INGEDE Method 4	Analysis of macrostickies in pulps	INGEDE International Association of the Deinking Industry
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Introduction

Stickies in pulps originate from tacky components in paper for recycling. They cause problems during paper production and converting as well as quality defects.

This method is widely accepted to measure the macrosticky content of pulps.

1   Scope

This INGEDE Method is used to analyse macrostickies in pulps.

2   Terms and definitions

Macrostickies:

Tacky components originating from paper for recycling which can be analysed from the residues of a laboratory screening (see also the corresponding leaflet of ZELLCHEMING).

3   Principle

The method describes a laboratory screening procedure for pulps of a paper recycling process. The reject of this screening procedure is prepared in such a way that the macrostickies can be determined by means of an image analysis system.

4   Equipment and auxiliaries

4.1   Equipment

4.1.1 Disintegration

Any device which fulfils the requirements of ISO 5263-1 may be used for disintegration of samples.

4.1.2 Screening

Macrostickies can be separated from recycled pulp suspensions using various laboratory screening devices. Possible screening devices are the Haindl classifier (ZM V/1.4/86), the Somerville tester (TAPPI T 275 sp-07) or the Pulmac Master Screen (TAPPI 274 sp-08).

4.1.3 Slotted plate

For pulps in deinking, the use of a slot width of 100 µm is recommended. Other slot widths have to be reported as deviation from the method.

NOTE:

Screening investigations with slotted plates of nominally the same slot width showed significant differences in the screening result (see INFOR Project 118). The maximum slot width correlates with the macrosticky area. Therefore it is recommended to measure all slots widths on the slotted plate. A ZELLCHEMING test method for the quality requirements of slotted plates in laboratory screening devices is available.

4.1.4 Reject dewatering and drying

Any device which fulfils the requirements of ISO 5269-2 may be used for dewatering and drying the dewatered screening rejects e. g. the Rapid-Köthen unit. Additionally, an oven which fulfils the specifications of ISO 287 is required.

4.1.5 Image analysis

An image analysing system comprising a flatbed scanner and PC with a suitable control and analysis program is used for the measurements. The scanner is to be calibrated to ensure reproducibility of the measurements.

Technical requirements of the flatbed scanner:

- Scanning area ≥ ISO A4
- Optical scan resolution ≥ 2000 dpi
- Colour depth 48 bit
- Optical density DMAX ≥ 4,0

Requirements on measuring accuracy of flatbed scanner after warm-up period (see scanner manual) and under scanning conditions (see chapter 5.6).

- Reproducibility of mean grey value (8 Bit) is ±1. That means that an ISO A4 sample has to be scanned 10 times without any movement of the sample; all mean grey values of total sample area should be within 2 grey values.
- Deviation of colour value (RGB 8 Bit) ≤ 5. That means that after calibration a scanned image of IT8-Target should not deviate more from associated reference file than ± 5 values in every colour channel R, G, B.

Suitable scanner: DOMAS ScannerAdvanced or Techpap SIMPALAB proposed Scanner

 "ScannerAdvanced" is a name given by PTS to a commercial scanner that was accredited by PTS. This scanner device is delivered with the DOMAS 3.0 version.

The software should be able to detect white particles on a black background. Suitable software package are DOMAS 3.0 and above image analysis software as well as Techpap SIMPALAB software.

4.2 Test material

The following testing material may be used:

- Black water-based ink, e. g. Pelikan No. 4001

- One sided, silicone-coated release paper (60 g/m²)

- Filter paper: medium to large pores, medium filtration speed, machine finished, good wet strength, white, e.g. Munktell Filtrak 1289, 240 mm diameter

- Special fused alumina powder: white, sharp-edged particles, grain size 220 according to FEPA Method.
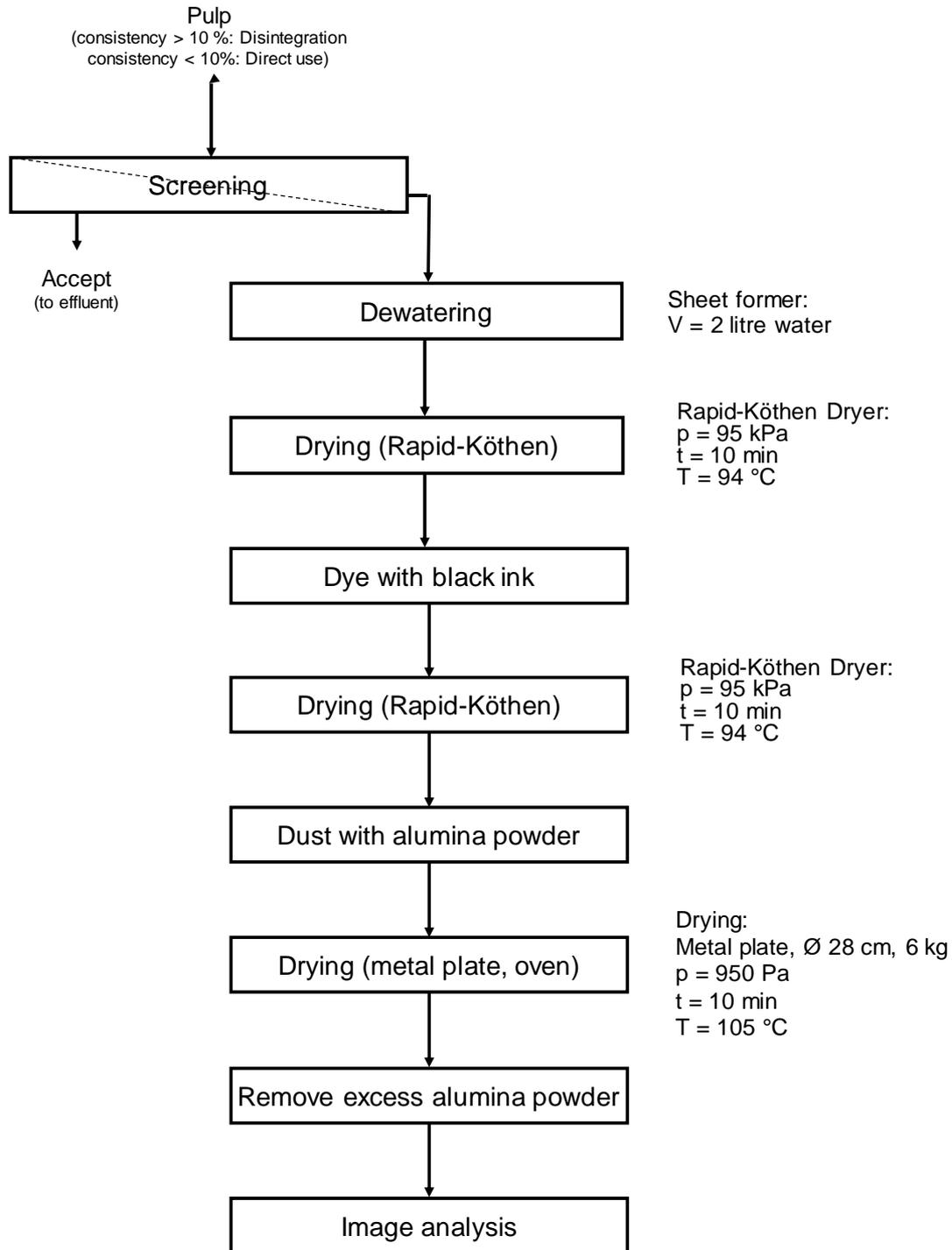
(Sources of supply see chapter 7.4)

Analysis of macrostickies in pulps

5 Procedure

Pulp
(consistency > 10 %: Disintegration
consistency < 10%: Direct use)

Screening

Accept
(to effluent)

Dewatering — Sheet former:
V = 2 litre water

Drying (Rapid-Köthen) — Rapid-Köthen Dryer:
p = 95 kPa
t = 10 min
T = 94 °C

Dye with black ink

Drying (Rapid-Köthen) — Rapid-Köthen Dryer:
p = 95 kPa
t = 10 min
T = 94 °C

Dust with alumina powder

Drying (metal plate, oven) — Drying:
Metal plate, Ø 28 cm, 6 kg
p = 950 Pa
t = 10 min
T = 105 °C

Remove excess alumina powder

Image analysis

Figure 1: Test procedure INGEDE Method 4

5.1 Sampling and sample preparation

Pulp suspensions with a consistency up to 10 % can be used immediately for screening without further preparation. However, pulp with higher consistency must be disintegrated before screening. 50 g oven-dry pulp in 2000 ± 25 ml is disintegrated. Disintegration is fulfilled in a device in accordance with ISO 5263-1 whereby the disintegration process is restricted to five minutes. Longer periods of mechanical stress should be avoided in order to prevent changes of the sticky size distribution in the sample.

5.2 Screening

5.2.1 General

For a statistically sound statement about the macrosticky content, the screening of three individual portions, each containing 50 g of oven-dry material from one sample, is recommended. Some pulps may cause difficulties during screening due to high long fibre content or high level of contamination. In this case the pulp amount can be split into portions (e.g. 2 x 25 g) and/or the screening time can be elongated. In case of Pulmac Master Screen the reduction is preferred vs. the change of the screening programme. A reduction of the sample's quantity is also necessary if the load of stickies is so high that the stickies overlap heavily after contrasting.

Any deviation has to be reported.

When using a plastic screen plate, mechanical stress can lead to material fatigue and destruction of the slotted plate. For this reason the use of a metal plate is recommended.

5.2.2 Screening conditions overview

The following table gives an overview about screening equipment and conditions.

Table 1: Screening conditions

Equipment	Reference	Water flow	Stroke	Duration (Pulp input + further screening)
Haindl classifier	ZM V/1.4/86	10 l/ min	480 double strokes per minute	5 min + 5 min
Somerville tester	T 275 sp-07	8,6 l/ min	700 rpm	2 min + 18 min
Pulmac Master Screen	T 274 sp-08	Depends on programme setting (Modus B)		

5.2.3 Haindl classifier

The screening with the Haindl classifier is performed according to ZM V/1.4/86, without using the McNett unit. In order to guarantee problem-free screening of 50 g of oven-dry pulp, unlike ZM V/1.4/86 the screening conditions should be set up as follows. The stroke frequency of the membrane should be increased to 480 double strokes/ minute (maximum stroke rate). Because of the resulting turbulence increase in the screening chamber, the height of the cylindrical supply vessel wall should be increased from 130 mm to 370 mm. The container can be extended using an acrylic glass top. The washing water flow should be 10 litres per minute for the entire screening duration. After continuously adding pulp for 5 minutes, the pulp continues to be screened for 5 minutes until screening is complete.

5.2.4 Somerville tester

The screening in a Somerville tester is performed referring to T 275 sp-07. 50 g oven dry pulp is poured into the screen box within the first 2 minutes of 20 minutes overall screening duration.

5.2.5 Pulmac Master Screen

When using the Pulmac Master Screen, 50 g of oven-dry pulp is added to the supply chest. The screening which follows is automatic. Programme setting should be "Modus B". Before screening, a wet filter paper which retains the reject when the screening is complete must be placed onto the sieve in the dewatering unit (autofilter).

5.3 Dewatering the reject

The reject is flushed from the slotted plate into a container using about one litre of water. Using a moistened white paper filter above the sheet forming wire the reject is dewatered in the sheet former (Rapid-Köthen model). It is advisable to operate the sheet former manually. When the reject sample and an additional litre of water are in the sheet former, the aeration is started before dewatering. After dewatering, the specimen which has been formed is placed onto a couching board with the bottom of the filter (reject-free side). If the load of stickies is so high that the stickies overlap after contrasting (chapter 5.5), the reject has to be portioned to several filter papers. It is also possible to separate overlapped stickies 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).

When using the Pulmac Master Screen, the reject is dewatered in the unit automatically using the same type of filter paper. The dewatered specimen can be removed after the screening is complete. It is also laid onto a couching board with the bottom side of the filter.

5.4 Drying

The top side of the specimen is then covered with the coated side of the silicone-coated sheet of release paper. Then the sample is dried for 10 minutes in the sheet dryer (Rapid-Köthen model) at 94 °C and a pressure of 95 kPa.

5.5   Sticky examination

After drying, the stickies are examined by utilising their adhesive properties in order to provide the contrast to the specimen's background which is required for image analysis. Before removing the silicone coated release paper the specimen is cooled down for a short time. Heavy sticky particles could adhere to the silicon paper and must be transferred back to the filter paper.

The dried specimen is then drawn through a submersion bath containing black water-based ink, so that the entire surface is covered. The dyed specimen is then laid with its bottom side on a piece of blotting paper (bleached sheet of cellulose or tissue), so that any excess ink is absorbed. Then the specimen is dried for another 10 minutes, the top side covered with the previously used silicone-coated release paper.

In order to avoid discoloration of the drying equipment, the specimen should be placed between two couching boards during drying.

Subsequently after a short cooling down, the specimen is completely covered with a thick, even layer of white special fused alumina powder, the top and bottom sides are covered with couching board and it is then dried for 10 minutes in an oven at 105 °C. The specimen is loaded with a pressure of 950 Pa (6 kg metal plate, Ø 28 cm) to fix the powder on the tacky areas. The metal plate should be stored in the oven permanently to keep the high temperature. After the procedure is complete, the specimen should be removed from the oven. Excess, loose powder has to be removed with a soft cosmetic brush, without applying pressure, whilst holding the specimen in a vertical position.

After the stickies have been contrasted inspect the stickies visually. It is important that the stickies do not overlap. The visual inspection also serves to check whether all white hydrophobic impurities such as pieces of plastic film have been removed. In order to do this, the components to be eliminated should either be removed using tweezers or marked using a black permanent marker so that they are not detected during the subsequent image analysis.

5.6   Image analysis

The prepared specimen is then analysed using a scanner-based image analysis system. When selecting the measuring area, the preparation area should be used in order to analyse as many of the stickies which were retained during screening as possible. The largest possible measuring area should be selected.

The top side of the recommended 3 specimens per sample are to be assessed by the image analysis system. The arithmetic mean of the 3 measured values is to be calculated. Scanning conditions: The sheets should be free of crinkles and waves to lie flat on the scanner. An opaque batch of black carton should be used as background. Every specimen should be scanned one time from top side with 8-bit grey modus, 600 dpi and reflective light.

If the scanner is idle for more than 15 minutes, a blank scan has to be made before any new measurement.

Parameterisation of image analysis software: The threshold value and the size classification are defined in the following. Other threshold setting is regarded as deviation from this method and must be reported. When setting the class limits, the size of the slots in the slotted plate which

was used for screening is set as lower limit (regular 100 µm). Smaller stickies cannot be expected because of the sticky surface increase which is associated with the drying process. The final class may not have an upper limit, so that all stickies are recorded.

The amount of pulp used (typically 50 g oven-dry) has to be known as input value for the image analysis software to calculate the macrosticky area as described in chapter 5.7.

In case of using DOMAS image analysis system the following parameters are recommended:

- Set "Slot width" to "100" (µm)
- Select "Circular sample with border"
- Select "Light contrasted stickies"
- Select Threshold method "fixed threshold" and set parameter to "95"
- Select "Size classification" with "circle equivalent diameter" and select "ingede4.kls"
- Set "Pulp mass depended" and "…g"
- Select "Image source" "scan series" select "stickies_1.scn"
- Set "No. of samples" to "3"
- Select "Average series of results"

NOTE:

If the load of stickies is very high, it is recommended to reduce the amount of stickies on the filter paper instead of changing the threshold. In that case follow the same procedures as for overlapping (chapters 5.3 and 5.5).

In case of using Techpap SIMPALAB software:

- Open Family (from Menu Parameter)
- Select the family "ingede4.cfg" from the list

All the settings are pre-installed for the measurement of stickies. The threshold of measurement, sizes for classification (100–200 µm, ...) and other parameters are already determined in the file "ingede4.cfg", and set automatically.

5.7 Calculation of the macrosticky content

The results of the image analysis should be given in mm² of stickies per m² of specimen. This value should be then converted into mm² of sticky area per kg of pulp (see Equation 1). The specimen area which was actually measured by the image analysis system in relation to the covered filter paper surface (or in maximum the inner diameter of the sheet former) and the amount of material used during screening (recommended 50 g of oven-dry pulp) have to be taken into consideration.

Equation 1: Macrosticky area in mm²/kg

$$Macrosticky\ area = \frac{Sticky\ area\ in\ \frac{mm^2}{m^2} \cdot Specimen\ area\ in\ m^2}{Amount\ of\ material\ in\ kg}$$

The use of 50 g of oven-dry pulp and dewatering using the Rapid-Köthen unit results in a conversion factor of 0,634 for converting the area-based sticky area into a weight-based sticky area. Then the mathematical mean of the individual results should be calculated for the three specimens which were made from each pulp sample.

It is advisable to calculate the coefficient of variation and to repeat the measurements if the coefficient of variation is higher than 10 %.

The measurements can be shown separately for the determined size classes and also as the total sticky area for all size classes.

6 Report

The following should be noted in the test report:

- Designation of the sample

- Type of screening unit used

- Type of slotted plate used

- Type of image analysis system used

- Threshold setting if other than defined

- Average macrosticky content in mm²/kg of the recommended three individual samples and coefficient of variation

- Any deviation from this method.

7 References

7.1 Cited standards and methods

- ZELLCHEMING Technical Leaflet RECO 1, 1/2006 "Terminology of Stickies"; www.zellcheming.com/service/ , follow "leaflet" and "RECO"

- ZM V/1.4/86: Simultaneous determination of shives and fibre fraction content (in German); www.zellcheming.com/service, follow "leaflet" and "TEST"

- ZELLCHEMING Technical Leaflet RECO 1, "Anforderungen an die Güte von Schlitzplatten für Labor-Sortieraggregate" (engl.: "Quality Requirements of Slotted Plates for Laboratory Screening Devices"); www.zellcheming.de

- TAPPI T 275 sp-07: Screening of Pulp (Somerville-Type Equipment)

- TAPPI T 274 sp-08: Laboratory screening of pulp (Master Screen-type instrument)

- ISO 5263-1 (2004): Pulps – Laboratory wet disintegration – Part 1: Disintegration of chemical pulps

- ISO 5269-2 (2005): 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

- FEPA: www.fepa-abrasives.org

7.2 Literature and other related documents

- Ackermann, C.; Putz, H.-J.; Göttsching, L.: INGEDE Method for the Analysis of Macro Stickies in DIP. Das Papier 51 (1997), no. 6, 271-282 (in German)

- Ackermann, C.; Putz, H.-J.; Göttsching, L.: Improved Macro Sticky Analysis for DIP based on Screening. Progress in Paper Recycling 7 (1998), no. 2, 22-32

- German INFOR Project 118: Improvement of Reproducibility of standardized Macrosticky Methods (Final Report: PMV/PTS September 2009)

- H.-J. Putz, E. Hanecker: Untersuchung relevanter Einflüsse auf Makro-Stickyergebnisse. Wochenblatt für Papierfabrikation, 139 (2011), no.2, 116-123

7.3 Sources

The INGEDE Method is based on the INGEDE Project 38 94 PTS/PMV "Developing methods for performing quantitative analyses of micro and macro stickies".

PTS (www.ptspaper.de), PMV (www.pmv.tu-darmstadt.de)

7.4   Sources of supply

Silicone paper:

- 60 g/m^2, for example from Gieselmann Stanztechnik GmbH, Germany, www.gieselmann-stanztechnik.de

Special fused alumina powder:

- Elektrokorund Alodur SWSK 220 from Treibacher Schleifmittel

- Obtained from PMV (Papierfabrikation und Mechanische Verfahrenstechnik), TU Darmstadt, Alexanderstraße 8, 64283 Darmstadt, Germany

Filter paper:

- Type 1289, Munktell & Filtrak GmbH, Niederschlag 1, 09471 Bärenstein, Germany, www.munktell.com

Ink:

- Pelikan No. 4001 or Parker Quink

DOMAS

- File:

    "ingede4.kls": www.ingede.org

- Software

    "DOMAS Calibration Tester", PTS Heidenau and Munich

- Scanner:

    DOMAS ScannerAdvanced, PTS Heidenau and Munich

- Image analysis software:

    DOMAS 3.0, PTS Heidenau and Munich

SIMPALAB

- File:

    "ingede4.cfg"

- Software:

    Simpalab, current version 3.02.00 Techpap SAS Grenoble

- Scanners:

A list of compatible scanners is available from Techpap SAS Grenoble (www.techpap.com, sales@techpap.com).

Annex A

Size classification

Definition of the size classes of an equivalent diameter of a circle:

from (µm)	to (µm)
>100	≤ 200
>200	≤ 300
>300	≤ 400
>400	≤ 500
>500	≤ 600
>600	≤ 1 000
>1 000	≤ 1 500
>1 500	≤ 2 000
>2 000	≤ 3 000
>3 000	≤ 5 000
>5 000	≤ 10 000
>10 000	≤ 20 000
>20 000	≤ 50 000
>50 000	≤ 200 000

For use within DOMAS software the size classification is determined by file "ingede4.kls"(see software attachment).

For use within Techpap SIMPALAB Software the size classification is determined by file "ingede4".