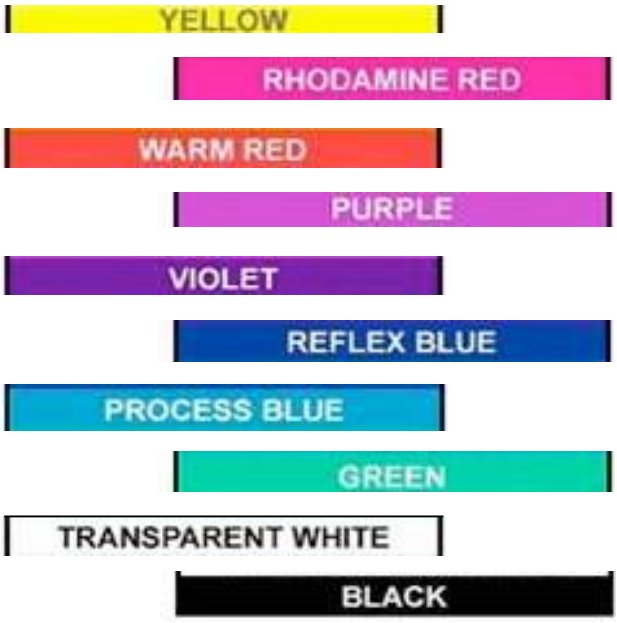
Test articles with labels applied can be tested printed or un-printed. For example, if the adhesive is the feature being tested, an unprinted label is desirable. However, APR advises all inks to be tested as well.

When the inks or print technologies are the test subject, all printed applied labels, as well as direct printed labels, can be tested with intended inks and graphics or with generic inks. Test articles with printing at the indicated levels will be compared to control articles without labels.

When using generic inks, the following ink pattern options may be used:

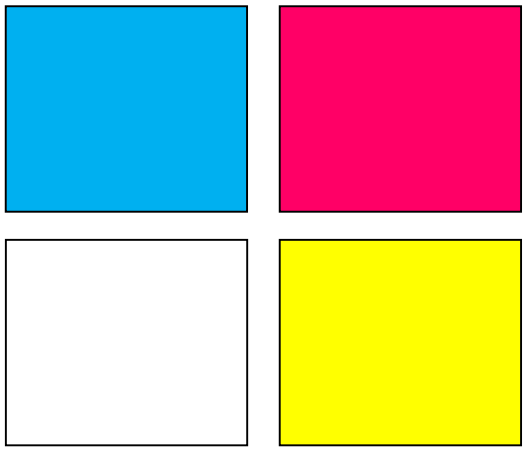
Option 1 Pantone
(One Label Multiple Colors or product)

Option 2 Pantone
(Multiple Labels or product with flood coat on each)



Option 3 w/4 Color
(One Label Multiple Colors or product)

Option 4 w/4 Color
(Multiple Labels or product with flood coat on each)



Granulating PE Film and Flexible Products to Flake, FPE-P-03

Background

PE products are reduced in size by shredding or granulating. Grinding products in a rotary granulator is typical in a lab environment to simulate both processes. This step creates PE flake that is used in subsequent process steps. Granulating is also intended to liberate package components such as labels, fitment and attachments from the PE product.

Some film and flexible package structures may not be suitable for a lab scale grinder. In that case granulation is optional, provided the test articles are suitable for blending with controls consistently and for extrusion for pellet testing.

Equipment Required

- Weigh scale (±0.01 grams)
- Rotary plastic granulator fit with a screen containing holes within the range required for densification or extrusion. The machine is to be evacuated via gravity (without pneumatic transport)
- Compressed air line and/or shop vacuum cleaner to use in cleaning the granulator

Materials Required

- Test article containing innovation or design feature as defined in FPE-P-02
- Control article as defined in FPE-P-01
- Soft cloths for cleaning the granulator
- Products such as plastic pails or bags to hold granulated flake samples

Practice Steps

Test and control articles are each ground separately. The granulator is cleaned before and after granulating each sample:

1. Observe all safety practices relevant to the machine, including lock-out procedures.
2. Clean the plastic granulator prior to use with compressed air and/or a shop vacuum. Wipe up any fines or other contamination with a clean cloth, if necessary. Be aware of material hang-up inside the granulator behind the cutting head which may be difficult to reach and inspect. Do not leave fragments of the cleaning cloth in the machine.
3. Before grinding, retain five (5) 100g samples each of the Control and Test articles in case additional testing is needed.
4. Check to ensure the granulator screen is properly installed with the proper diameter holes.
5. Weigh the required number of Test and Control articles to provide the desired weight of granulate for each sample as specified in the test to be conducted

6. Granulate a given sample by manually feeding the articles into the granulator.
    a. Granulated material may include PE flakes, label pieces, and closure pieces.
7. Store each sample in a sealed and labeled product.
8. Retain 5 100-gram samples of each ground sample for use in case additional testing is needed.
9. Clean the granulator between each sample.

Film Densification for PE Film and Flexible Product or Flake, FPE-P-04

Background

Film densification in the industrial PE recycling process occurs at the feed of the extruder or prior to extrusion depending on the technologies. In commercial recycling facilities, flake is sometimes washed and dried, then densified to prepare for production. Washing is not a Processing Practice recommended for APR lab scale tests of film and flexible packaging innovations or design features. However, densification is necessary to proceed with testing.

To model the commercial process there are two potential densification methods that can be used depending on the equipment capability of the laboratory – oven densification and mechanical densification. Generally, oven densification is used when the material cannot be pre-granulated and mechanical densification is used for granulated flake. Granulation is not required if the mechanical densifier used will accept whole articles as feed.

Equipment Required

- Weigh scale (±0.01 grams)
- Film densifier or oven capacity capable of 200°C
- Compressed air line and/or shop vacuum cleaner to use in cleaning the film densifier

Materials and Reagent Required

- Shop rags for cleaning
- Oven nonstick pans
- Products such as plastic pails or bags to hold samples pre-and post-processing
- Prepared flake or film produced from the control article
- Prepared flake or film produced from the test article

Oven Practice Steps

1. Prepare nonstick pans for material
2. Place 500-750-gram samples of material in pans
3. Set oven temperature by using the film DSC primary peak melt temperature +- 5 degrees or no greater than 190°C
4. Place sample in oven for no longer than 40 minutes depending on the known properties of the material. Both the control and the test should be submitted to the same conditions
5. Remove pan after time has been met and let cool.
6. Remove sample from pan and grind per the FPE-P-03 procedure

Mechanical Film Densifier Practice Steps

1. Samples prepared must be dry and at room temperature
2. Add granulated flake or whole articles to the film densifier for 10-20 minutes depending on the material melt temperature
3. Ensure that the material temperature during the process does not exceed the primary melting temperature of the individual sample as determined by DSC.
4. Once material agglomerates, tap water is introduced to cool material
5. When desired particle size is reached for proper extrusion feeding remove material
6. Dry the material with ambient air till less than 1% moisture is reached
7. Granulation per the FPE-P-03 practice is not required.

Melt Filtration and Pelletization, FPE-P-05

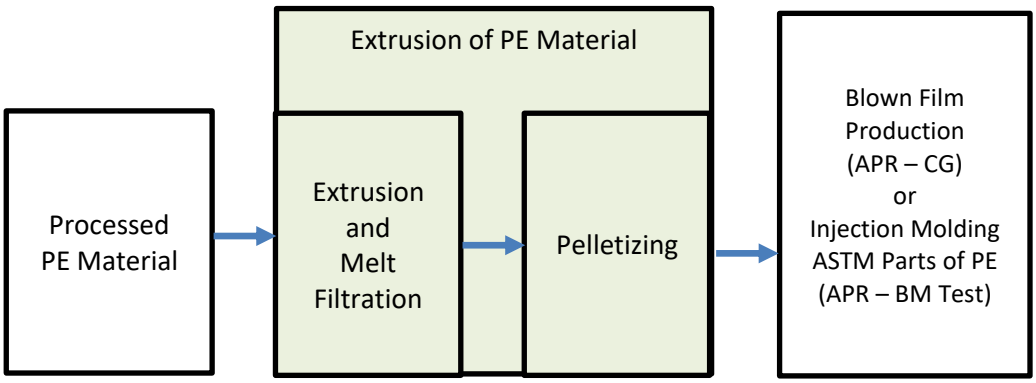
Background

In the industrial PE recycling process, the PE material is melted in an extruder and the melted polymer is filtered so that the flake can be converted to pellets. This Practice represents a standard method to extrude and create PE pellets from blended PE materials.

The practice includes the steps of drying, extrusion and pellet forming. At the same time, it defines required observations and samples for further testing.

Practice summary and illustration

Material is blended as required for a specific test method or Critical Guidance Protocol, and then dried in a drier. Dried flake is melted and filtered in an extruder to create cut pellets. Melt pressure is recorded ahead of the melt filter.



Note: There are situations in laboratory recycling assessments where it is necessary to add a heat history to PE resin to simulate a molding operation. This Extrusion Practice can be followed to add a heat history to PE samples – flake or pellets. Melt filtration and pressure measurement is not necessary when the practice is used only to add a heat history.

Equipment required

- Dryer capable of drying PE material and/or pellet at up to 90° C
- Extruder suitable for PE materials processing in the laboratory.
    - A 25 to 35 mm extruder with a 24:1 to 36:1 L/D is suitable for laboratory use.
    - A single-screw extruder is strongly recommended. If a twin-screw extruder is used, it shall be noted. Twin-screw extruders used for APR testing should not have reverse flights, mixing flights or star flights and should only contain forward conveying spiral screw elements. If a twin-screw extruder is used, this must be reported with the results.
    - Melt residence time in the extruder should be no more than 6 minutes.

- The extruder requires a means for maintaining dried PE flake in a dry state while in the hopper and during extrusion.
- The extruder is equipped with a breaker plate and screen pack.
- The extruder must display pressure values ahead of the screen so that pressure data may be recorded.
- The extruder requires water bath and pelletizer.

- A scale for measuring extruder output.

Materials required

- Products such as plastic pails or bags to hold final pellet samples
- Screen pack for the extruder - 40/150/40 mesh
- Processed control article, processed test article, consisting of blends of flake or densified material.
- In some cases where size reduction is not possible, whole test articles are bulk fed.

Practice steps

1. Densification may be required to allow good feeding into the extruder feed throat and to achieve steady pressure readings ahead of the screen pack. If densification is used both the control and test will need to be prepared using the same practice. Reference P-FPE-04.
2. Weigh the required amount of flake or pellet for processing the required minimum run time of 30 minutes. Dry the flake or pellet in a dryer at 80-90°C for a minimum of 30 minutes and up to 1 hour to remove surface moisture.
3. Starting with the control material, prepare an extruder suitable for PE flake or pellet processing in the laboratory.
4. Extrusion Steps
   - Extrude the control article flake or pellet first at DSC secondary peak temperature identified for the innovation, +10°C to 20°C, but not to exceed 245°C.
     a. Use DSC results to narrow down temperature range based on specific material requirements. Temperatures may be adjusted up to 25% higher than the highest DSC peak to make sure all components of the innovative film formulation melt, as long as the extrusion melt temperature stays in the 190 – 245C range. If DSC secondary peak exceeds 240°C, do not proceed.
        i. The melt will be filtered with a clean 40/150/40 mesh melt filter
        ii. The melt is extruded through a die into strands of approx. 2.5mm diameter. The strands are rapidly cooled in water and fed into a pelletizer. It is important to achieve similar pellet sizes for each of the test and control materials.
     b. Start run timer after a steady state has been reached
     c. Record pressure values ahead of the screen pack at a minimum of every five minutes for a 30 minute's run time after the steady state has been reached. This may be done with a continuous automatic pressure recorder or manually.
     d. Record any occurrence of unusual conditions such as sticking, fumes, odor or build-up occurring at the feed throat or die exit of the extruder.

e. Record surface irregularities such as, but not limited to, porosity, roughness, grainy appearance, shape, gloss etc. of the strand and pellet.
f. Save a sample of extruded pellets for melt flow index measurement. Sample pellets to be obtained halfway through run time or later at random, as long as steady-state is ongoing. Additional samples can be retained for color and other measurements as desired.
g. Retain a 100-gram sample from this practice step for later use as necessary.
h. Purge the extruder and change the screen pack between each run with virgin PE resin
i. Store the pelletized samples in pails or bags
j. Extrude all subsequent samples at the same conditions; achieve pellets of similar size for each sample set when the pellets will be used for a film or part production.
k. Processing conditions should be the same for control and test within allowable ranges. Any process changes must be recorded and reported.

5. Repeat steps 5a through 5k for test variable blends. Extrude test variable blend at same conditions as control blend with a melt temperature delta to control of +/- 5°C. Use same screw design and screw RPM for control and test. If performance and results vary record and report differences.

Steps Required to Assess Pressure Values

For the control and test material:

After the steady state has been reached, calculate the initial average pressure, P_i, over the first five minutes of extrusion as well as the average pressure value for the final five minutes of extrusion, P_f. When:

$(P_f – P,) / P_i$ this is record as evidence for later determination of screen pack build-up.

Record control and the test graphically to represent the potential for screen pack build up.

Blown Film Extrusion of PE Pellet, FPE-P-06

Background

In the industrial PE recycling process, extruded PE pellets are manufactured to the end application of blown film. This document presents a standard practice to use the PE pellets from the blended PE control articles and test articles to make a blown film material for lab testing.

Practice summary and illustration
Pellet is blended as required for a specific test method or Critical Guidance Protocol, and then dried in a drier. Dried pellet is melted and filtered in an extruder to create blown film.

Equipment required

- Dryer capable of drying PE flake and/or pellet at up to 90° C
- Laboratory scale
- Blown film test machine suitable for pellet to film processing
    - Melt residence time in the extruder should be no more than 6 minutes.
    - The extruder must display pressure values ahead of the screen so that pressure data may be recorded.
    - Extruder fitted with screen pack mesh 40/60/40
    - The extruder is capable of a blow up ratio of 2.5.
- A scale for measuring extruder output.

Materials required

- Products such as plastic pails or bags to hold final film samples
- Processed control pellet blend and processed test pellet blend

Practice steps

1. Weigh the required amount of pellet blends for processing the required minimum run time of 30 minutes.
2. Dry the flake or pellet in a dryer at 80-90°C for a minimum of 30 minutes and up to 1 hour to remove surface moisture.
3. Starting with the control material, prepare an extruder
4. Extrusion Steps
    a. Extrude the control pellet blend first into a blown film sample for testing.
    b. Use DSC results from the innovative film to narrow down temperature range based on specific material requirements. Temperatures may be adjusted up to 25% higher than the highest DSC peak to make sure all components of the film formulation melt, as long as the extrusion melt temperature stays in the 190 – 245C range. A change in temperature must be documented and reported and requires a re-start of steady state conditions.

c. Target throughput range should be between 0.2 kg/hr/cm of die circumference and 0.75 kg/hr/cm, with an allowable variance of 10% lower or 25% higher based on specific machine capabilities.
d. The melt will be filtered with a clean 40/60/40 mesh melt filter.
e. Die gap range should be between 0.8mm and 2.0mm. Blow up ratio target is 2.5.
f. Film thickness range for all test samples and control must be between 1.5 and 2.5 mils. Within this range, the actual thickness must not vary more than 20% in either direction from the target range. For example, if the target is 1.5 mils, the allowable range is 1.5 to 1.8 mils.
g. Start run timer after the bubble has been wound and steady state has been reached.
h. Record pressure values every five minutes for a 45-minute run time after a steady state has been reached.
i. Record any occurrence of unusual conditions such as sticking, fumes, odor, or build-up occurring at the feed throat or die exit of the extruder.
j. Retain 5-10 meters of film from this practice step for testing. This film is to be obtained halfway through run time or later at random, as long as steady state is ongoing.
k. Purge the extruder between each run with virgin PE resin. Ensure that no residue is present after purging.
l. Repeat steps 6a through 6f for test variable blends. Extrude test variable blend at same conditions as control blend with a melt temperature delta to control of no more than +/- 20°C. Processing conditions should be the same for control and test within allowable ranges. Any process changes must be recorded and reported.
m. Blown film is the most stringent application and the preferred way to test an innovation or design feature. If a party proposes testing a cast film innovation into a cast film, they should contact APR for a consultation on the required procedures.

Steps Required to Assess Pressure Values

For the control and test material:

After the steady state has been reached, calculate the initial average pressure, P_i, over the first five minutes of extrusion as well as the average pressure value for the final five minutes of extrusion, P_f. When:

$(P_f – P,) / P_i$ this is record as evidence for later determination of screen pack build-up.

Record control and the test graphically to represent the potential for screen pack build up.

Injection Molding ASTM Parts, FPE-P-07

Background

This document presents standard practice to Injection mold PE material into ASTM parts to later be tested for mechanical properties. Not all APR test protocols will require injection molded parts for testing.

Practice Summary

PE materials are dried, and injection molded into ASTM parts for mechanical properties.

Equipment Required

- Dryer capable of drying PE flake and/or pellet at up to 90° C
- Lab Scale injection molding machine allowing no more than 6 minutes of injection barrel residence time.
- ASTM part mold (for Tensile Strength, Flexural Modulus, and/or Izod Impact)
- Mold temperature control equipment

Materials and Reagent Required

- PE material control, consisting of flake or pellet
- PE test material , consisting of flake or pellet

Practice Steps

1. Weigh the required amount of flake or pellet for processing the number of parts required for each control and test material.
2. Dry the flake or pellet in a dryer at 80-90°C for a minimum of 30 minutes and up to 1 hour to remove surface moisture.
3. Starting with the control material, prepare the injection machine to provide:
    a. Target melt temperature at nozzle of 190-245°C depending on primary base resin
    b. Target mold temperature 20-40°C
    c. No more than 6 minutes barrel residence time
4. Injection mold the required number of parts for testing. Processing conditions should be the same for control and test. In the event of process change, it is required to record this change and report.
5. Tested parts to be obtained ½ way through run time or later at random.
6. Purge injection unit with virgin PE resin between samples

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	August 7, 2020	
2	April 27, 2022	Added changes approved by Film Committee: Extrusion temp by DSC (FPE-0-05); more specific instructions on allowable control materials (FPE-0-01); more specific instructions on preparing printed films (FPE-0-02).
3	August 2, 2022	Revised film extrusion temperature range; removed redundant instructions for FAR test