

1 Purpose and scope of application

This INGEDE method is used to analyse macro stickies in undeinked or deinked pulp (DIP). It is based on a laboratory screening procedure, where the reject is prepared after screening in such a way that an image analysis of the adhesive impurities can take place.

2 Equipment**2.1 Disintegration**

Any device which fulfils the requirements of ISO 5263 may be used for disintegrating dewatered samples.

2.2 Sorting

Macro stickies can be separated from recycled pulp suspensions using various laboratory sorting devices. Possible sorting devices are the Haindl classifier (ZM V/1.4/86), the Somerville tester (TAPPI UM 242) or the Pulmac MasterScreen. The use of narrow slots is recommended, e.g. 100 µm.

2.3 Reject dewatering and drying

Any device which fulfils the requirements of ISO 5269/2 or DIN 54 358-T01 may be used for dewatering and drying the dewatered sorting rejects, e.g. the Rapid-Köthen unit. Additionally, an oven which fulfils the specifications of ISO 287 is required.

2.4 Sticky examination

The following testing material may be used:

- Black water-based ink, e.g. Pelikan No. 4001
- Silicone-coated release paper with very good separating capability.
- Filter paper: medium to large pores, medium filtration speed, machine finished, good wet strength, white, e.g. Ederol No. 12, 240 mm diameter.

- Special fused alumina powder: EK white, particle size 220.

(Reference sources see chapter 10)

2.5 Image analysis

An image analysis system comprising a flatbed scanner and a PC with a suitable control and analysis program is used for the measurements. The flatbed scanner should work according to the reflectance principle with a minimum resolution of 600 × 600 dpi. The software must be able to detect white particles on a black background.

3 Sampling and sample preparation

Pulp suspensions up to a consistency of 10 % can be used immediately for screening without further preparation. However, deinked pulp with higher consistency must be disintegrated before screening. Disintegration takes place in accordance with ISO 5263, whereby the disintegration process is restricted to five minutes. Longer periods of mechanical stress should be avoided in order to avoid changes of the sticky size distribution in the sample.

4 Procedure

The overall procedure is depicted in Figure 1.

4.1 Sorting

When using the Pulmac MasterScreen, 50 g of deinked pulp is added to the supply chest. The sorting which follows is automatic. Before sorting, a wet filter paper which retains the reject when screening is complete, must be placed onto the sieve in the dewatering unit (autofilter).

When using the Haindl classifier, screening takes place as per the "simultaneous measurement of shives and fibre fractions" procedure as per ZM V/1.4/86, but without connecting to the McNett unit. In order to guarantee problem-free sorting of 50 g of oven-dry deinked 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 size of the cylindrical supply vessel wall should be increased from 130 mm to 370 mm. The container can be extended using a Plexiglas top. The washing water flow should be 10 litres per minute for the entire screening duration. After continuously adding pulp for 5 minutes, the deinked pulp continues to be screened for 5 minutes until screening is complete.

Remark 1: When using a plastic screen, mechanical stress can lead to material fatigue and destruction of the slotted plate. For this reason the use of a metal plate is recommended.

For a statistically sound statement about the macro sticky content, the screening of three individual samples, each containing 50 g of oven-dry material from one sample, is recommended.

Remark 2: Increasing the number of tests leads to insignificant improvements to the precision of the measurements only.

4.2 Dewatering the reject

After screening in the Haindl classifier, all the reject is flushed from the slotted plate into a container using about a litre of water, and then dewatered in the sheet former (Rapid-Köthen model) using a moistened white paper filter above the sheet forming wire. 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).

When using the Pulmac MasterScreen, 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.

4.3 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.

4.4 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. In order to achieve this, the silicone-coated release paper should be removed after drying. 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), so that any excess ink is absorbed. Then the specimen is dried for another 10 minutes (as in chapter 4.3), 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, the specimen is completely covered with a thick 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 a visual inspection takes place. This 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. Since experience has shown that the proportion of these types of particle is small, this manual action only involves a minimum effort.

5 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. If only smaller of the surface of the specimen can be measured, the largest possible measuring area should be selected.

When setting the class limits, the size of the slots in the slotted plate which was used for screening should be used as the lower limit (e.g. 100 µm). Smaller stickies cannot be expected because of the sticky surface increase which is associated with the drying process. All other classes can be varied at will. Only the final class may not have an upper limit, so that all stickies are recorded.

6 Test report

The results of the image analysis should be given in mm² of sticky per m² of specimen. This value should be then converted into mm² of sticky area per kg of deinked pulp (*see eqn 1 below*). Here 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) should be taken into consideration.

The use of 50 g of oven-dry deinked 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 material sample.

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

The following should also be noted in the test report:

- type of screening unit used
- type of slotted plate used
- type of image analysis system used.

7 Sources

Reference was made to the following standards in this method:

ZM V/1.4/86: Simultaneous determination of shives and fibre fraction content (in German)

ISO 5263: Pulps – Laboratory wet disintegration

ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Koethen method

DIN 54 358-T01: Manufacture of laboratory sheets for physical testing – Rapid-Koethen method (in German)

TAPPI UM 242: Shive content of mechanical pulps (Somerville fractionator)

ISO 287: Paper and Board – Determination of moisture content – Oven drying method

8 Comments

The INGEDE method is based on the INGEDE project 3894 PTS/IfP “*Developing methods for performing quantitative analyses of micro and macro stickies*”.

9 Bibliography

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

10 References

Special fused alumina powder: Obtained from Institut für Papierfabrikation, TU Darmstadt, Alexanderstrasse 8, D-64283 Darmstadt

(eqn 1)

$$\text{macro sticky area in } \frac{\text{mm}^2}{\text{kg}} = \frac{\text{sticky area in } \frac{\text{mm}^2}{\text{m}^2} \times \text{specimen area in m}^2}{\text{amount of material in kg}}$$

Silicone-coated release paper: Herma, Adhesive
Paper Department, Sales Department, Fabrik-
strasse 16, D-70794 Filderstadt
Filter paper: Binzer & Munktell Filter GmbH,
type Ederol No. 12
Ink: Pelikan No. 4001 or Parker Quink.

(see page 5 for Fig. 1, Flow sheet of the method)

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**Analysis of Macro Stickies
in Deinked Pulp (DIP)**

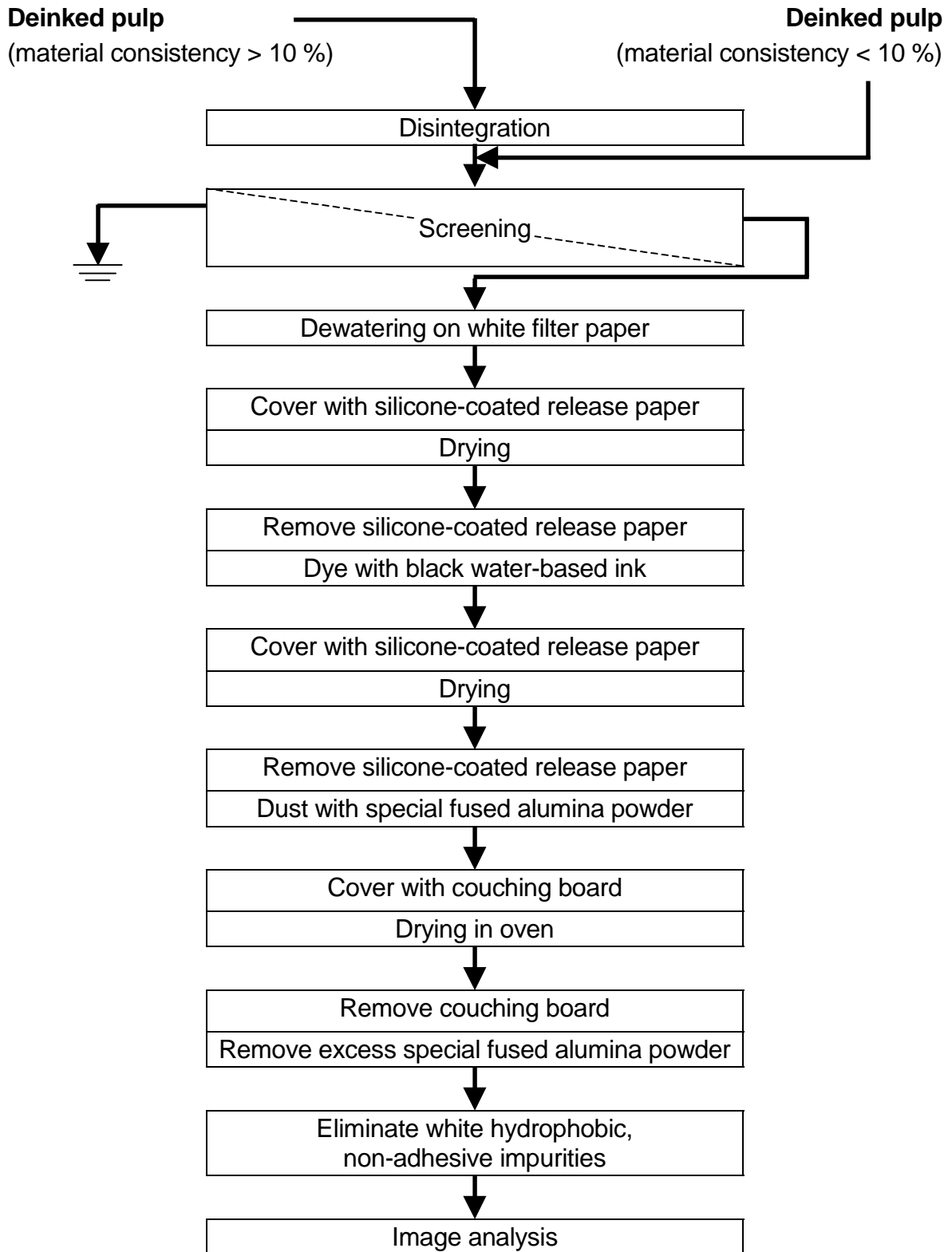


Fig. 1
Flow sheet of the method