| INGEDE Method 13 | Assessment of the Recyclability of Printed Products<br>Testing of the Macrosticky Formation Rate of Adhesive Applications | INGEDE International Association of the Deinking Industry |
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8 Pages

## 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 in a deinking plant.

## 1 Scope

This INGEDE method aims to characterise adhesive applications behaviour (on a weight basis) in terms of macrostickies formation rate during disintegration. For the application of this method it is necessary to know the content of adhesive application in the printed product to be tested. The portion of the adhesive, which is not recovered by this method, is also a measure for microstickies and potential secondary stickies. Thus the test result can be also considered as estimation for not screenable adhesive particles.

## 2 Terms and definitions

Macrostickies, microstickies, potential secondary stickies:

Refer to INGEDE Method 12, chapter 3, 9 and to Zellcheming Technical Leaflet RECO 1, 1/2006 “Terminology of Stickies”

## 3 Principle

The general background of assessing the recyclability of adhesive applications is described in INGEDE Method 12, chapter 3 "Principle".

For the test the amount of adhesive material in the recovered paper sample must be known.

The pulping process is carried out as in INGEDE Method 12. Then the pulp is treated with a cellulase in order to digest most of the fibres. The sample is afterwards screened as described in INGEDE Method 4. The evaluation of the amount of adhesive, which can be recovered onto the 100 µm slotted plate is significant for the macrosticky formation rate and for its potential removal ability from the process; the part passing the slots represents the amount of adhesives remaining in the process, as not screenable micro or colloidal stickies. This part is known to be detrimental for paper recycling.

The test can be combined with INGEDE Method 12. In that case it becomes possible to estimate a rough percentage of the not screenable adhesive particles, below the critical size limit of 2 000 µm (circle equivalent diameter). It has been determined by pilot trials that this size limit is supposed to represent 100 % removal rate in industrial screening.

On the basis of this final result, it is then possible to compare the behaviour of different paper products containing adhesive applications.

## 4 Equipment and auxiliaries

The following equipment, test materials and chemicals are required:

### 4.1 Equipment

- Analytical balance up to 1 000 g with an accuracy ≤ ± 0,001 g
- Analytical balance up to 3000 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)
- Water bath thermo regulated at 37 °C ± 1 °C
- Magnetic agitation stirrer or blade stirrer
- Glass vessels with a volume of 2 or 3 litres
- 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.

### 4.2 Test material

- 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
- 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
- Enzyme cellulase “ONOZUKA R10” distributed by YAKULT Pharmaceutical Ind. Co. LTD (Japan)
- 0,5 mol/l Buffer solution acetic acid / sodium acetate; pH 4,6.

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## 5 Procedure

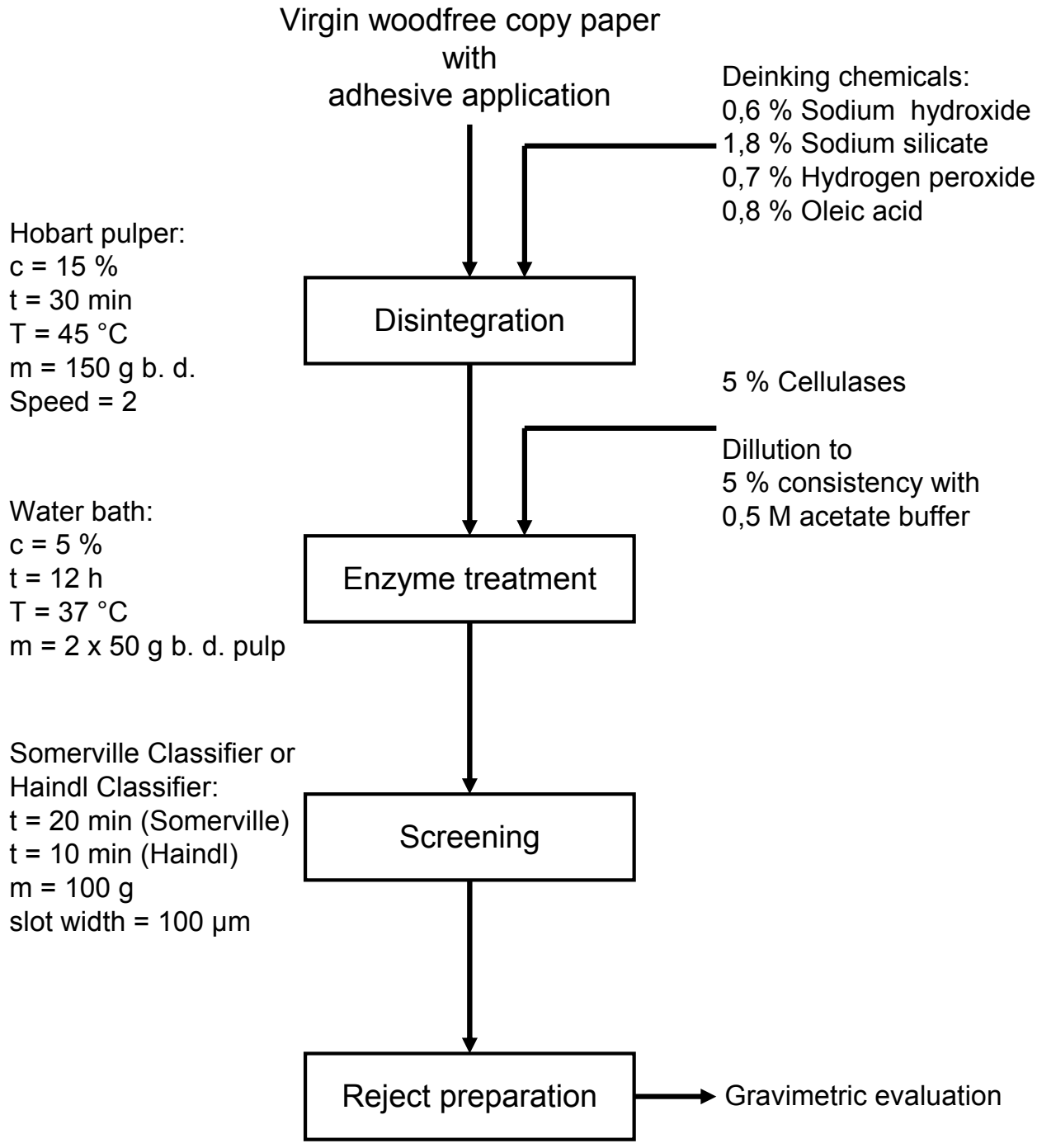


**Fig. 1: Estimation of the macrostickies generation during disintegration according to INGEDE Method 13**

## 5.1 Sample preparation

### 5.1.1 Preparation of adhesives

Please refer to INGEDE Method 12, chapter 5.

### 5.1.2 Weighing the samples

As gravimetric measurement is done a sufficient quantity of adhesive must be introduced in the test. 0.3% adhesive material in about 150 g of paper sample seems suitable.

## 5.2 Disintegration

The amount of adhesive introduced in the pulper is in the range of 0,3 %.

The pulping conditions are defined as in INGEDE Method 12:

Raw materials: Copy paper is used for the disintegration. Total raw material mass (incl. added adhesive application): 150 g oven-dry pulp. The raw material is cut into pieces of 1-2 cm² size.

Chemical formulation: 0,6 % soda, 1,8 % silicates, 0,7 % peroxide, 0,8 % oleic acid.

Speed of the Hobart pulper: 2

Temperature: 45 °C

Consistency: 15 %

Pulping time: 30 minutes.

After disintegration, 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 receptacle. If the test is combined with INGEDE Method 12, the pulp suspension after its homogenisation is divided into adequate parts to perform either the screening or the enzymatic treatment.

## 5.3 Assessment of macrosticky formation rate

### 5.3.1 Enzymatic treatment

50 g oven-dry pulp (3 to 5 % consistency) is introduced in a 2 l vessel (2 vessels for 100 g).

2,5 g (5 % based on dry pulp) of cellulase is added in each vessel. The pulp is diluted to 5 % consistency by the addition of 0,5 M acetate buffer.

The vessels are plugged and placed in a bath equipped with a thermal regulation at 37 °C (± 1 °C) under gentle agitation.

The digestion (hydrolysis) of the fibre material requires 12 hours (12 h are sufficient); usually, for greater convenience overnight is applied.

#### 5.3.2 Laboratory screening

The slurry obtained after digestion (hydrolysis) of 100 g pulp is poured into the laboratory screening device, one vessel after the other; both vessels are added, one after the other, taking care for a sufficient water flow.

Either a Somerville or a Haindl apparatus equipped with a 100 µm slotted plate is used. The duration of the screening phase is 20 minutes for Somerville, 10 minutes for Haindl.

#### 5.3.3 Collection of the contaminants

After screening, the contaminants trapped onto the slotted plate are recovered in an adequate volume of water. The suspension water with the contaminants is filtered with a pre-weighed paper filter in the Rapid-Köthen hand sheet former and then dried (as in INGEDE Method 4).

Before weighing, the filter is placed in an oven (105 °C), for 1 hour, in order to remove the residual humidity.

If some cellulose materials are still included in the rejects, a silicone paper is put on the filter. The filter is rolled with a 2 kg roll and slightly brushed with a paintbrush to remove the not sticky materials which are not stuck on the filter.

#### 5.3.4 Measurement

The stickies mass $m_1$ (g) is assessed by weighing the filter and subtracting the weight of the pre-weighed filter.

### 5.4 Evaluation

Calculation of the macrosticky formation rate: $R_{MS}$ (in percent)

$m_1$: is defined as the weight of rejects recovered from 100 g treated and screened pulp.

$m_2$: is defined as the adhesive quantity introduced in the pulper (per 100 g of pulp).

$$R_{MS} = \frac{m_1}{m_2} \times 100\ [\%]$$

#### 5.4.1 Evaluation in combination with the INGEDE Method 12

A possible but not exact estimation of the screenable part of the stickies can be found in the annex of this method.

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## 6 Report

The following should be reported:

- Number and type of adhesive applications
- Amount of adhesive content
- Calculation of the macrostickies formation rate: $R_{MS}$ (in percent)
- In the case that the test is combined with INGEDE Method 12 for a rough estimation of the screenable adhesive part (annex):
  Share of macro stickies per adhesive application $S_{MS}$ that cannot be separated under industrial conditions and share of total sticky material < 2 000 µm ***ST***
- Comparison to possible reference applications
- Any deviations from the conditions of this test method

## 7 References

### 7.1 Cited standards and methods

- DIN 50 011-T01: Material, constructional element and appliance testing; ovens, terms, requirements.
- DIN EN ISO 20 287: Paper and board: Determination of the moisture content after the warming cabinet process.
- ZM V/1.4/86: Simultaneous determination of content of shives and fibre fractions.
- Zellcheming Technical leaflet RECO 1, 1/2006 “Terminology of Stickies”
- INGEDE Method 4: Analysis of macro stickies in deinked pulp.
- INGEDE Method 11: Assessing the recyclability of print products – Deinkability Test.
- INGEDE Method 12: Assessment of the Recyclability of Printed Paper Products – Testing of the Fragmentation Behaviour of Adhesive Applications
- 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 method was developed by CTP and added to the INGEDE methods in the frame of the INGEDE project 93 03 CTP/PMV/PTS TASK Force "Adhesives Eco-Design". The results were reported by **Brun, J., Delagoutte, T., Hamann, L. and Putz, H.-J**:as “Eco-design: A European methodology to characterize the behaviour of adhesive products during paper recycling” at the 3rd CTP/PTS Recycling Symposium, March 16-18, 2004, Grenoble, France and at the 11th PTS/CTP Deinking Symposium, April 27-30, 2004 - Leipzig, Germany (Doc CTP n°2132).

The original method was published in Progress in Paper Recycling, Vol 10(4), pp 14-19, August 2001), **Delagoutte,T. and Laurent A.**: "Modified method for the quantification of primary stickies in a recycled pulp".

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**Annex:**

If the method is used in combination with INGEDE Method 12, the following calculation can be used to get a rough estimation about the screenable part of the adhesive application in this test.

Share of macrostickies < 2 000 µm (in %) per adhesive application (this amount will not be separated completely in screening and removed completely under industrial conditions), determined on the curve representing the cumulative area distribution in percentage: $S_{MS}$

Share of total sticky material < 2 000 µm (in %): ***ST***

$$ST = \frac{R_{MS} \times S_{MS}}{100} + \left(100 - R_{MS}\right)[\%]$$

Combining a share of weight and a share of area in one term is scientifically not exact. Therefore the following evaluation can be only used as a rough estimation for the industrial screening ability of adhesive applications. This is also the reason that is an annex and not part of the official method.