

INGEDE Method 17

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7 Pages

Assessment of microsticky test methods



Introduction

Stickies belong to unwanted substances in recovered paper pulps. They originate from adhesive material in the recovered paper. Large macrostickies are removable mainly by means of screening systems equipped with fine slots. Small macrostickies and microstickies remain in the pulp to a significant extent, have the ability to form secondary stickies and thus cause big problems during paper production and converting as well as quality defects.

The testing of macrosticky content is established with INGEDE Method 4. But there is no reliable method for testing microstickies. A harmonised procedure to assess microsticky methods will help in the classification and development of a method for measuring the concentration of microstickies. Quantitative sticky evaluation methods are necessary to provide clear and repeatable information on the stickies content of recycled pulp. It is an essential prerequisite for a successful sticky control to reduce sticky problems in mills and consequently increase their productivity.

The described procedure is also intended as a tool for paper mills wanting to assess their own in-house methods.

1 Scope

This INGEDE Method describes a procedure to evaluate the efficiency of microsticky test methods. It serves to characterize methods of measuring the microsticky content regarding their ability to detect the presence of microstickies in recovered paper pulps and identify them.

2 Terms and definitions

The terms macrosticky and microsticky refer to INGEDE Method 12 and to the ZELLCHEMING Technical Leaflet RECO 1, 1/2006 "Terminology of Stickies". The following definition is an excerpt of this ZELLCHEMING Leaflet.

Primary stickies are introduced with the raw material recovered paper, while secondary stickies are formed by physico-chemical reactions during the paper production process.

The distinctive feature between macro- and microstickies is their screenability behaviour under standard testing conditions, which is usually determined using the slot width of a laboratory screen as a separation criterion. Recommended is a slot width of 100 µm for recycled fibre pulp for graphic paper applications. Stickies found in the screening residues are macrostickies, whereas the sticky components present in the accepts of the screen are referred to as microstickies.

3 Principle

The principle of the procedure for assessing the efficiency of microsticky measurement methods is based on the preparation of stock samples that have defined incremental (stepwise) shares of microstickies (= a predefined series of samples whose microsticky content decreases from one sample to the next). The individual samples are to be tested according to the microsticky measurement method to be evaluated. If the microsticky concentration measured in the sample series reflects the decrease adequately, the method can be considered suitable for verifying the presence of microstickies. The ideal case would show a linear decrease. A curve, that does not show a strict linear decrease, but a clear decline, could be accepted too. If constant or increasing concentrations are found across the mixing stages, the method is to be considered as not suitable for the correct measurement of the microsticky concentration. Of course the microsticky method to be evaluated should be in accordance with the detection criteria of the definitions specified in the ZELLCHEMING Technical Leaflet RECO 1, 1/2006 "Terminology of Stickies".

4 Equipment and auxiliaries

4.1 Equipment

- Analytical balance up to 1 000 g having an accuracy of $\leq \pm 0.001$ g
- Analytical balance up to 3 000 g having an accuracy of $\leq \pm 0.1$ g
- Balance, capable of weighing the dried mat of fibres with an error of less than 0.1 %
- Drying oven (temperature of the drying oven shall be $105\text{ °C} \pm 2\text{ °C}$)
- Filtering device, such as a Büchner funnel (diameter 90 mm to 150 mm)
- Standard disintegrator according to ISO 5263
- Distribution container holding 10 l
- Beakers
- Desiccator
- Weighing container

4.2 Test material

Representing the sticky-loaded pulp:

- Recycled fibre pulp

Representing the sticky-free pulp:

- Wood free, virgin fibre based copy paper or:
- Bleached short fibre chemical pulp

5 Procedure

5.1 Sampling and Sample Preparation

To produce the sample batches containing different amounts of microstickies, a sticky-free pulp and a sticky-loaded pulp are necessary and have to be mixed in different proportions.

Prerequisites:

- Each batch of samples has to contain a defined proportion of microstickies according to the fractions of the sticky-loaded and sticky-free pulps. It has to be made sure that the pulp system does not change in the course of time.
- The samples should contain a representative microsticky load. As they occur in industrial stock or sticky-loaded pulp it is not recommended to use model substances / model stocks, since the characteristics of microstickies formed during the recycling process are not always comparable to the characteristics of adhesives and binding agents used as model substances.
- It should be ensured, that the concentration of microstickies in pulp A (sticky-loaded) and its mixtures with pulp B (sticky-free) will meet the concentration limit, which can be detected by the microsticky measurement method to be evaluated.

5.2 Samples

5.2.1 General

Sample A: sticky-loaded pulp

The sample should be prepared from a suitable microsticky-loaded recycled fibre pulp (RCF sample from industrial production) containing a relatively high share of microstickies. It is recommended to use a RCF pulp at a consistency level close to that of the pulp mixture to be analysed (1 % o. d.) to avoid dilution and reduction of the microsticky levels respectively (avoiding excessive dilution in the laboratory).

The total sample amount shall be sufficient for preparing the series of incremental pulp samples for at least 4 single tests of the microsticky content. The minimum amount of total RCF pulp depends on the material needed for one test of the microsticky test method to be assessed. The consistency of the pulp sample is to be determined according to ISO 4119.

Sample B: sticky-free pulp

Woodfree copy paper

The sample is prepared according to ISO 5263. Respectively 30 g oven dry paper is torn into pieces of 25 mm x 25 mm and soaked for 4 hours at least. The overall amount necessary to cover all pulp mixtures depends on the volume of the mixture and the microsticky measurement method to be evaluated.

Bleached short fibre chemical pulp

the sample is to be prepared from a chemical pulp which is guaranteed not to contribute to any microsticky formation (e.g. chemical pulp of very low content of extractives). To ensure reliable macro sticky measurements, a short fibre pulp is to be used. It may be procured via the PMV,

TU Darmstadt (see chapter 7.4).

The chemical pulp is to be conditioned and the dry matter content is determined according to ISO 638:1978.

The amount of sticky loaded and sticky free pulp necessary to cover all pulp mixtures to be analysed has to be calculated, considering 4 single tests for any of the incremental pulp mixtures analysed with the microsticky measurement method to be evaluated. The consistency of the pulp sample is to be determined according to ISO 4119.

5.2.2 Preparation of sample A: sticky- loaded pulp sample

The Sample A is diluted with tap water to a consistency of about 1 %. The hardness of the tap water to be used for all dilution steps should range between 15 °dH and 20 °dH.

5.2.3 Preparation of sample B: sticky-free pulp sample

The pulp or the soaked wood free, virgin copy paper has to be desintegrated in the laboratory in compliance with ISO 5263-1:2004. The pulp is not to be refined and will be diluted with tap water to 1 % stock consistency.

5.2.4 Preparation of the different sticky-containing pulp samples (sample batches)

According to the principle of the test procedure definite mixtures of sticky-free and sticky loaded pulps are to be prepared and analysed by the microsticky measurement method to be evaluated. The microsticky-loaded pulp (sample A) is mixed with the microsticky-free pulp (sample B) at the defined ratios given below. Both pulp samples are brought to a stock consistency of 1 % and mixed in an appropriate container.

Five sample batches are to be prepared with the following incremental amounts (mixing ratios):

- 100 % sample A + 0 % sample B
- 75 % sample A + 25 % sample B
- 50 % sample A + 50 % sample B
- 25 % sample A + 75 % sample B
- 0 % sample A + 100 % sample B

The mixtures of the suspensions are based on dry mass. The duration of mixing is set to 10 minutes. Mixing will be done at ambient temperature. It is recommended to use an overall pulp volume of 5 l for mixing in the distribution container (to ensure adequate mixing or homogenisation of the suspension).

5.3 Measurement of microstickies using the microsticky method to be evaluated

The 5 sample batches with different microsticky ratios are then analysed according to the microsticky method to be evaluated. The measurements must be done immediately after the preparation of samples. The number of repetitions should be in accordance with the method to be evaluated. In case of doubt, multiple measurements should be preferred.

5.4 Analysis

The evaluation should give information whether the assessed microsticky measurement method allows to adequately differentiating between the pulp mixtures, and whether a precise trend is visible regarding the microsticky content of individual pulp mixtures.

The microsticky measurement method can only be considered suitable if the obtained results correspond with the portion of microstickies contained in the particular mixtures. The ideal case would show a linear decrease with reduced proportions of the sticky-loaded pulp in the various mixtures. But as mentioned, a curve which shows a clear decline instead of a strict linear decrease, is also acceptable. If a decrease is evident, it can be assumed that the microsticky measurement method is suitable. If constant or increasing values are found across the mixing stages, the method should be considered as unsuitable for a correct measurement of microstickies.

A graphic presentation of the results depending on the proportion of the sticky-loaded pulp is helpful to observe the trend of the microsticky decrease:

- Calculate the theoretical concentration of microstickies present in the different stepwise mixtures and indicate the calculated values (calculation based on the values for 100 % sample A and 100 % sample B).
- Calculate the differences between measured and calculated values. Indicate the differences in % (an example is given in Fig. 1).
- Compare analysed and calculated values, whereas deviations between results given by the analysis and results given by the theoretical calculation of the microsticky concentrations should be in the range of $\pm 25\%$. This range is considered the limit for the acceptance of the analysing method. If the analytical result is not within the recommended range, check – before making a final evaluation – whether the measuring range of the respective method provides for the necessary differentiation (if necessary, modify and adjust the boundary conditions, e.g. by choosing a suitable sticky-containing stock).

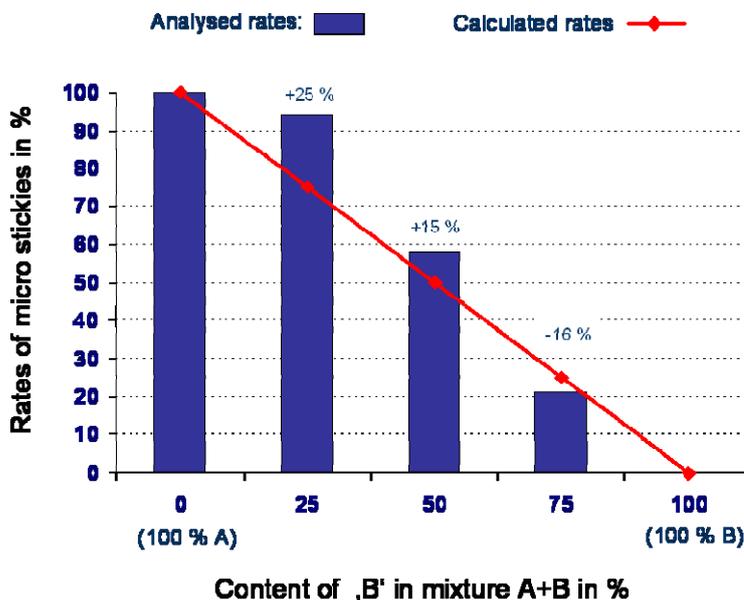


Fig.1 Comparison of analysed and calculated rates of microstickies

6 Report

The following should be recorded in the test report:

- Identification of the type of microsticky measurement method to be evaluated.
- Identification of the microsticky loaded pulp used for preparation of the dilution series (source and consistency).
- Identification of the sticky-free pulp (Recommendation: bleached beech sulphite pulp).
- Results of the microsticky contents of different sample batches with incrementally decreasing microsticky contents: Graphic representation including deviations from the ideal curve, with deviations given in percent.
- Evaluation of the microsticky measurement method investigated.
- Deviations from the conditions stipulated for 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"
- ISO 287 (2009): Paper and Board – Determination of moisture content of a lot – Oven drying method
- ISO 5263-1 (2004): Pulps – Laboratory wet disintegration
- EN ISO 4119:1996: Pulps – Determination of stock concentration

7.2 Literature and other related documents

INGEDE project No. 103 05 PTS/PMV/CTP – Development of a harmonised procedure to assess microsticky methods

7.3 Sources

This INGEDE Method was developed and tested within the scope of INGEDE Project 10305 PTS/PMV/CTP “Development of a harmonised procedure to assess microsticky measurement methods”. The procedure of analysing definite mixtures of sticky-free and sticky-loaded pulps has proved to be a suitable tool to evaluate the efficiency of microsticky measurements.

The dissolved and colloidal material of different pulps may interact and affect the ability of the contaminants present in the pulps to deposit.

7.4 Material sources

Chemical pulp (bleached beech sulphite pulp) obtained from: Chair of Paper Technology and Mechanical Process Engineering (PMV), TU Darmstadt, Alexanderstraße 8, 64283 Darmstadt

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