



# Harmonised European laboratory test method to generate parameters enabling the assessment of the recyclability of paper and board products in standard paper and board recycling mills

**Short title: CEPI recyclability laboratory test method**

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## 1. INTRODUCTION

The paper and paper board value chain is an example for circularity, displaying very high recycling rates. Moreover, technical innovation, is creating new products from paper and -board materials and other cellulose fibre-based products that are increasingly replacing other traditional packaging materials.

To maintain and further increase the sustainability and circularity of the paper and board value chain and to help EU Member States and other European countries meet high recycling targets<sup>1</sup>, it is important to ensure that paper and board-based materials and other cellulose fibre-based products (e.g. moulded fibre products) are recyclable by the paper industry. The paper manufacturing and converting industry has issued joint guidance on paper-based packaging recyclability<sup>2</sup> at national and European levels. To confirm recyclability, it is necessary to define a harmonised test method as a basis for assessing the general recyclability of these materials and products.

The harmonised test method emulates the most common phases of the industrial processes to measure the main parameters of recyclability of paper and board-based materials and other cellulose fibre-based products based on current knowledge and technology.

This makes it possible to:

- Supplement the evaluation of recyclability required by EN 13430 with regard to paper and board-based materials and other cellulose fibre-based products that are sent for recycling in the paper industry.
- Guide eco-design, in terms of recyclability, of paper and board-based materials and other cellulose fibre-based products currently in use, as well as new materials under development and additives used in the converting phase that can affect the recyclability of the final product.
- Support declarations related to the recyclability of materials or products based on grading systems developed by third-party organizations.

## 2. SCOPE AND FIELD OF APPLICATION

This document describes a laboratory scale method for determining the key parameters for evaluating the level of recyclability of paper and board-based materials and other cellulose fibre-based products, e.g. moulded fibre products, emulating the relevant phases of standard paper and board recycling mills without deinking technology<sup>3</sup> or other special features to recycle paper for producing new paper and board.

This method enables analysing both process parameters (coarse reject, fine reject, dissolved and colloidal substances and stickie particles with a diameter smaller than 2 mm) and quality parameters (sheet formation and interfering materials like

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<sup>1</sup> E.g. directives 2018/851/EU, 2018/852/EU set high recycling targets for municipal waste and paper-based packaging (85% by 2025, 90% by 2030).

<sup>2</sup> Cepi, FEFCO, Citpa, ACE: Paper-based packaging recyclability guidelines. 2019. Confederation of Paper Industries: Paper and Board Packaging Recyclability Guidelines, Revision One, Published January 2020.

<sup>3</sup> EPRC: Assessment of printed product recyclability: Deinkability score User's manual, [www.paperforrecycling.eu](http://www.paperforrecycling.eu)

adhesiveness and visual impurities) of products produced from recycled fibres. This document considers only the minimum characteristics of paper and board products that can be generally recycled. Therefore, it does not take into consideration additional specifications necessary to valorise the paper and board products using deinking technologies. It also does not include parameters of recyclability in mills with specialised processing technology.

### 3. NORMATIVE REFERENCES

This document incorporates, by way of dated or undated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed below. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated into it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 643	Paper and board - European list of standard grades of paper and board for recycling
DIN EN 12231-11	Food processing machinery - Mincing machines - Safety and hygiene requirements
ISO 15 360	Analysis of macro stickies in pulps
ISO 5 263-1	Pulps - Laboratory wet disintegration - Part 1: Disintegration of chemical pulps
ISO 5 269-2	Pulps - Preparation of laboratory sheets for physical testing Part 2: Rapid-Koethen method
ISO 1762	Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 525 °C
ISO 15 360-2	Recycled pulps — Estimation of Stickies and Plastics — Part 2: Image analysis method
ISO 21 993	Paper and pulp — Deinkability test for printed paper products
UNI 11 743	Paper and board - Determination of parameters of recyclability of cellulose-based materials and products
ISO 638-1	Paper, board and pulps — Determination of dry matter content by oven drying method — Part 1: Solid materials
ISO 638-2	Paper, board and pulps — Determination of dry matter content by oven-drying method — Part 2: Suspensions of cellulosic nanomaterials
ISO 6060	Water quality: determination of the chemical oxygen demand.
ISO 4119	Pulps – Determination of stock concentration
ISO 4046	Paper, board, pulps and related terms – Vocabulary – Part 1
EN 13430	Packaging – Requirements for packaging

	recoverable by material recycling
ISO 21993	Paper and pulp: Deinkability test for printed paper products ISO 4119 Determination of stock concentration of pulps
TAPPI/ANSI T 275	Screening of pulp (Somerville-type equipment)

#### 4. TERMS AND DEFINITIONS

While the term “paper” is defined by ISO 4046, the following terms and definitions are applicable for the purpose of this standard (note: the scope of the standard includes paper and board-based materials and other cellulose fibre-based products like moulded fibre products).

Paper and board: a web comprising substantially (at least 50 %) of cellulosic fibres forming hydrogen bridge bonds, which may contain fillers and coatings. Moulded fibres are also included in this definition.

Cellulose fibre-based products: Finished objects (such as packaging, printed materials, articles for domestic use, etc.) comprised of over 50 % (in weight) of paper and board.

Paper and board for recycling (EN 643): Natural fibre-based paper and board suitable for recycling and consisting of:

- paper and board in any shape.
- products made predominately from paper and board, which may include other constituents that cannot be removed by dry sorting, such as coatings, laminates, spiral bindings, etc.

Use of paper and board for recycling: This refers to the processes used in the recycling of paper and board in the paper industry. These processes include mainly the pulping of paper and board for recycling, the separation of non-cellulose components, and the cleansing of the recovered pulp. Different types of equipment are used depending on the type of paper and board to be recycled and the end product requirements.

Recyclability: Ability to be treated in a recovered paper treatment plant according to recognised rules of engineering so as to ensure that the secondary fibre furnish allows the undisturbed and cost-effective manufacture of a recycled fibre-based new paper of acceptable quality.

## 5. PRINCIPLE

The recyclability of materials or products made predominately from paper and board is determined by means of laboratory procedures that emulates the most relevant industrial phases in standard paper and board recycling mills dedicated to the recycling of paper and board. In particular, this method defines the parameters of interest for the verification of recyclability as follows:

- Ease at which the fibres can be separated using the standard process and equipment.
- The potential to form sheets out of the recovered fibres without significant disruption.
- The visual appearance when formed into sheets.
- The level of coarse and fine rejects.
- The level of fragmentation of disrupting materials (adhesives, metals, plastic film).
- The level of or colloidal solids below 10 microns resulting from non-paper components in the tested sample.

A flowchart showing the different phases of the method is shown in annex A.

## 6. APPARATUS

- 6.1 Aluminium trays for the determination of the evaporation residue.
- 6.2 Analytical balance with accuracy of  $\pm 0.01$  g
- 6.3 Barrels
- 6.4 Beakers
- 6.5 Black water-based ink (e.g. Pelikan No. 4001) compliant with ISO 15 360 (optional)
- 6.6 Metal plates (brass or steel) for adhesiveness test with pressure 1.18 kPa (or weighing 3.7 kg, 20 cm diameter)
- 6.7 Metal plates (brass or steel) for macro stickies test with pressure 0.950 kPa (or weighing 6 kg, 28 cm diameter)
- 6.8 Buechner funnel porcelain complaint with DIN EN 12331, with diameters of 125 mm and 150 mm and equipped with a suction flask and a water jet pump
- 6.9 Carrier boards and cover sheets
- 6.10 COD cuvette tests (e.g. ranges 15-150 mg/l O<sub>2</sub> and 150-1,000 mg/l O<sub>2</sub>) (optional)
- 6.11 Couching roller
- 6.12 Corundum powder compliant with ISO 15 360 (optional)
- 6.13 Cuvette heating block (e.g. Lange LT 20 Temp. 150  $\pm$  5 °C) (optional)
- 6.14 Cuvette hack (optional)
- 6.15 Digital Thermometer
- 6.16 Eppendorf variable pipette 1,000 – 5,000  $\mu$ l (optional)
- 6.17 Filter paper grade 388, with diameter 125 mm – 150 mm (basis weight 84 g/m<sup>2</sup>, filtration speed 10 s/10 ml, deposition range 12 – 15  $\mu$ m)
- 6.18 Filter paper grade 388, with diameter 150 mm (basis weight 84 g/m<sup>2</sup>, filtration speed 10 s/10 ml, deposition range 12 - 15  $\mu$ m)

- 6.19 Filter paper grade 1289 diameter 240 mm (basis weight 84 g/m<sup>2</sup>, filtration speed 20 s/10 ml, deposition range 8 – 12 µm) (optional)
- 6.20 Forced air oven able to maintain the required temperatures (60 °C and 105 °C) with accuracy of ± 2 °C
- 6.21 Image analysis system comprising:
- Scanner (e.g. EPSON V-750 PRO) with minimal optical resolution of 2000 dpi
  - Software for analysing area and size distribution of adhesive particles (macro stickies), complaint with ISO 15360-2<sup>4</sup>
- 6.22 LED light panel
- 6.23 Photometer measuring device (e.g. Hach Lange) (optional)
- 6.24 Rapid Koethen sheet former, complaint with ISO 5269-2
- 6.25 Silicon paper (60 g/m<sup>2</sup>) compliant with ISO 15 360 (optional)
- 6.26 Somerville-Fractionator complaint with TAPPI/ANSI T275, equipped with one perforated screen plate with 5mm hole diameter (for details see annex B) and one slotted plate with 0.15mm wide slots (for details see TAPPI/ANSI T275)
- 6.27 Standard disintegrator complaint with ISO 5263-1
- 6.28 Vacuum desiccator

## 7. PREPARATION OF SAMPLES

The quantity of tested material or product must be sufficient to carry out all the measurements indicated by the method. An indicative quantity is 250 g air-dry weight.

Perform a double determination of the moisture content of the product or material in compliance with ISO 638.

Weigh one aliquot of air-dried material or product, with a precision of ± 0.01 g corresponding approximately to (50 ± 1) g dry weight.

If the tested product weigh less than 50 g, weigh an additional fraction of product in order to obtain a sample with a total aliquot of 50 g dry weight.

If the product or material weights more than 50 g, it is necessary to ensure that the sample contains the same proportion of elements different from the base product or material (e.g. labels, seals, hot-melt application, metallisation, ink application, etc) as the tested product or material.

Any relevant information allowing a correct and proportional sampling must be present in the technical data sheet provided with the sample. The technical data sheet must contain the minimum content of information indicated in the Annex F.

Cut the sample into pieces of 3 cm x 3 cm (± 0.5 cm) in size.

To avoid problems with the functioning of the disintegrator, easily-removable non-paper components, like metal clips and parts of rigid plastic material, can be removed and weighed separately from the rest of the sample. The weight of non-paper components should not be included in the 50 g oven dry material for disintegration, but in sum of total constitutes the coarse reject.

All sample quantities indicated hereinafter refer to the calculated dry weight of matter dried in an oven at (105 ± 2) °C.

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<sup>4</sup> The PTS DOMAS or Techpap SIMPALAB software or equivalent are considered suitable.

If the material or product contains water strength agents (WSA) and has been produced less than 30 days before the test, it has to be stored for the remaining time needed to reach 30 days from the date of production. Alternatively, it can be aged at  $60^{\circ} \pm 1$  C for 72 hours. The accelerated aging is needed to mimic the natural aging of the material between production and recycling where for example a post curing of wet strength agents may still happen. In case of materials and products without WSA, make sure the sample is at least 15 days old from the date of production, therefore no aging is necessary.

For more details about the sample preparation, like complex sample preparation and photo documentation, check the detailed work description in annex E.

## 8. PROCEDURE

The method comprises the following phases:

- Disintegration;
- Filtrate Analysis;
- Determination of the 5 mm hole residue (Coarse Reject);
- Determination of the consistency after the coarse screening (AC);
- Sheet adhesion test and visual appearance test of the accept of the coarse screening;
- Determination of the 150  $\mu$ m slot residue (Fine Reject);
- Sheet adhesion test and visual appearance test of the accept of the fine screening
- Determination of the content of adhesive particles (macro stickies);

### 8.1 Disintegration

This section entails the sample disintegration and preparation of the total stock for the subsequent analyses.

Disintegrate approximately precisely ( $50 \pm 1$ ) g oven dry sample using the disintegrator compliant with ISO 5263-1, but diluting the sample with tap water at ( $40 \pm 1$ ) °C, the pH mildly alkaline (7 – 8). The total volume of sample and water must be approximately 2,000 g, so that a stock consistency of 2.5 % is achieved. No pre-wetting or soaking of the sample must be done. The disintegration time is 10 min (30,000 revolutions).

The disintegration time can be prolonged to 20 minutes, in a second batch under the same conditions, when the coarse reject of the sample contains a significant amount of fibrous material and the coarse reject amount is above the threshold defined in the evaluation protocol of the CEPI Test Method version 2 - Part I. In this case, the results of the measurement taken after 10 min and 20 min disintegration must be included in the Test Report.

For more detailed information about the disintegration, check the detailed work description in annex H.

### 8.2 Filtrate Analysis

This section describes the determination of the evaporation residue and, optionally, the chemical oxygen demand in the filtrate of the total stock.

Perform the filtration of the total stock immediately after the disintegration. Homogenise the total stock and filtrate a known amount of it (approximately 100 g) over a filter paper (diameter 150 mm) using the



Büchner funnel, if possible, without moisturising the filter paper. Use the filtrate to rinse the suction flask and return the filtrate and the filter cake to the total stock.

Filter more 200 g of total stock, retrieve the filtrate from the suction flask and filter it again using the same paper filter. Save the filtrate for the further procedures and return the filter cake to the total stock.

For more detailed information about the filtration, like the photo documentation of the filtrate, check the detailed work description in annex H.

### 8.2.1 Evaporation residue

After the filtration proceed immediately to the determination of the evaporation residue. Pour a known amount (approximately 70 g) of filtrate on a previously weighed aluminium tray and dry it in the oven at  $(105 \pm 2) ^\circ\text{C}$  until reaching a constant mass in compliance with ISO 638. Repeat the procedure to have a double determination of the evaporation residue.

If necessary, store the remaining filtrate in the refrigerator at  $4 ^\circ\text{C}$  it for further analyses.

Determine the evaporation residue of the tap water used in the disintegration.

The evaporation residue (ER) in (g residue / kg packaging) is calculated as follows:

$$ER \text{ filtrate (g residue/g filtrate)} = \frac{m3 (g) - m1(g)}{m2 (g)}$$

$$ER \text{ sample (g residue/g filtrate)} = ER \text{ filtrate (g/g)} - ER \text{ tap water (g/g)}$$

$$\frac{ER \text{ sample (g/g)}}{Packaging \text{ mass (g/L)}} = ER \text{ sample (L/g)}$$

$$ER \text{ sample (L/g)} = ER \text{ sample (kg/g)}$$

$$ER \text{ sample (kg/g)} \times 1,000,000 = ER \text{ sample (g residue/kg packaging)}$$

m1 = mass of the empty aluminium tray

m2 = mass of the filtrate that was taken in

m3 = mass of tray after drying

### 8.2.2 Chemical oxygen demand – COD (optional)

Perform a double determination of the chemical oxygen demand immediately after the filtration or until 24 h after it, if the filtrate is stored in the refrigerator.

Select the COD cuvette with the expected measuring range for the tests according to ISO 6060.

Following the cuvette test instructions, homogenise the cuvette solution and the filtrate, then pipette the volume of filtrate required for the test range into the cuvette.

If the cuvette solution immediately presents a green coloration, discard the sample and either use a higher measuring range or dilute the filtrate with deionised water.

Invert the cuvettes to stir the preparation and heat them in the heating block for 2 h at  $148 ^\circ\text{C}$ . Then, invert the cuvettes again and cool them to room temperature in the cuvette rack. Lastly, measure the COD using a photometer with absorbance of 600 nm.

Determine the COD of the tap water used in the disintegration.

The COD of the sample in (g O<sub>2</sub> / kg packaging) is calculated as follows:



$$COD\ sample\left(\frac{mg\ O_2}{L}\right) = COD\ filtrate\left(\frac{mg\ O_2}{L}\right) - COD\ tap\ water\left(\frac{mg\ O_2}{L}\right)$$

$$COD\ (g\ O_2/kg\ Packaging) = \frac{COD\ sample\left(\frac{mg\ O_2}{L}\right)}{Packaging\ mass\left(\frac{g}{l}\right)}$$

### 8.3 Determination of the 5 mm hole residue (Coarse Reject)

This section entails the determination of the coarse reject from the total stock according to the TAPPI/ANSI T 275.

Proceed with the coarse screening using the Somerville fractionator equipped with a perforated plate containing holes with 5 mm of diameter and set with a water flow of  $(8.6 \pm 0.2)$  l/min. The required characteristics of the referred plate are set out in annex B.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the total stock (including the filter cake mentioned in the filtrate analysis). Perform the coarse screening for 5 min, starting to counting the time when the sample starts to overflow the weir.

Collect the accept of the coarse screening in a specific container to be used for the subsequent procedures: determination of the consistency, sheet formation, fine screening and macro stickies determination (optional).

On completion of the test, transfer all the reject remaining on the plate to a specific container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the holes are also recovered and added to the reject.

Filter the reject over a rapid paper filter with 125 mm of diameter previously calibrated in the oven at  $(105 \pm 2)$  °C using a Büchner funnel. Then, place the filter paper between two cover sheets and pre-dry it in the dryer of the sheet former. Finally, dry the filter paper in the oven at  $(105 \pm 2)$  °C, until reaching a constant mass according to ISO 638.

Calculate the dry weight of the coarse reject, net of the weight of the paper filter, and express the result as a percentage with respect to the dry weight of the starting sample. The results can be rounded to the first decimal place.

For more details like the photo documentation of the coarse reject check the detailed work description in annex H.

### 8.4 Determination of the consistency after the coarse screening (AC)

This section entails the determination of the stock consistency according to EN ISO 4119 (stock consistency between 0.3 % and 1 %).

Homogenise the accepted fraction of the coarse screening and transfer a known amount of it (approximately 500 ml) into the tared beaker.

Filter the 500 ml of the accept over a rapid paper filter with 125 mm – 150 mm of diameter previously calibrated in the oven at  $(105 \pm 2)$  °C using the Büchner funnel. Then, place the filter paper between two cover sheets and dry each side of it in the dryer of the sheet former for 7 min  $(93 \pm 4)$  °C. Leave the filter paper cool down in the desiccator before weighting it.

Determine the dry mass of the filter cake as follows:

$$c (\%) = \frac{m_3 (g) - m_2 (g)}{m_1 (g)} \times 100$$

$m_1$  = mass of sample before drying

$m_2$  = mass of filter paper without sample

$m_3$  = mass of sample with filter paper after drying

### 8.5 Sheet adhesion test and visual appearance test of the accept of the coarse screening

This section entails checking the adhesiveness and visual appearance of the sheets produced from the accepted fraction of the coarse screening.

After homogenising the accepted fraction, take a sufficient amount of pulp (approximately 2,000 g) to form two hand sheets with  $(60 \pm 2)$  g/m<sup>2</sup> (corresponding to approximately 1.8 g in dry weight for each sheet). If the target weight  $(60 \pm 2)$  g/m<sup>2</sup> is not reached, adjust the amount of pulp required for sheet formation.

Dry the sheet in the Rapid-Koethen sheet former according to the ISO 5269-2. Next, place the lab sheet between a carrier board (bottom side) and a cover sheet (top side) and pre-dry them into the dryer of the sheet former for 7 min  $(93 \pm 4)$  °C. Then, dry them, without removing the support and cover, in the oven at  $(130 \pm 2)$  °C between two preheated brass plates (pressure of 1.18 kPa or 3.7 kg) for 2 min. Finally, cool them down in the desiccator for approximately 10 min.

Place the sheet between two metal plates at the same temperature, applying a pressure of 1.18 kPa (3.7 kg) on the entire surface of the sheet for 2 minutes. Then, perform the sheet adhesion test immediately after taking the lab sheet out of the desiccator by separating the cover sheet or carrier board from the it and assessing any damage or breakages in oblique light.

Express the result by assigning a rating based on the following scale:

- Adhesives absent: the sheet can be separated completely from the support and cover without any damage or breakages. Traces of fibres on the support and/or on the cover are permitted. Fragments of paper on the support and/or on the cover are not permitted;
- Adhesives partly present
- Adhesives present: the sheet does not conform to the definition of adhesives absent.

Example pictures of the three categories are shown in the Annex E.

Visually assess the quantity and type of visual appearance present on both sides of the sheets.

If there are any visual impurities, assign a rating through comparison with the decision tree provided in annex C.

For more details like the photo documentation of the sheet adhesion test and visual impurities check the detailed work description in annex H.

## 8.6 Determination of the 150 µm slot residue (Fine Reject)

This section entails the determination of the fine reject present in the accept after the coarse screening.

Proceed with the fine screening using the Somerville fractionator equipped with a plate containing slots with 0.15 mm of width and set with a water flow of  $(8.6 \pm 0.2)$  l/min.

Homogenise the accept from the coarse screening and take an aliquot of 20 g oven-dry for the fine screening.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the 20 g oven-dry accept. The pouring time should not be longer than 4 min. Perform the fine screening for 20 min, starting to counting the time when the sample starts to overflow the weir. During the screening, collect at least the first 50 l of the sorted material to be used for assessing the second adhesive test and visual impurities.

If possible collect the total amount fine screening accept and work with a thickener to do so. Use the collected and thickened pulp to perform the sheet adhesion test and the assessment of visual impurities. Description of the Thickener in the Annex D. The use of the entire fine screening accept material is preferred for reasons of representativeness.

On the completion of the test transfer all the reject remaining on the plate to a specific container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the slots are also recovered and added to the reject.

Filter the reject over a rapid paper filter with 125 mm – 150 mm of diameter previously calibrated in the oven at  $(105 \pm 2)$  °C, using a Büchner funnel. Then, place the filter paper between two cover sheets and pre-dry it in the dryer of the sheet former. Finally, dry the filter paper in the oven at  $(105 \pm 2)$  °C, until reaching a constant mass according to ISO 638.

Calculate the dry weight of the fine reject, net of the weight of the paper filter, and express the result as a percentage with respect to dry weight of the accepted pulp aliquot used for the test. The results can be rounded to the first decimal place.

For more details like the photo documentation of the coarse reject, check the detailed work description in annex H.

## 8.7 Sheet adhesion test and visual appearance test of the accept of the fine screening

This section entails checking the adhesiveness and visual appearance of the sheets produced with the accepted fraction of the fine screening.

After homogenising the accept, take a sufficient amount of pulp (approximately 9,000 g) to form two hand sheets with  $(60 \pm 2)$  g/m<sup>2</sup> (corresponding to approximately 1.8 g in dry weight for each sheet) in the Rapid-Koethen sheet former according to the ISO 5269-2. If the target weight  $(60 \pm 2)$  g/m<sup>2</sup> is not reached, adjust the amount of pulp required for sheet formation.

Dry the sheet in the Rapid-Koethen sheet former according to the ISO 5269-2. Next, place the lab sheet between a carrier board (bottom side) and a cover sheet (top side) and pre-dry them into the dryer of the sheet former for 7 min  $(93 \pm 4)$  °C. Then, dry them, without removing the support and cover in the oven at  $(130 \pm 2)$  °C between two preheated brass plates (pressure of 1.18 kPa or weighting 3.7 kg) for 2 min. Finally, cool them down in the desiccator for approximately 10 min.

Perform the sheet adhesion test immediately after taking the lab sheet out of the desiccator by separating the cover sheet or carrier board from the it and assessing the any damage or breakages in oblique light.

Express the result by assigning a rating based on the following scale:

- Adhesives absent: the sheet can be separated completely from the support and cover without any damage or breakages. Traces of fibres on the support and/or on the cover are permitted. Fragments of paper on the support and/or on the cover are not permitted;
- Adhesives partly present
- Adhesives present: the sheet does not conform to the definition of adhesives absent.

Example pictures of the three categories are shown in the Annex E.

Visually assess the quantity and type of visual appearance present on both sides of the sheets.

If there are any visual impurities, assign a rating through comparison with the decision tree provided in annex C.

For more details like the photo documentation of the sheet adhesion test and visual impurities check the detailed work description in annex H.

### 8.8 Measurement of adhesive particles - macro stickies (optional)

This section entails evaluating the quantity of adhesive particles (so-called macro stickies) present in the accepted fraction of the coarse screening. This section is not required if the technical data sheet states that there are no adhesive substances in the paper and board material or product.

Proceed with the measurement of macro stickies according to ISO 15360-2.

Perform the screening in the Somerville fractionator equipped with the 150 µm perforated plate for 10 min and set to work with a water flow of  $(8.6 \pm 0.2)$  l/min.

Homogenise the accept from the coarse screening and take two aliquots of 5 g oven dry pulp for the macro stickies determination. If the accept material is not enough to complete the test, the steps 8.1, 8.3 and 8.4 must be repeated to recover sufficient material.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the first aliquot of 5 g equivalent oven-dry of the accept. Perform the fine screening for 10 min, starting to counting when the sample starts to overflow the weir.

On completion of the test, transfer the reject on the plate in a specific container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the slots are also recovered and added to the reject.

Place a labelled filter sheet (grade 1289 diameter 240 mm) on the Rapid Koethen sheet former, moisten and smooth it. Then, pour the reject in the Rapid Koethen sheet former to aspirate the water from it. Ensure that any particles adhered to the sheet former column are recovered and added to the reject.

If there is a high content of stickies on the filter paper, so that they overlap each other, perform the screening step again with a lower sample amount or increase the sorting time.

Each deviation (reduction of the sample quantity/change of the sorting time) must be documented.

Carefully remove the filter from the sheet former, place it between carrier board (bottom side) and a silicon paper (siliconized side in contact with the stickies). Dry it in the dryer of the sheet former for 10 min  $(93 \pm 4)$  °C.

Pour the black ink onto a plate and dip the sheet into the ink. Place the dyed filter sheet between a carrier board (bottom side) and a silicon paper (top side). Dry it in the dryer of the sheet former for 10 min  $(93 \pm 4)$  °C.

Pulverise the sticky side of the filter sheet with corundum powder and place the filter sheet between the carrier board and silicon paper. Dry the sample between two preheated metal plates (pressure of 0.950

kPa or weighing 6 kg) for 10 min in a drying oven at  $(105 \pm 2)$  °C.

Remove the excess of corundum powder and check if the stickies are overlapping each other or if there are other sources of contamination. If so, either remove the hydrophobic contamination (e.g. plastic pieces) by hand using tweezers or colour them black using a permanent marker.

Perform the same procedure with the second aliquot of 5 g oven dry.

Use an image analysis software (e.g. PTS-DOMAS multispec) to measure the amount and size distribution of the adhesive particles.

According to the image analysis software used, set the dimensional limits of the particle classes at a minimum of 0.1 mm and a maximum of 2 mm in equivalent diameter.

The size distribution of particles (macro stickies) with diameter smaller than 2 mm must be given in mm<sup>2</sup> per kg of sample.

Calculate the average and the deviation of the size distribution of the adhesive particles (macro stickies) measured in the different repetitions, rounding the result to the nearest ten.

## 9. TEST REPORT

The test report must include at least the following information:

- a) Reference to this standard;
- b) Description of the material or product specifying the following:
  - Reference of the tested sample (product name or number), reference to the technical data sheet, production date of sample
  - A summary of the main information included on the sample data sheet (grammage, materials and shares in case of multilayer materials, adhesives, sealing, printing, metallisation, accessory components or other specific features useful to identify the sample)
  - Specific sample preparation, if any (e.g. emptying, removal of manually separable accessories intended to be removed before disposal)
  - Finished product or intermediate (component/ constituent)
    - i. Semi-finished - **sheets** of packaging material / substrate (paper, cardboard, solid board, corrugated board)
    - ii. Semi-finished - **sheets** of packaging material / substrate with “upgrading” (polymer/metal coating, print, varnish)
    - iii. Finished product - intermediate, not yet ready to be used
    - iv. Finished product - ready to be used
    - v. Finished product - used
  - Type of software used for image analysis
  - Photographic documentation of the material or product and its specimen during the testing, using transmitted and oblique light. See Annex H for more details.
  - Results of the test expressed in compliance with the criteria established in respectively paragraphs:
    - Coarse reject, expressed as a percentage, description of fibres/non-fibres and fragmentation

- Dissolved and colloidal substances, expressed as a percentage
  - Adhesiveness of the accept of the coarse screening, expressed as "absent", "partly present" or "present" and visual appearance, expressed with a level rating
  - Fine reject expressed as a percentage, description of fibres/non- fibres and fragmentation
  - Adhesiveness of the accept of the fine screening, expressed as "absent", "partly present" or "present" and visual appearance, expressed with a level rating
- Date and place of the test
  - Any deviation from the specified test procedure
  - In the event that it has not been possible to perform all steps of the test method in accordance with this standard or it is not possible to determine one or more measurement parameters due to the nature and/or characteristics of the sample material or product, the circumstance must be reported by the laboratory in the test report

#### Examples:

- Pulping resistance prevents the disintegrator from working or there is a risk of damage to the equipment
- Presence of dense flakes or foams prevents the transfer of the accept to the next stage
- Presence of metal particles or wet resistant resins distorts the assessment of macro stickies

Additionally, the test report may include the following information:

- a) An indication of the adhesive particles (macro stickies) content expressed as a total area (including those greater than 2 mm equivalent diameter), expressed as mm<sup>2</sup> of the macro stickies' area per kg of the sample as-is, in compliance with the ISO 15360-2 standard
- b) An indication of the ash content of the paper product or material determined in compliance with the ISO 1762 standard
- c) Results of the test expressed in compliance with the criteria established respectively in paragraphs 8.5 and 8.7 (adhesiveness, expressed as "absent", "partly present" or "present" and optical inhomogeneity, expressed with a level rating of an additional adhesiveness and optical inhomogeneity) performed on the accept of coarse screen phase
- d) Any specific comments, such as:
  - specific observations, e.g. changes in consistency after disintegration, long drainage time during sheet formation, foam formation
- e) Further photographic documentation regarding the results, as detailed in point f)

**Table of annexes (see separate annex document)**

A) Flowchart	page 2
B) Description of the plate for evaluation of the coarse reject	page 4
C) Decision tree for the evaluation of the visual appearance	page 6
D) Description of possible Thickener	page 8
E) Sheet adhesion test reference pictures of the carrier board after sheet adhesions test	page 9
F) Technical data sheet	page 12
G) Laboratory report template	page 14
H) Detailed work description	page 14