European PET Bottle Platform

Quick Test QT 508

Pressure Sensitive Label

Website version

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This test has been published by the European PET Bottle Platform and was devised by experts in the European plastics, packaging and recycling industries. The test is an indicative test only and is based on the best of our knowledge at the time of publication. It does not necessarily guarantee compliance with the different recycling schemes. Users are therefore advised to discuss the results with the EPBP who will check for specific and up-to-date information.

The European PET Bottle Platform cannot accept responsibility or be held liable for any loss or damage arising out of or in connection with the test results, their accuracy, or incomplete or misleading conclusions.

1. Introduction

The objective of the European PET Bottle Platform (EPBP) is to evaluate technologies and products to allow new PET bottle innovations whilst optimizing the environmental and economic consequences for the recyclability of PET.

EPBP has formulated guidelines to evaluate the influence of bottle innovations - such as barrier materials, resin formulations, additives and non-PET components in or on PET bottles - on R-PET recycling processes. Barrier materials can be applied as a coating, introduced in a co-injected multilayer configuration or blended with the matrix material. Additives can be incorporated into the base material during polymerisation or added during injection moulding in the form of liquid or solid master-batches. Other non-PET components can be labels, glue, sleeves, caps, printings, etc. .

Laboratory analyses on the recyclability of new innovative PET bottles or non-PET components can be relatively expensive and usually have a time delay between sampling and getting useable results of several months. Besides, assessing the test results is a difficult task that requires training and experience. This is often seen as an inconvenience.

For this reason, EPBP has developed a series of rapid and low-cost techniques for the quick assessment of PET bottles. All quick tests include a complete explanation of the scope, techniques, equipment and test conditions, as well as a “summary interpretation” explaining how to use the test results. Quick tests can either be executed at the internal laboratory facility or by an independent test laboratory with minimal investment in equipment.

The results of the quick tests are purely indicative, and may not be considered as an advice, a recommendation or a formal approval by EPBP. For a complete assessment, further tests are required to highlight all possible effects of innovative PET bottles on the recyclability of collected bottles into r-PET, the processing of the r-PET into products and the final product properties. Please contact EPBP for more information.

SAFETY PRECAUTIONS
This guideline is intended for use by qualified personnel who recognize safety hazards and are familiar with the safety precautions required in regard to application of this guideline. The appropriate laboratory safety procedures must be used before, during, and after testing operations.

2. Quick Test QT508

Scope

Several systems can be used to label a PET bottle. The following test protocol is designed to evaluate pressure sensitive labels by providing guidance on the behaviour of the adhesive during the recycling process. The label should detach from the bottle and float and the adhesive layer should remain on the label, with a minimal amount dissolved in the caustic wash and with no adhesive remaining on the washed PET flake.

This test protocol is meant to be carried out in combination with an oven test (see EPBP QT500).

Background

Pressure sensitive labels are applied to PET bottles with light pressure and without activation or heat. The surface of the label intended to come into contact with the bottle is covered with a layer of adhesive that forms a bond when pressure is applied. Labels are typically supplied in rolls, with a release liner that protects the adhesive and assists label handling.

The adhesive should be designed to allow the release of the label during the hot (pre)wash of the recycle process and to allow subsequent sink/float separation of label residuals from PET flakes. The fate of the adhesive itself is very important. Ideally the adhesive should remain on the label when it detaches from the bottle, it should leave no traces on the PET flakes which may lead to discoloration during the processing of recycled PET. The amount of adhesive dissolved into the process water should be reduced to a minimum.

Principle

The following quick test has been designed to assess the behaviour of the adhesive when PET bottles with pressure sensitive labels applied are submitted to a simulation of the recycling process. The test is carried out using the bottles panel only, i.e. the part of the bottle where the label is applied. This allows easier cutting with scissors, without generating fines.

Apparatus

- Digital camera
- Heavy duty (e.g. electrician) scissors
- Lab scale (500 g +/- 0,01 g)
- 250ml and 100 ml crystallizers
- 1000 ml beakers. The beaker internal diameter should be 9-10 cm.
- Hot plate stirrer, or similar equipment capable of heating up to 90°C
- Suitable diameter watch glass to cover beaker when heating
- Clamping device to install overhead 600 rpm stirrer.
- Stirrer impeller. If a magnetic stirrer is used, the magnet should be 4-5 cm long.
- Thermometer
- Caustic soda solution (1.0 % Sodium hydroxide)
- Distilled or deionised water (referred to as “distilled water”, below)
- Ceramic funnel with vacuum filtration

- Manual stirrer (i.e. glass rod)
- Filter paper
- Lab oven with air circulation, with a maximum temperature of 250°C

Short Test Description

Sample panels are cut from PET bottles and weighed on a lab scale with 0.01 g accuracy. The sample pressure sensitive labels are applied to the sample panels, allowing sufficient time for the adhesive to cure. The weight of the label is calculated by difference between the weight of the labelled panel and a control panel without labels. The sample panels (panel plus label) are carefully cut into 10-12 mm square pieces using scissors to avoid generation of fines, and the pieces are transferred to the lab simulation of the recycling process. The test should be carried out with 100% solid coloured labels (preferably red) because this facilitates identification of residual labels glued to PET flakes.

After the process, the floating (label) and the sinking (PET) fraction are collected, dried and weighed. The weight of the sinking fraction should be equal to the weight of the initial panels. There should be no residual label pieces on or around the PET flakes, indicating that all the labels have detached. The weight of the floating fraction should be equal to the initial weight of the labels with the adhesive, signifying that the adhesive remained with the label. If the sum of the weights of the sinking and floating fractions is lower than the initial sample weight, this is an indication that part of the adhesive (or other components) have dissolved into the process water.

The QT508 test is further complemented by an oven test (according to procedure QT500) to be performed on the sinking (PET) fraction, using an unprocessed sample as reference, to better highlight any glue residuals left on the panel. This test is to be documented separately according to the QT500 procedure.

Samples

- PET Bottles
- Test Labels (100% solid coloured, preferably red)

Procedure

- Cut out the panel of the bottle, i.e. the part of the bottle where the label is applied. Each cut panel should be 2-3 mm larger than the label.
- Weigh each panel separately record and add the panel weights until a total weight of 100-200 g (depending on bottle and label size) has been reached. A minimum of 15 bottles and labels should be tested. Record the total weight of the panels as “A”.
- Cut out an additional sample, approximately the same amount of the above sample “A”. This sample is meant to be put aside to be used as reference for the oven test. Cut labelled PET bottle wall panel with scissors in a controlled environment. Avoid generating fines. Flake samples should be approximately square and in the range of 10-12 mm. Mark this sample as “Reference” and put it aside in a clean glass container.
- Report label size and structure, including the density and the amount of adhesive by surface unit (if known from supplier). Document the label colour and design with one or more photographs.
- Apply a test label to each panel.

- Weigh and record each labelled PET panel separately and sum the bottle walls weight. Record this weight as “B”.
- Allow labels to cure at room temperature for 72 hours.
- Weigh and record each labelled PET bottle wall panel separately and sum the bottle walls total weight. Record the weight as “C”. Document sample with one or more photographs.
- Calculate the weight loss during curing and record it as “D”:

$$D = B - C$$

- A significant weight loss during curing “D” is an indication of the presence of volatile compounds in the adhesive. While this is not a parameter that will affect recycling per se, a significant weight loss is worth investigating.
- Calculate the total weight of the labels + adhesive and record it as “E”:

$$E = C - A$$

- Cut labelled PET bottle wall panel with scissors in a controlled environment. Avoid generating fines and the loss of non-labelled flaked sample. Flake samples should be approximately square and in the range of 10-12 mm. Check the weight after cutting to ensure that there are no losses (maximum weight difference +/- 0,02%). Document the sample with one or more photographs. OPTIONAL: count and record the number of flakes.
- Completely submerge the flakes in the caustic solution at 80°C. The ratio between the flakes and the caustic solution should be 4:1, i.e. 400 ml solution for each 100g sample. Switch on stirrer to 500 rpm and leave for 5 minutes. Make sure that the labels do not stick to the stirrer, to the sides of the beaker or to each other, it is important to ensure free agitation of flakes and label particles. Record the process with a series of photographs. Any stickiness should be recorded.
- Observe the caustic solution colour after the test. If any noticeable discoloration, report it and document the caustic solution colour with a photograph. In order to highlight differences, take a sample of the solution (be careful not to include flakes) and place it into an appropriate container. Place alongside a reference sample of fresh (caustic solution in a similar container and take a picture of the two containers against a neutral background. Discoloration may indicate a potential ink bleeding issue. It may be worth considering a further investigation with a label bleeding quick test (see EPBP quick test QT 507 for reference)
- Carefully collect the floating fraction and put it aside in a crystallizer.
- Collect the sinking fraction by filtration on a ceramic funnel connected to a vacuum pump. Use small amounts of distilled water and a suitable tool to collect flakes on the bottom of the beaker and from the stirrer. Do not lose flakes in the transfer process.
- Carefully remove the flakes from the funnel and place them in a beaker containing distilled water at room temperature. The ratio between the flakes and distilled water should be 4:1, i.e. 400 ml solution for each 100 g initial sample. Add the floating fraction that was put aside before. Gently stir the solution manually for about a minute, and allow to settle for 10-15 minutes. Ensure that no flakes or labels remain on the stirrer. When settling has occurred, document with one or more photographs.

- Carefully collect the floating fraction, place it on filter paper to allow gross removal of water and then transfer it into a crystallizer of suitable size. Qualitatively inspect the flakes for residual stickiness of the adhesive.
- Collect the sinking fraction by filtration on a ceramic funnel connected to a vacuum pump. Transfer it on filter paper to allow gross removal of water and then transfer into it a crystallizer of suitable size. Be careful not to lose flakes in the process.
- Dry each fraction in an oven at 80°C for 1 hours.
- Weigh the floating fraction and report the weight as “F”. Document the sample with one or more photographs.
- Qualitatively inspect the floating fraction for residual stickiness of the adhesive.
- If any PET flakes are glued with label pieces in the floating fraction, document them with a photograph, then carefully separate them from the label. Weigh them and report the weight as G.
- If G > 0, then calculate the new weight of the floating fraction as

  $$H = F – G$$

  Otherwise assume H = F
- Weigh the sinking fraction and report the weight as “J”. Document the sample with one or more photographs. Record any labels that did not separate from the PET flake. Take a separate photograph of any labels that did not separate from the PET flakes. Check the flakes surface and verify that it is not sticky.

  OPTIONAL: If flakes have been counted after cutting, count the flakes again and check that the numbers match, i.e. no flake has been lost in the process.
- Pre-heat the oven to 220°C.
- Place the sinking fraction on a tray and place the tray in the oven at 220°C for 1 hour.
- Remove the tray from the oven, allow the sample to cool down and spread it on a clean, white surface. Check for any discoloration that may indicate adhesive residue. Document the sample with one or more pictures. A colour measurement (L-a-b) with a Minolta spectrophotometer is optional.
- Calculate the difference between the initial label and adhesive weight and the recovered floating fraction and report it as “K”

  $$K = E – H$$
- Calculate the ratio between the floating fraction and the initial label and adhesive weight and express it in percentage. Report the value as “L”

  $$L = (F/E) \times 100$$
- If K > 0, then calculate the ratio between the non-recovered floating fraction and the initial label and adhesive weight and express it in percentage. Report the value as “M”

  $$M = (K/E) \times 100$$
- Calculate the difference between the initial panel weight and the recovered sinking fraction and report it as “N”

  $$N = A – K$$

- Calculate the non-recovered (dissolved) weight amount and report it as “P”

$$P = C – (F + J)$$

- • If P > 0, then calculate the ratio between the dissolved fraction and the initial label and adhesive weight and express it in percentage. Report the value as “Q”

$$Q = (P/E) \times 100$$

- Perform an oven test according to procedure QT500, using the sinking (PET) fraction as a sample and the “Reference” sample that was put aside at the beginning of this test as a reference. Perform the test and report the results of the test results following the QT500 test procedure.

Results

The ideal result is the one where J = A and E = H with minor differences, that can be explained with deviations due to experimental setup (e.g. reproducibility in weighting). Such results indicate full label detachment, with the adhesive remaining on the label and no adhesive or other label component dissolved in the washing water.

Any significant deviation from the ideal result will need to be investigated. In particular:

- If J > A and no label residue can be seen, this could be an indication that some residual adhesive is left on the flakes. The oven test carried out on the sinking (PET) fraction should confirm this (refer to EPBP QT500).
- If P > 0, then an amount of sample has been dissolved in the caustic washing water. If no discoloration of the caustic washing solution is observed, this could be either label surface coating (if present) or adhesive.
- In any case, the situation where K > 0 by a value that cannot be explained with the analytical setup is an indication of incomplete sink float separation of the label.
- The fate of the non-recovered label amount should be investigated, by drawing hypotheses based on the test results and, if appropriate, by performing other investigations to get a confirmation. It is important to ensure that the non-recovered fraction is not detrimental to recycling.

Test report

The test report must include the following information:

- Reference to the EPBP Quick test QT508
- A full and complete identification of the material tested.
- Description of the samples before, during and after testing (especially on colour changes, haze, deposits, sinking or non-detached label fragments, residual stickiness, etc.)
- The photographs indicated in the test procedure should be taken and be supplied as electronic files for reference. Photographs should be of reasonable quality, with good lighting and suitable background. Additional photographs are welcome whenever useful for documenting specific situations. To keep the report file to a size reasonable for e-mail circulation, pictures can be resized upon incorporation in the report and repetitive pictures can be omitted, provided the report contains a list of all the pictures taken with file name and picture description the listed photographs are made available as separate JPG files.

- Details of any deviation from the test method, as well as any incident which may have influenced the results
- Test figures. Use the table below as reference.
- Documentation and reporting of the QT500 oven test following the requirements of the QT500 test procedure.
- Date and place of the test.

Remark

This quick test is designed as a quality indicator to monitor a single critical parameter in PET recycling. Other specific tests are needed to carry out a full screening for possible effects of innovation on the recyclability of collected bottles, the processing of r-PET into products and the final product properties. Please contact EPBP for more information.

Table

Variable	Value	Reference in the procedure
Total weight of the panels	X.xx g	**A**
Sample weight before curing	X.xx g	**B**
Sample weight after curing	X.xx g	**C**
Weight loss during curing	X.xx g	**D**
Weight of labels and adhesive	X.xx g	**E**
Weight of the floating fraction	X.xx g	**F**
Weight of residual flakes in the floating fraction	X.xx g	**G**
Corrected weight of the floating fraction	X.xx g	**H**
Weight of the sinking fraction	X.xx g	**J**
Difference between the initial label and adhesive weight and the recovered floating fraction	X.xx g	**K**
Ratio between the floating fraction and the initial label and adhesive weight	X.xx %	**L**
Ratio between the non-recovered floating fraction and the initial label and adhesive weight	X.xx %	**M**
Difference between the initial panel weight and the recovered sinking fraction	X.xx g	**N**
Non-recovered (dissolved) weight amount	X.xx g	**P**
Ratio between the dissolved fraction and the initial label and adhesive weight	X.xx %	**Q**