Protocol

Measurement protocol for the releasability of adhesives in the cold water washing process of rigid PP and PE packaging with plastic labels

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Project: Cold releasable adhesives

Developed by NTCP

Powered by KIDV

Content

1 Introduction

Commissioned by the Netherlands Institute for Sustainable Packaging, The National Test centre of Circular Plastics (NTCP) has created a measurement protocol for the releasability of adhesives in the cold washing process during recycling of rigid plastic packaging. This protocol is designed to assess the behaviour of adhesives on releasability in cold water, simulating a step in the recycling process. It has been established to provide guidelines on how to measure, with a simple procedure, the adhesive residues on plastic packaging after it has been treated by the shredding and washing processes. The plastic packaging is divided into cap, label and container. According to the description of KIDV Recycle Check, the container is the main component of the packaging. The conditions are that the label should be detached from the packaging and that there should be no glue residues on the container. The adhesive layer should preferably remain on the label or otherwise be dissolved in the water. By using this simple procedure, it is not necessary to be assessed by a trained and experienced professional, and a minimum of equipment is required to perform it. This protocol contains a simple detailed procedure that includes an explanation of how to interpret the results and several tables for filling in the key data. This test can be executed in the internal laboratory of the company or in independent test laboratories. The results of the experiments will provide insights into the evaluation and quantification of the effect of the releasability of adhesives (applied for *e.g.* labels) in the washing process with cold water, as well as on the container to which the adhesive is attached. Please, note that the results of these experiments cannot be used as certification. For a full assessment or questions, please contact NTCP.

2 Scope

The scope of this test protocol is the release of glued plastic labels from rigid PP and PE packaging.

In case adhesives are used to attach labels to packaging, recyclers attempt to remove the labels and adhesive from the packaging before it is recycled. For rigid PP and PE packaging with plastic labels this is done by washing the shredded packaging with cold water. Four situations, or combinations thereof, can occur.

1. The label with adhesive remains on the container.
2. The label is released from the container and the adhesive remains on the label.
3. The label is released from the container and the adhesive remains on the container.
4. The label is released from the container and the adhesive dissolves in the washing water.

This protocol is designed to identify and quantify the four situations during the two steps of the label release process:

- Shredding of container material containing glued plastic label material;
- Cold washing of the shredded material.

3 Material

To perform the protocol, the following materials are needed:

- Digital camera
- Heavy duty scissors (*e.g.* metal cutter)
- External micrometer (± 10 µm)
- Ruler
- Laboratory scale (500 g ± 0.5 g)
- Shredder able to shred plastic material to 20 mm flakes (standard industrial size). For example, NTCP uses the Shini granulator model SG-2336E.
- Cylindrical laundry net (net for washing clothing with two nets, with approx. dimensions of height=12.5 cm, diameter=6.5 cm), this net must be filled with approx. 30% of the maximum volume of the net. The inside mesh size should be between 0.5 and 1 mm. The outside mesh side should be bigger than 1 mm.
- Washing machine that can control temperature and water volume (T = 30 ºC and V = 12 L).
- Microscope 250 x magnification
- Packaging with one label applied in a representative way; preferably samples from the production line (30 pcs.).
- Packaging without any label applied on (30 pcs).
- Attached Excel sheet to fill out the obtained data and to execute relevant calculations.

4 Experimental procedure

The protocol was established according to the following steps (See Figure 1), each of these steps will be explained in detail after the list of steps:

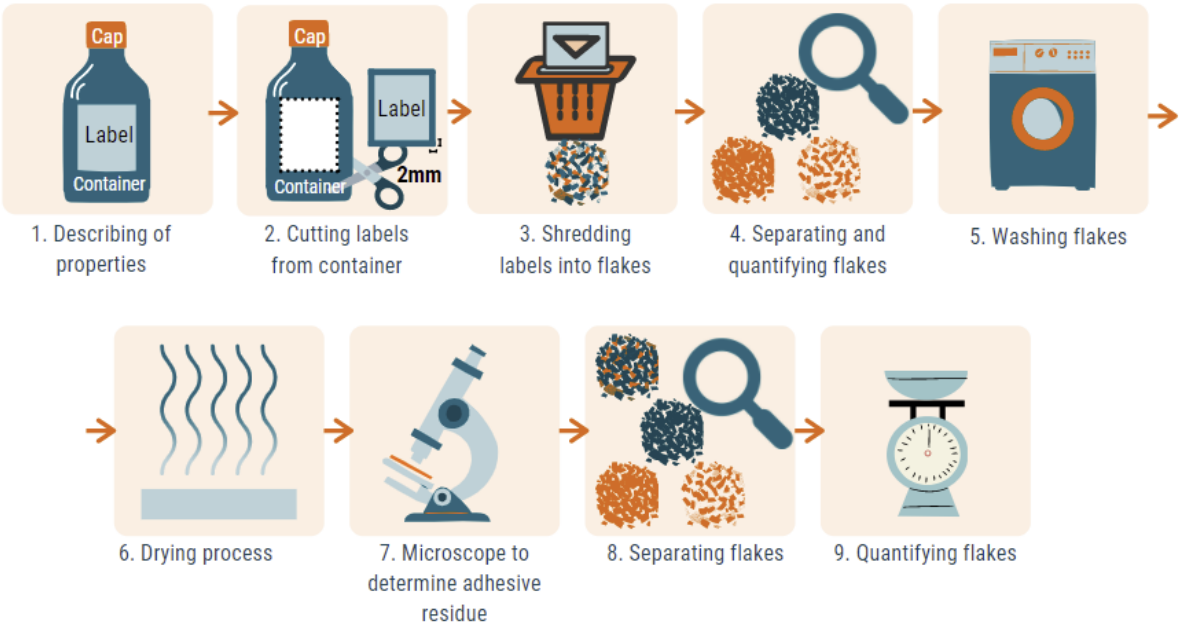


Figure 1 Schematic representation of the label release protocol.

The established protocol mimics the washing step of the recycling process at small scale, from that reason this protocol is established taking into account that the labels are being applied mechanically, not by hand. Test one sample (combination of packaging, adhesive and label) at the time. A detailed description of each step is described below. The obtained data from these steps can be filled out in attached Excel file, which executes the needed calculations automatically.

Step 1. Container properties:

1.1. Determine the packaging properties (see Table 1 in the Excel) by visual inspection and by extracting from the specification sheet (can be requested from the supplier).

1.2. Take a picture of the packaging with the label. Peel off the protective layer of an individual label and take a picture of the side were the adhesive was applied (the adhesive can be applied uniformly in all the surface area or in a form of a pattern).

1.3. Capture the results in Table 1 in the attached NTCP adhesives protocol Excel file.

Step 2. Cut the label from the container:

2.1. Cut the container with the label using scissors or cutters. Cut just around the label and avoid pressing the label onto the packaging (see Figure 2). A minimum amount of 100 g (container + label) is needed in order to realistically shred the material. Note: For reproducibility test, is needed use the same weight per sample.

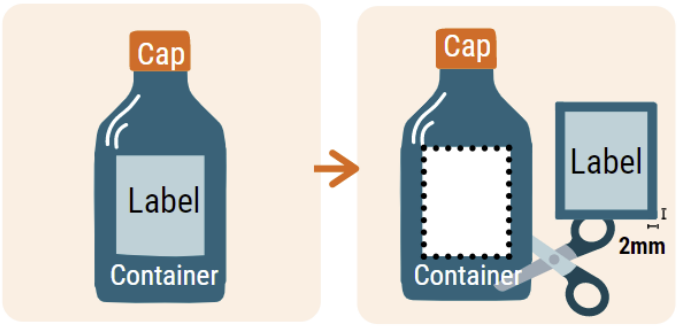


Figure 2 Procedure to cut the container material containing the label.

2.2. Take a picture and weigh the sample of at least 100 g (approx. 25 panels) using a tray and assign the letter (**A**). Note: Do not write on the sample.
2.3. Weigh the cut pieces of the un-labelled containers using a tray and assign the letter (**X**). Note: Do not write on the sample.

Step 3. Shredding process:

3.1. Shred the sample **A** to 20 mm flakes (this size is used as the standard industrial size) in approx. 20 min.
3.2. Weigh and take a picture of the sample after shredding, assign the letter (**H**).
3.3. Collect the material losses from the shredder machine (and/or sieve), weigh these shredding losses and assign the letter (**F**).

Step 4. Flake separation and quantification of the shredding process (Effect of the shredding process):

4.1. Separate and quantify the shredded sample according to Table 2 in the attached NTCP adhesives protocol Excel file.
4.2. Take the collected losses (Letter F) and separate and quantify it according to Table 3 in the attached NTCP adhesives protocol Excel file.

Step 5. Washing process:

5.1. Carefully, put the shredded sample (**H**) into the laundry net. The amount of sample must be 30% of the total net volume (See Figure 2).

Figure 3 30 % filled laundry net.

5.2. Place the sample in a standard washing machine and set the minimum water level and the temperature at T = 30 °C, total washing time (residence time of the sample H in the washing machine) t = 5 minutes.

Step 6. Drying process:

6.1. Carefully remove the flakes from the laundry net and spread them as much as possible on an oven tray. If the container and labels are attached together do not detach by hand.
6.2. Dry the sample at room temperature overnight.
6.3. Take a picture of the sample on the tray and weigh the sample, assign the letter (**Y**).
6.4. Separate and quantify the shredded sample according to Table 4 in the attached NTCP adhesives protocol Excel file.

Step 7. Microscope check:

7.1. Visually inspect the container flakes on residual adhesive. If there is any adhesive on the container flake, this will be quite obvious, and easily recognizable. For instance, the container flakes are attached to pieces of label flakes or it is noticeable that are some rest of adhesive (stickiness) in the container flakes.

7.2. In case of doubt, place the doubtful flakes under the microscope to assess whether any adhesive is still present on the plastic container flake (see Figure 4).

7.3. Take a picture of the microscope measurement of each doubtful flake.

Figure 4 Picture of a sample taken by the microscope.

Step 8. Flakes separation and quantification of the final sample (mass balance closure):

8.1. Separate the sample flakes into the following groups and fill up the numbers in Table 5 in the attached NTCP adhesives protocol Excel file to verify the mass balance.

Step 9. Container flakes quantification

9.1. Use the following formula to quantify the total weight of the container at start assig the letter (**Z**) and fill in Table 6 in the attached NTCP adhesives protocol Excel file..

Threshold: If the percentage of **C is > 90 %** the adhesive is releasable.

5 Test report

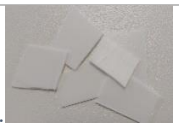
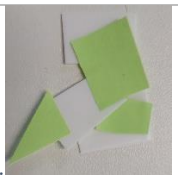
The test report must contain the following information:

1. Date, place and the name of the person/company who performed the protocol.
2. Reference to this protocol (This protocol cannot be used as a certificate).
3. Filled the grey cells in the tables 1, 2, 3, 4, 5 and 6 in the attached NTCP adhesives protocol Excel file. In addition, in the Appendix a Table contains the letters, the definition and, the formula's used in the protocol.
4. The photos in the tables must have a reasonable quality and visibility.
5. Remarks on observations during the process.

Appendix

Letter	Definition	Formula (if relevant)
A	Cut panels (not shred)	A=B+C+D+E+F+G+N+X
B	Clogged flakes **after washing**	
C	Container flakes **after washing**	
D	Label flakes **after washing**	
E	Container + label flakes **after washing**	
F	Losses **after shredding**	F=O+P+Q+R+S+T
G	Fines	G=T+V
H	Sample **after shredding**	H=I+J+K+L+M+N
I	Container + label flakes **after shredding**	
J	Label flakes after shredding	
K	Container flakes **after shredding**	
L	Container with glue flakes **after shredding**	
M	Clogged flakes **after shredding**	
N	Fines after shredding	
O	Container + label flakes losses **after shredding**	
P	Labels flakes losses **after shredding**	
Q	Container flakes losses **after shredding**	
R	Container flakes with glue flakes losses **after shredding**	
S	Clogged flakes losses **after shredding**	
T	Fines losses **after shredding**	
U	Container with glue flakes **after washing**	
V	Fines **after washing**	
W	Container with glue flakes	W= U+L
X	Other losses	
Y	Total **after washing**	Y=B+C+D+E+U+V
Z	Total weight container at start	Z= A – P – D + R Z = C+ W + E + G + a
a	Other losses after washing	

Table 1 Example of material classification

Quantification of the flakes type after shredding	*Letter reference in the procedure*	*Picture*
Shredded sample	*H*	*i.e.* 
Container + labels flakes	*I*	*i.e.* 
Label flakes	*J*	*i.e.* 
Container flakes	*K*	*i.e.* 
Container with glue flakes	*L*	*i.e.* 
Clogged flakes (flakes that are stuck together)	*M*	*i.e.* 
Fines	*N*	*i.e.*