

Introduction

A good recyclability of printed products is a crucial feature for the sustainability of the graphic paper loop. It belongs to the focus of INGEDE activities to safeguard and improve recyclability.

One of the measures is to provide tools for the assessment of the recyclability in the two aspects:

- Deinkability
- Screenability of adhesive applications

Therefore a set of methods was developed which simulate unit operations of a deinking plant and allow conclusions about the behaviour of a printed product and the adhesive applications in a deinking plant.

This procedure deals with the fragmentation behaviour of adhesive applications after pulping as one aspect of recyclability assessment. The method is based on the general requirement that it should be possible to separate adhesive applications mechanically. The fragmentation behaviour determines the screenability (see ERPC Scorecard “Assessment of Print Product Recyclability – Scorecard for the Removability of Adhesive Applications”).

1 Scope

This INGEDE method describes a procedure for testing the fragmentation behaviour and screenability of adhesive applications on paper products. It is suitable for known and for unknown amounts of adhesives in the recycled paper sample.

2 Terms and definitions

Macrostickies:

ZELLCHEMING Technical Leaflet RECO 1, 1/2006 “Terminology of Stickies”, determined by means of INGEDE Method 4.

Stickies is the term for adhesive (tacky) particles that occur when recycled fibres are utilised. Macrostickies is commonly the term for the tacky residues on the screening plate after a fractionation.

Adhesive Applications:

Adhesive spine

Are the adhesive back binding of printed books, magazines, journals and catalogues.

Side glue

One or two pages at the front and one or two pages at the back side of a printed product are part of the binding. The adhesive spine and the side glue form together the adhesive binding.

Glued-in inserts

These are adhesive applications to glue samples or leaflets mostly for commercial purposes into or at printed products.

PSA

Is the abbreviation for pressure sensitive adhesives, typically used for labels and stickers.

3 Principle

This method is determined to simulate the screening ability of adhesive applications in a deinking process. The two essential process steps are pulping and screening.

This method describes the laboratory pulping process by defining the physical conditions and the addition of deinking chemicals (Figure 1).

The separation of adhesive applications from the pulp is done by screening according to INGEDE Method 4.

The particle size distribution of the macrostickies is measured, thus allowing the assessment of the screening ability of the adhesives application in an industrial process.

The setting of the screening ability limit of < 2 000 µm equivalent circle diameter was proven in semi-industrial pilot plant trials and confirmed by test results from industrial processes.

One driving force to develop this assessment method is the fact that normally the amount of adhesives in a printed product is unknown. If it is known, the test can be combined with INGEDE Method 13.

4 Equipment and auxiliaries

4.1 Equipment

- Analytical balance up to 1 000 g with an accuracy of $\pm 0,001$ g
- Analytical balance up to 3 000 g with an accuracy of $\pm 0,1$ g
- Hobart pulper model N 50, supplied by HOBART GmbH, equipped with a blade type stirrer (see INGEDE Method 11)
- Haindl classifier in accordance with ZM V/1.4/86 or Somerville tester according to TAPPI T 275 sp-07 or Pulmac Master Screen-type instrument according to TAPPI T 274 sp-08
- Slotted plate with a slot width of 100 µm
- Rapid-Köthen sheet former in accordance with ISO 5269/2 respectively
- Drying cabinet in accordance with ISO 287

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- Scanner-based image analysis system with a minimum resolution of 600 × 600 dpi, e. g. DOMAS, SIMPALAB

4.2 Test material

- Woodfree, virgin fibre based copy paper with an ash content of 20 ± 3 % ash determined at 525 °C
- Test material for sticky visualisation according to INGEDE Method 4

4.3 Chemicals

The required standard deinking chemicals are listed in INGEDE Method 11:

- Sodium hydroxide p. A.
- Sodium silicate, density 1,3–1,4 g/cm³
- Hydrogen peroxide, e. g. 35 %
- Oleic acid, extra pure

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5 Procedure

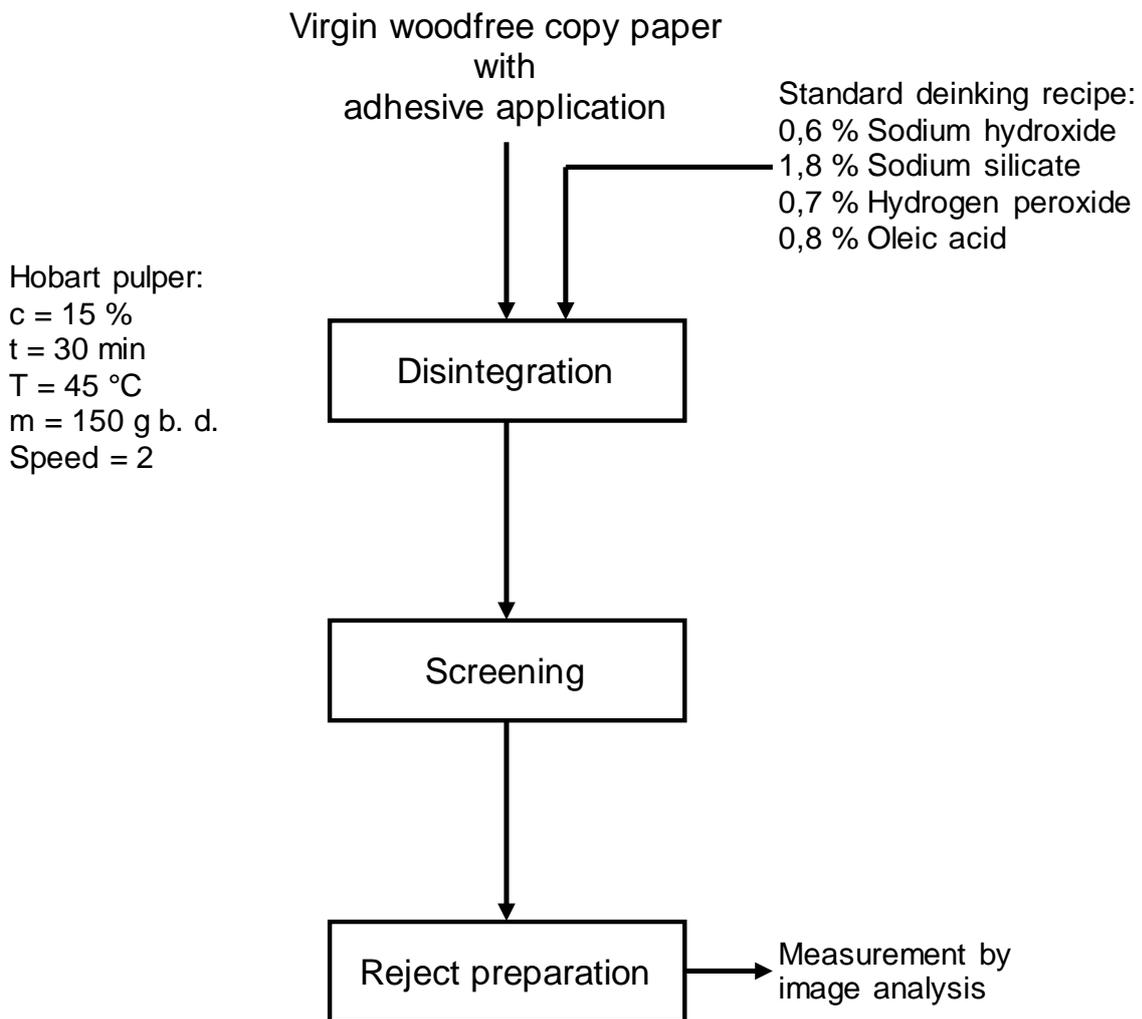


Figure 1: Testing fragmentation behaviour of adhesive applications

**Assessment of the Recyclability of Printed
Paper Products****Testing of the fragmentation behaviour of adhesive
applications****5.1 Preparation of adhesive applications**

It is recommended to store the samples under climate conditions according to ISO 187 for 24 hours. Use the recommended amount of adhesive applications as it is described below otherwise state the utilized amount or area in the report. Vary the amount of adhesive applications only for the case not reaching representative results or reduce the amount of adhesive applications if stickies heavily overlap on the reject filter.

Adhesive spine

The evaluation of a printed product includes the testing of all adhesive applications. The different adhesive applications of one printed product are tested separately and the results are added weight-proportional (mm^2/kg). Bookbinding backs which may consist of different adhesive types are tested as one compound as far as no further information is required. The spine and side binding is normally tested together in one test. The both pages at the front and at the end of the printed product should not be separated from the adhesive back unless they contain adhesive applications which should be evaluated independently.

Adhesive magazine and catalogue backs should be separated by means of a saw, leaving approximately 4 cm of the page width attached to the adhesive back. The following use of individual pieces is recommended for the test.

Table 1: Recommendation for the use of magazine/ catalogue back

Width of magazine or catalogue back	Length of each piece	Number of pieces
< 4,5 mm	2,5 cm	5
4,5–6,9 mm	2,5 cm	4
7,0–9,9 mm	2,5 cm	3
10,0–19,9 mm	2,5 cm	2
20,0–30,0 mm	1,0 cm	4
> 30,0 mm	1,0 cm	3

Side glue

If the side glue is of special interest, it is tested separately. The glued sides of the spine of a printed product are prepared similar to the adhesive backs: After separating the first two pages at the front and at the back from the spine, cut a stripe from these pages of 4 cm width including the glued area. Then cut the stripe into pieces of the recommended length in the table.

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Glued-in inserts

Glued-in inserts have to be tested separately from spine and side glue.

Inserts made of fibrous material (paper) should not be separated from the printed product page to avoid adhesive losses. The adhesive stays covered with paper on the bottom and on the top side. Cut out the adhesive leaving a frame of paper of approximately 2 cm around the adhesive. Then cut pieces with maximal 2 cm length (which might possibly mean to cut the adhesive).

It might be necessary to use several samples of glued-in inserts to generate a sufficient amount of stickies, e.g. 5 applications. Record the amount for subsequent calculation and report.

Inserts made of plastic materials are detached from the sample without removing the adhesive or with transferring the adhesive carefully back to the printed product page. Cover the adhesive with a clean part of the printed product page and then cut out the adhesive while leaving a frame of paper of 2 cm around it. Then cut pieces with maximal 2 cm length (which might possibly mean to cut the adhesive).

PSA application in printed products

Finished paper label products, e.g. in special editions, journals or magazines may contain huge flat PSA applications. Use 100 cm² of the PSA application and stick them on woodfree copy paper. Then cut them into 1–2 cm² pieces. If one printed product contains less than 100 cm² PSA application use those of several issues of the printed product. Record the number of products for subsequent calculation and report. After performing the test, express the test result in mm²/kg printed product.

PSA applications – not a final product

Not finally applied PSAs, stickers or labels, are stuck on woodfree copy paper and pressed one time with a press roll (2 kg). It is recommended to use 100 cm². This area has to be cut after attaching to the woodfree copy paper into 1–2 cm² small pieces before pulping. Record the grammage of the PSA in g/m².

If possible, the area and mass of all tested individual adhesive applications should be recorded. That will allow calculating the test result in relation to these figures.

5.2 Sample preparation

For pulping purposes, virgin fibre based copy paper (20 ± 3 % ash) is used. The total mass of copy paper and adhesive application to be tested is 150 g oven-dry. The paper should be provided in 1–2 cm² sized pieces.

5.3 Disintegration

On order to simulate an industrial sticky fragmentation, it is necessary to use a Hobart pulper under the following conditions. The total mass of copy paper and adhesive application tested is 150 g oven-dry. The total suspension volume in the vessel is 1000 ml.

In the beginning, the vessel of the Hobart pulper is filled with hot water of about 50 °C. After removing the water from the vessel, the copy paper is added as well as 300 ml of the basic chemical solution prepared in accordance with INGEDE Method 11 and tempered dilution water to a total of 925 ml. The dilution water should be heated to such a level that, after the addition of all pulp components, the temperature in the pulper is 45 °C. Directly after the start of the pulping process, set in motion by switching on the rotor at speed 2, the peroxide solution (75 ml), also prepared in accordance with INGEDE Method 11, is added. The prepared adhesive application to be tested is then stirred in immediately afterwards.

Especially during the first five minutes of the disintegration process, any solid particles that attach to the wall of the vessel should be pushed back in to ensure a complete treatment of all solid material. The pulper can be stopped briefly for this purpose.

The pulping time is 30 minutes in total. In order to keep the temperature constant during pulping and to prevent stock losses, the pulper should be fitted with a tightly closing lid.

5.4 Screening

In order to ensure that all generated sticky fragments are taken into account in the evaluation, the entire prepared stock (150 g oven-dry) is screened in portions. For this purpose, the pulped stock is filled up with water to a total volume of 3 000 ml, and the dilution water is used at the same time for rinsing out the pulper vessel. After its homogenisation, the pulp suspension is divided into three equal parts of 50 g oven-dry each. Depending on the contaminant concentration the operator can also decide to screen in portions of 25 g oven dry pulp. For this, divide the pulp suspensions into six equal parts of 25 g oven-dry (500 ml) each and dilute them to 1 liter.

The screening procedure follows INGEDE Method 4.

5.5 Specimen preparation

After each individual screening, the residue is treated according to INGEDE Method 4. It is recommended to prepare one filter sample from each individual screening. Pay attention that no overlapping of stickies occurs on the filters. In the case of presence of larger sticky fragments which can occur especially when testing adhesive backs, check visually if smaller stickies are overlaid before drying the dewatered residue. Try to separate them carefully on the filter or transfer the larger sticky fragments on an additional filter. Big, cubic sticky particles must be transferred on an additional filter (in a later step smaller and flat particles are better covered by the alumina powder).

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Besides dewatering, the preparation of the residue includes the part steps of drying and sticky visualisation. The visual check of the contrasted filter preparations is omitted, as there are no other hydrophobic particles than those to be tested contained in the residue. All hydrophobic particles that occur as a result of the adhesive application are taken into account in the following measurement by image analysis.

5.6 Measurement by image analysis

The treated filter preparations are then evaluated with the aid of a scanner-based image analysis system at a resolution of 600 dpi. The area to be measured should be selected in such a way as to ensure that all macro stickies are recorded.

Ensure that one class limit is fixed at the identical equivalent circle diameter of 2 000 μm when defining the class limits. The lowest measuring limit, in view of the method concerned, is 100 μm . When defining the upper limit, it must be ensured that no large sticky fragments are excluded.

DOMAS or SIMPALAB systems can be used. The following defined class limits have to be set:

100 μm , 200 μm , 400 μm , 600 μm , 1 000 μm , 2 000 μm , 3 000 μm , 5 000 μm , 10 000 μm and larger than 10 000 μm .

5.7 Evaluation

Add up the results of single measurements which were obtained due to the screening of individual portions. The final test results are expressed in mm^2/kg printed product air dry, for this, calculate as follows.

Adhesive spine

After the image analysis, the results for adhesive backs are obtained in $\text{mm}^2/\text{analysed filter area}$. Calculate the sticky area for the overall back length and divide it by the mass of the printed product (catalogue, magazine...) in kg. The result is mm^2 stickies per kg printed product.

Side glue

Proceed similar to the adhesive spines.

Glued-in inserts

The result of the glued-in insert is divided by the number of used inserts for one test. Divide then by the mass of printed product mass. The result is mm^2 stickies per kg printed product.

PSA application in printed products

The results of PSA application are obtained in mm^2 stickies per 100 cm^2 which have been tested. Based on that, calculate the sticky area for the effective PSA area present in the printed product. Divide then by the mass of printed product and get the result in mm^2 stickies per kg printed product.

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applications**PSA applications – not a final product

Calculate the theoretical mass of the tested 100 cm² ($m_{100\text{ cm}^2}$):

$$m_{100\text{ cm}^2} = w_{\text{PSA}} \cdot 0,01 m^2 \quad w_{\text{PSA}} \quad \text{grammage of the label, g/m}^2$$

- “Scorecard for the Removability of Adhesive Applications” (ERPC):

Calculate the amount of macrostickies per kg label product. This value is expressed in mm²/kg label and is transferred to the Scorecard.

- Macrostickies per kg printed product:

It is assumed that the share of the complete label (paper plus adhesive) is about 2,5 % of the complete printed product. Based on this calculate the factor

$$Factor = \frac{25g}{m_{100\text{ cm}^2}}$$

Multiply the sticky area in mm²/100 cm² with this factor. The result is the macrosticky area per kg printed product under the approval.

The following characteristic quantities from the accumulative result of the measurement of the three individual preparations are used for evaluation purposes:

A_{total} in mm²/kg printed product: Total area of macrostickies

A_{MS} in mm²/kg printed product: Total area of macrostickies < 2000 µm identical equivalent circle diameter

S_{2000} in %: Share of macrosticky area below a particle size of 2000 µm identical equivalent circle diameter

A_{600} in mm²/ kg printed product: The macrosticky content in the size classes below 600 µm identical equivalent circle diameter

A_{1000} in mm²/kg printed product: The macrosticky content in the size classes between 600 µm and 1 000 µm identical equivalent circle diameter

A_{2000} in mm²/kg printed product: The macrosticky content in the size classes between 1000 µm and 2 000 µm identical equivalent circle diameter

Presupposing knowledge of the adhesive mass or its application area respectively, it is possible to put the measured area of macrostickies in relation to these figures as mm²/g adhesive or mm²/cm² application respectively.

6 Report

The following should be recorded in the test report:

- Number and type of adhesive applications, amount used in the test if different
- A_{total} in mm²/kg per single tested adhesive application and overall result for the printed product
- A_{MS} and S_{2000} per single tested adhesive application and overall result for the printed product
- Deviations from the conditions of this test method

7 References

7.1 Cited Standards and methods

Reference was made to the following standards in this method:

- ZELLCHEMING Technical leaflet RECO 1, 1/2006 "Terminology of Stickies"
- ZM V/1.4/86: Gleichzeitige Bestimmung des Gehaltes an Splittern und Faserfraktionen . http://www.zellcheming.de/download/merkblaetter/merkblatt_5_1_4_86.zip
- ISO 1762 – Paper, board and pulps – Determination of residue (ash) on ignition at 525 °C
- TAPPI T 275 sp-07: Screening of Pulp (Somerville-Type Equipment)
- TAPPI T 274 sp-08: Laboratory screening of pulp (Master Screen-type instrument)
- INGEDE Method 4: Analysis of macrostickies in deinked pulp
- INGEDE Method 11: Assessment of Print Product Recyclability - Deinkability Test
- ERPC: Assessment of Printed Product Recyclability – Scorecard for the Removability of Adhesive Applications www.paperforrecycling.eu
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method
- ISO 287 (2009): Paper and Board – Determination of moisture content – Oven drying method

7.2 Sources

This INGEDE Method was developed and tested within the scope of INGEDE project 66 99 PMV "Evaluation of recyclability of print products with particular consideration of adhesive pulp components" in 2001. In the course of the INGEDE project 129 09 "Preparation of an adhesive application database and development of a recyclability scoring system" the INGEDE Method 12 was revised in 2010.

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