

1 Purpose and scope of application

This INGEDE method describes the preparation of test sheets made from recovered paper pulps and filtrates from deinking processes for determining optical characteristics. The method covers the preparation of the test sheets as filter pads and also handsheets, the latter both with and without the use of recirculated water. The method can be used for industrial as well as for laboratory samples.

2 Equipment and material

Irrespective of sheet formation type:

- Distribution device (volume: 10 l)

Filter pad formation:

- Büchner funnel with appropriate vacuum device (water jet pump or vacuum pump with moderate vacuum)
- Filter paper: intermediate pores, e. g.: Whatman Grade 41, www.whatman.com
- Analytical balance up to 3,000 g having an accuracy of at least ± 0.1 g

For filter pads and retention of water based inks:

- Cationic Polyacrylamide (CPAM) – high molecular weight, low cationic charge – a polymer used for sludge dewatering. Use the CPAM as solution of 1 g/l concentration (powder diluted in tap water).

Laboratory handsheet formation:

(according to ISO 5269-2, DIN 54 358 part 1 or Zellcheming leaflet V/19/63)

- Standard sheet former (model: Rapid-Köthen) with dryer (vacuum 95 kPa, 94 °C)
- Paper cover sheets and carrier boards according to ISO 5269-2

Sample preparation from filtrate samples:

- Vacuum filtration unit with 39 mm bottom inside diameter of the funnel
- Water jet pump or vacuum pump
- Exsiccator
- Cellulose nitrate membrane filter (from Sartorius, \varnothing 50 mm), pores \varnothing 0.45 μ m.

Remark: If different sizes of funnel and filter paper are used, the sample volume defined in chapter 3 has to be adapted accordingly.

3 Sampling and sample preparation

3.1 Pulp samples

A sample should be analysed in the laboratory after sampling a representative quantity of material at the relevant recovered paper processing stage or taking a sample from laboratory deinking tests. The consistency of the material should be measured according to DIN EN ISO 4119.

After the consistency of the material has been measured, the sample is diluted and homogenised to a consistency of 8 g/l in a distribution device. After the consistency has been measured again, a sample can be taken for preparing the test sheet. No pH adjustment is required.

Pulp suspensions up to a consistency of 10 % can be used immediately for sheet preparation 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. At a consistency of 2 % periods of mechanical stress should be held short in order to avoid changes in size distribution of unwanted particles, e. g. ink and stickies.

3.2 Filtrate samples

The filtrates which are produced when manufacturing filter pads for the measurement of optical properties of deinked pulp as described in chapter 4.1.1 of this method are used to prepare the membrane filter samples. There, the filtrates obtained by dewatering 4 g of oven-dry material (in 1 litre of suspension) using a filter paper are taken completely, homogenised and 100 ml of them is used for further processing. Special attention should be paid to the fact that precisely 1 litre of suspension volume should be drained. If the Büchner funnels to prepare the filter pads are larger or smaller than 160 mm \varnothing , the volume of the suspension has to be adapted accordingly.

The preparation of the two filter pads for measuring the optical properties of deinked pulp produces two filtrates from each sample.

The filtrate of filter pads prepared with the help of flocculants according to chapter 4.1.2 must not be used for optical evaluation.

4 Procedure

4.1 Pulp samples

At least two test sheets should be made.

4.1.1 Filter pads

A basis weight of 225 g/m² should be reached. For each filter pad 4 g of oven-dry material should be used in case of a Büchner funnel of 160 mm diameter. The suspension which is removed is topped up with tap water to a volume of 1 litre. The filter pad is formed using a Büchner funnel which has been covered by a moistened filter paper. After filtering and carefully removing the filter paper, the wet filter pad is laid between two new sheets of filter paper before drying. The drying time in the Rapid-Köthen dryer is 10 minutes.

It can be useful to take two filter papers in order to avoid marks by the funnel holes. The pad is then pre-dried together with one filter still stacking on it for 7 min. After rewetting the filter, it can be peeled off without destroying the wire side of the filter pad. It follows a second drying period in the vacuum dryer without the already used filter paper but between two new sheets of filter paper.

The dried filter paper should not be removed from the filter pad until immediately prior to measuring the optical properties.

The filtrate from the filter pad preparation can be collected and used for optical assessment of filtrate quality according to chapter 4.2 of this method. In that case only one filter paper may be used during filtration.

4.1.2 Filter pads for better retention of water based inks

Dilute the pulp to approximately one litre in order to make a homogeneous pad. For pulp containing water based inks the adjustment of the pH is required. Mix during the pH adjustment (approximately 1 minute) and adjust to pH 6 by adding drop by drop $\text{Al}_2(\text{SO}_4)_3$ (330 g/l). Add 5 ml of the flocculant solution.

4.1.3 Laboratory handsheet formation – General procedure

An appropriate volume of material should be taken from the distribution device for each handsheet. After standard laboratory handsheet formation has taken place, drying takes place in the Rapid-Köthen dryer between carrier board and a cover sheet. The drying time should be 7 minutes. The carrier board and the cover sheet should not be removed from the handsheet until immediately prior to measuring the optical characteristics.

4.1.4 Handsheets for determination of the dirt particle area A

Handsheets for the determination of the dirt particle area (A) are prepared with fresh water in order to reach better contrast for the optical analyses. Grammage m_A should amount to approx. 42.6 g/m^2 ($\pm 1.6 \text{ g/m}^2$) (related to oven-dry substance).

4.1.5 Handsheets for the determination of Kubelka Munk parameters

Handsheets for the determination of Kubelka Munk parameters specific light absorption coefficient (k) and specific light scattering coefficient (s) are prepared with recirculated water. Their opacity should not exceed 97 % in the near infrared area.

A homogeneous quantity of suspension corresponding to 1.35 g of oven-dry substance is being taken from the distribution container to prepare a laboratory sheet in compliance with ISO 5269/2, which after dewatering is being removed from the wire section and disposed of. The filtrate obtained in the process (white water) is being retained and used to dilute the next sheet. To increase the concentration of the white water, this procedure is to be repeated for four times without changing grammage by altering the amount of suspension. It is only after formation of the 5th sheet that the dewatered sample is being removed from the wire section and dried between carrier board and cover sheet in the Rapid-Köthen drier (RK drier) for a minimum of seven minutes.

After determination of the actual grammage, the suspension quantity required for sheet formation is modified for the first time so as to obtain a laboratory sheet of a grammage m_A of approx. 42.6 g/m^2 ($\pm 1.6 \text{ g/m}^2$) (related to oven-dry substance).

Note: The above grammage corresponds to a laboratory sheet weight of $1.35 \pm 0.05 \text{ g}$ after RK-drying.

The altered suspension quantity is then used to prepare two more laboratory sheets (sheets 6 and 7) with the concentrated filtrate, which are also dried between carrier board and cover sheet in the RK drier for a minimum of seven minutes. To facilitate the following optical measurement, it is recommended to mark top side and wire side.

Prior to optical assessment, the two laboratory sheets have to be conditioned in compliance with DIN EN 20187. The sample grammage to be determined after conditioning to a standard reference atmosphere ought to be approx. 45 g/m^2 . The value is to be rounded off to 0.1 g/m^2 .

Laboratory sheets required to enrich white water may be dried and used for piling to determine reflectance factors R_∞ .

4.2 Filtrate samples

100 ml of the filter pad filtrate is completely drained using a cellulose nitrate membrane filter in a vacuum filtration unit. Then, the membrane filter is removed and dried in an exsiccator. The filtrate from the two filter pads produces one membrane filter sample each.

Reference membrane filters are made in the same way, but using exclusively 100 ml of tap water (without pulp).

5 Analysis

The test sheets should be measured and analysed according to INGEDE Method 2.

6 Test report

The type of sheets used for measuring the optical characteristics should be recorded in the test report.

7 References

- DIN EN 20187: Paper, board and pulps – Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (ISO 187:1990).
- DIN EN ISO 4119: Pulps – Determination of stock concentration (ISO 4119: 1995).
- INGEDE Method 2: Measurement of optical characteristics of pulps and filtrates from deinking processes.
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing, Part 2: Rapid-Köthen method.
- ZM V/19/63: Preparation of samples for measuring the whiteness of cellulose materials (in German).

8 Sources

This method has been published for the first time in 1997. A major revision was done according to the definitions made in INGEDE Project 85 02 CTP/PMV/PTS – European Deinkability Test Method. In 2006, also parts of the INGEDE Methods 3 and 10 were transferred to this method.

Contact:

INGEDE e. V. (International Association of the Deinking Industry)
Office
Gerokstr 40
74321 Bietigheim-Bissingen, Germany

Tel. +49 7142 7742-81
Fax +49 7142 7742-80

E-Mail office@ingede.org
www.ingede.org