

Introduction

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

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

- deinkability
- screening ability of adhesive applications.

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

This procedure can be used to evaluate the suitability of adhesive applications for recycling. The method is based on the general requirement that it should be possible to separate adhesive applications mechanically.

1 Scope

This INGEDE method describes a procedure for testing the fragmentation behaviour and screening ability 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",
- also refer to INGEDE Method 4.- Sticky particles in the reject of a non-pressure laboratory screening using a slotted plate as separating element (evaluation by INGEDE Method 4)

Adhesive backs:

- Adhesive binding of printed books, consisting of back and side binding

PSA:

- Pressure sensitive adhesives (PSA), 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 (Fig. 1).

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

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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 was proven in semi industrial pilot plant trials and confirmed by test results from industrial processes.

One driving force to develop this type of assessment method is the fact that normally the amount of adhesives in a printed product is not known. If it is known, the test can be combined with INGEDE Method 13.

4 Equipment and auxiliaries

The following equipment, test material and chemicals are required:

4.1 Equipment

- Analytical balance up to 1 000 g with an accuracy $\leq \pm 0,001$ g
- Analytical balance up to 3 000 g with an accuracy 0,1 g
- Hobart pulper model N 50, supplied by HOBART GmbH, equipped with a blade type stirrer (see INGEDE Method 11)
- Haindl Fractionator in accordance with ZM V/1.4/86 or Somerville Fractionator according to TAPPI T275 sp-98
- Slotted plate with a slot width of 100 µm
- Rapid-Köthen sheet former in accordance with ISO 5269/2 or DIN 54 358 respectively
- Drying cabinet in accordance with ISO 287
- Scanner-based image analysis system with a minimum resolution of 600 × 600 dpi, e. g. DOMAS, SIMPATIC.

4.2 Test Material

- Black water-based ink, e.g. Pelikan, no. 4001
- Silicone paper with a very high release force
- Filter paper, medium to wide pore size, medium-speed filtering, machine finished, moisture resistant, white, 240 mm diameter, e. g. Ederol no. 12
- Aluminium oxide powder:
- EK white, grain size 220
- Newsprint paper (virgin fibre based)
- Wood free, virgin fibre based copy paper with an ash content of 20 % ± 3 % ash determined at 575 °C.

4.3 Chemicals

- The required deinking chemicals are as listed in Chapter 6.2 of 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

5 Procedure

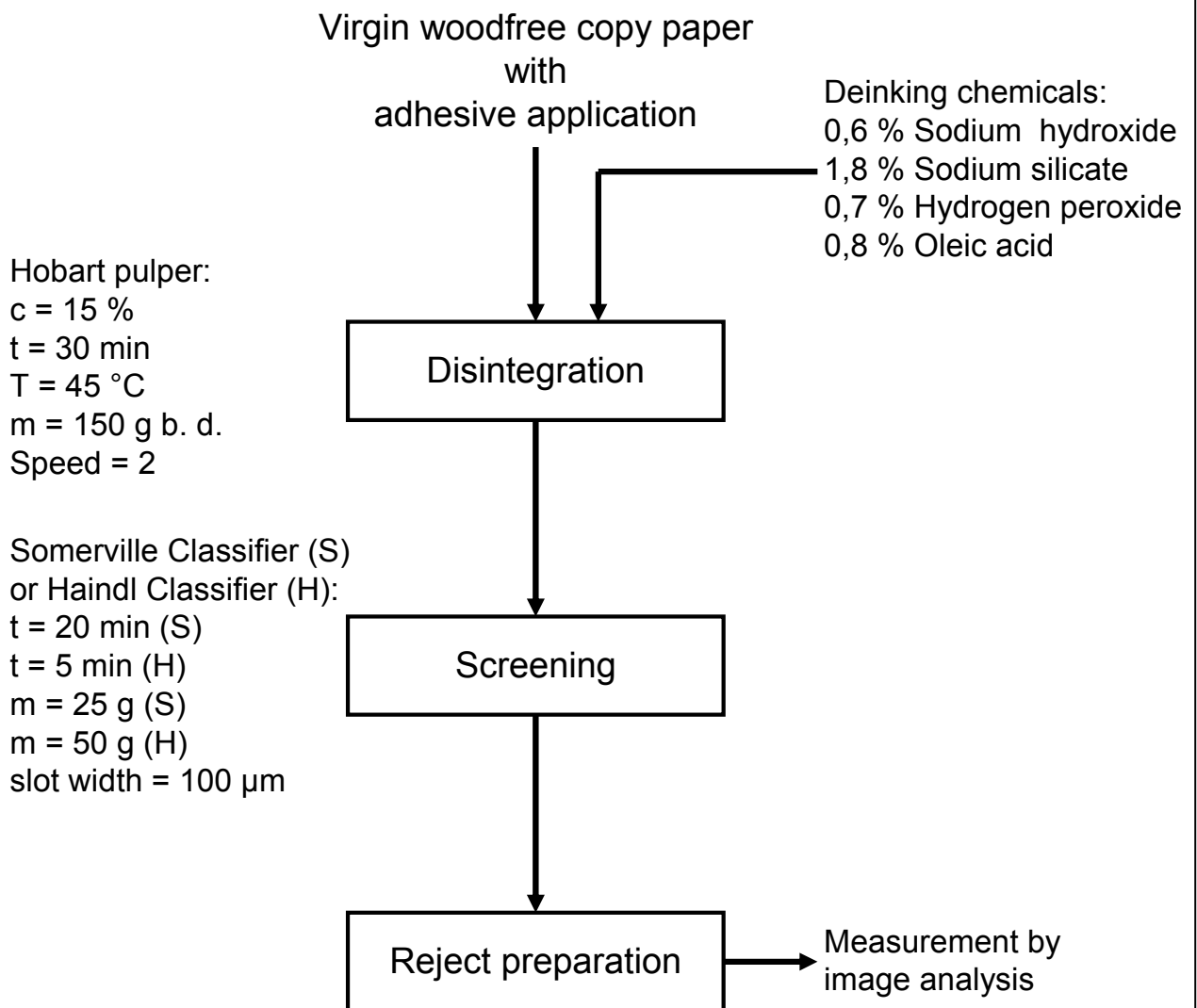


Fig. 1: Sticky Forming Potential Test

5.1 Sample preparation

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. Similarly, the two first and two last page parts of the print product to be tested should be separated from the adhesive back. Glued-in products have to be removed.

Applications of pressure sensitive adhesives (PSA), usually stickers or labels have to be stuck on a newsprint paper and pressed one time with a press roll (2 kg).

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Glued-in inserts in magazines/catalogues have to be used completely in the test. Both pages, the insert and the magazine paper, have to be used to avoid adhesive losses on one of the two pages.

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

5.2 Preparation of adhesive applications

Very important is the use of a suitable sample quantity, which has to be selected for carrying out the test. Such quantities on the one hand are suitable that do not lead to overlapping on the reject samples prepared after laboratory screening (e. g. less than 10 cm² with adhesive backs, 100 cm² with labels). On the other hand a sufficient amount of stickies has to be measured to obtain representative results.

In the case of magazine and catalogue book binding backs pieces of 1,0 cm or 2,5 cm length are cut from the separated back (see chapter 5.2). The following use of individual pieces is recommended for the test.

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

Table 1: Recommendation for the use of magazine/catalogue backs in the sticky test

From the attached PSA label 100 cm² are recommended to be used for the test. This area has to be cut after attaching to the newsprint into 1-2 cm² small pieces before pulping.

Due to the fact that the adhesive amount for glued-in inserts is very small, it is recommended to use at least 5 applications for the test.

5.3 Disintegration

For practice-oriented sticky fragmentation, it is necessary to use a Hobart pulper under the following conditions.

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.

In the beginning, the vessel of the Hobart pulper is filled with the copy paper, 300 ml of the basic chemical solution prepared in accordance with chapter 6.2 of the INGEDE Method 11 and tempered dilution water to a total of 925 ml. The dilution water should be heated to such a level

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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 chapter 6.2 of INGEDE Method 11, is added. The 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 so as to ensure complete treatment of all solid material. The pulper can be stopped briefly for this purpose.

The pulping time lasts a total of 30 minutes. In order to keep the temperature largely constant during this time 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. When using a Haindl Fractionator, there are three screenings with 50 g oven-dry pulp each. In case of Somerville, make six portions with 25 g oven-dry pulp each. The results are added up.

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 six equal parts of 25 g oven-dry (500 ml) each and diluted to 1 litre in each case.

5.4.1 Haindl Fractionator

By screening in a Haindl Fractionator the procedure is similar to the process for the "Gleichzeitige Bestimmung des Gehaltes an Splittern und Faserfraktionen" (Simultaneous determination of the content of shives and fibre fractions) in accordance to ZM V/1.4.86, but without connection to the McNett device. The screening conditions correspond to those of INGEDE Method 4, for which the use of a 100 µm slotted plate is defined.

The pulp is added in two steps: with the start of the laboratory screening system, half of the quantity of fibre pulp to be processed in each individual screening process (25 g oven-dry) is entered into the fractionator and screened for 2.5 minutes. Following this, 25 g oven-dry pulp is again added during the process. Screening is stopped after a total of 5 minutes and the residue is used for sample preparation in accordance with chapter 5.5. The screening process is subsequently repeated two more times.

5.4.2 Somerville Classifier

By screening in a Somerville classifier the following procedure is used: Six screenings of the suspension with 25 g oven-dry pulp are used for each individual Somerville screening. The suspension is poured into the Somerville and screened for 20 minutes.

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. Care has to be taken that no overlapping of stickies occurs on the filters.

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Besides dewatering, the preparation of the residue includes the part steps of drying and sticky visualisation. Only the visual check of the contrasted filter preparations is omitted, as there are no further hydrophobic particles contained in the residue in addition to the adhesive application to be tested.

Before drying the dewatered residue, care should be taken that, in case of the presence of larger sticky fragments which can occur especially when testing adhesive backs, no smaller stickies are overlaid.

All hydrophobic particles that occur as a result of the adhesive application are taken into account in the following measurement by image analysis. This is valid also for particles that have no tacky properties.

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.

When defining the class limits, care should be taken to ensure that one class limit is fixed at an identical equivalent circle diameter of 2 000 μm . The lower 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 Simpatic systems can be used. The following defined class limits have been stated: 100, 200, 400, 600, 1 000, 2 000, 3 000, 5 000, 10 000 and larger than 10 000 μm .

5.7 Evaluation

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

Share of macrostickies < 2 000 μm (identical equivalent circle diameter) S_{MS} in %

Total area of macrostickies < 2 000 μm (identical equivalent circle diameter) A_{MS} in mm^2

The identical equivalent circle diameter of 2 000 μm marks the size limit from which a complete separation of coarsely-dispersed stickies from recovered pulp suspensions is possible under industrial screening conditions. Therefore both evaluation parameters refer to the share and area of stickies that cannot be completely removed under industrial screening conditions and that consequently should be kept to an absolute minimum.

It is recommended to base the standardisation on mm^2/kg print product or $\text{mm}^2/\text{application}$ respectively.

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 mm^2/g adhesive or mm^2/cm^2 application respectively.

6 Report

- The following should be recorded in the test report:
- Number and type of adhesive applications

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Applications**

- Share of macro stickies S_{MS} per adhesive application that cannot be separated under industrial conditions in %.
- Macrostickies area per reference unit (mass of printed product, mass of adhesive applications or area of adhesive applications) that cannot be separated under industrial conditions
- Comparison to reference applications
- Possible 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:

- DIN EN ISO 20 287: Paper and board: Determination of the moisture content after the warming cabinet process.
- ZM V/1.4/86: Gleichzeitige Bestimmung des Gehaltes an Splintern und Faserfraktionen, . http://www.zellcheming.de/download/merkblaetter/merkblatt_5_1_4_86.zip
- Zellcheming Technical leaflet RECO 1, 1/2006 "Terminology of Stickies".
- INGEDE Method 4: Analysis of macro stickies in deinked pulp.
- INGEDE Method 11: Assessment of Print Product Recyclability - Deinkability Test.
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method.
- TAPPI UM 242: Shive content of mechanical pulp (Somerville Fractionator).

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.

This first major revision was established in 2004 as a result of the common INGEDE Project 9303 of CTP, PTS and PMV "Adhesive Eco-design: Task Force for the Evaluation of Adhesive Products designed to be less Detrimental to Paper Recycling".

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