INGEDE Method 1 **December 2014** 6 Pages	**Test sheet preparation of pulps and filtrates from deinking processes**	INGEDE International Association of the Deinking Industry

Introduction

Pulp made of paper for recycling typically contains printing inks which influence its optical properties. Cleaning and flotation remove small impurities and printing inks whereby the removal efficiency depends also on the printing process applied. For the determination of the residual ink content, the reflectance of light in the near infrared region is measured. The reflectivity of light gives indication of the fine and filler content which influences the light scattering coefficient and of the ink content that alters the light absorption coefficient. The calculation of scattering coefficients requires paper specimens with an opacity less than 95 % (ISO 9416) that is fulfilled best by machine papers.

Pulp samples taken along the deinking process or pulp samples from deinkability tests (INGEDE Method 11) have to be treated. This INGEDE Method describes the preparation of filter pads where fine and ink losses during preparation are negligible. Filter pads are opaque which hinders the calculation of the light scattering coefficient s. Assuming a constant light scattering coefficient is not a recommended approach due to the fact that light scattering varies from pulp to pulp, for example, when the ash content changes. INGEDE Method 1 therefore describes the preparation of handsheets using recirculated water. The method can be used for industrial as well as for laboratory pulp samples.

1 Scope

This INGEDE method is used to prepare test sheets and filter pads of pulps from the deinking process and laboratory samples.

2 Principle

For testing purposes, filter pads are prepared from industrial or laboratory pulp samples using a Büchner funnel and defined filter paper. Handsheets are prepared using the Rapid-Köthen method from industrial pulps under defined conditions. The filtrate samples are drained over a membrane filter and compared with a reference membrane filter made with tap water.

Optical measurements are conducted according to INGEDE Method 2.

3 Equipment and auxiliaries

3.1 Equipment

- Distribution device (volume: 10 l)
- Büchner funnel with appropriate vacuum device that allows a pressure difference ≥ 60 kPa
- Filter paper: Munktell type 1289
- Analytical balance up to 3000 g having an accuracy of at least ± 0,1 g

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

Filtrate darkening:

- Cellulose nitrate membrane filter: Sartorius type 11306-050N, ø 50 mm, pore ø 0,45 µm
- Vacuum filtration unit with 39 mm bottom inner diameter of the funnel
- Water jet pump or vacuum pump
- Desiccator

3.2 Chemicals

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

4 Samples

4.1 Pulp samples

A sample should be analysed in the laboratory after taking a representative quantity of material from the relevant recovered paper processing stage or taking a sample from a laboratory deinking test. The consistency of the material should be measured according to 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 sheet forming. Disintegration takes place in accordance with ISO 5263-2, 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 the size distribution of unwanted particles, e. g. ink and stickies.

4.2 Filtrate samples

The preparation of filter pads for measuring the optical properties generates filtrates which are used to prepare the membrane filter samples afterwards. The preparation of the two filter pads produces two filtrate samples from each pulp sample.

5 Procedure

5.1 Filter pads

At least two filter pads are prepared of the pulp samples respectively.

The filter pad is formed using a Büchner funnel which has been covered by a moistened filter paper. The prepared filter pads have a basis weight of 225 g/m². A filter paper diameter of 150 mm and a maximum Büchner funnel diameter of 160 mm are recommended. In this case, 4,0 g oven-dry pulp material is used and the suspension is topped up with tap water to a volume of 1 litre.

Other filter diameters may be used referring to table 1. The diameter of the Büchner funnel corresponds to the filter diameter and should not exceed the maximum value in table 1. Usually, Büchner funnels are purchased by their nominal diameter that is identical with the filter diameter.

If a differing size of Büchner funnel and filter paper are used, the sample volume has to be adapted according to table 1. The consistency of the pulp sample remains 0,4 %.

Table 1: Pulp volume for Büchner funnel filtration

Diameter max Büchner funnel in mm	Diameter filter paper (Munktell 1289) in mm	Oven dry material in g	Sample volume at 0,4 % consistency in ml
120	110	2,15	538
135	125	2,75	688
160	150	4,00	1000
195	185	6,10	1525

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

Experience has shown that the support of a thin wire made of nylon helps to avoid marks. For this purpose use a nylon wire with a mesh width of about 140 µm and a mesh diagonal of about 190 µm and place it under the filter paper. This option is allowed when preparing the filter pads, but not if the filtrate is analysed for filtrate darkening. For optical assessment of the filtrate quality according to chapter 5.5 collect the filtrate obtained from filter pads prepared with one filter paper.

5.2 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, dry the sheet 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.

5.3 Handsheets for determination of the dirt particle area A

At least two handsheets for the determination of the dirt particle area (A) are prepared with fresh water in order to achieve better contrast for the optical analyses. Grammage m_A should amount to 42,6 g/m^2 ± 1,6 g/m^2, related to oven-dry substance.

5.4 Handsheets for the determination of Kubelka Munk parameters

Handsheets for the determination of the Kubelka Munk parameters specific light absorption coefficient (k) and specific light scattering coefficient (s) are prepared with recirculated water. Their opacity should not exceed 95 % in the near-infrared area.

A homogeneous suspension sample 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. After dewatering it is removed from the wire section and either disposed of or used as laboratory sheet for piling to determine the reflectance factor R_∞. 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 repeated four times without changing the oven-dry substance. The fifth sheet is removed from the wire section and dried between carrier board and cover sheet in the Rapid-Köthen drier for at least seven minutes. Determine the grammage of the sheet.

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 42,6 g/m^2 ± 1,6 g/m^2, related to oven-dry substance.

Note: The above grammage corresponds to a laboratory sheet weight of 1,35 ± 0,05 g after RK-drying.

The adapted suspension sample is then used to prepare two more laboratory sheets (sheets 6 and 7) from the concentrated filtrate, which are also dried between carrier board and cover sheet in the Rapid-Köthen dryer 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 ISO 187. The sample grammage after conditioning in a standard reference atmosphere shall be 45 g/m^2. The value is rounded to 0,1 g/m^2.

5.5 Filtrate samples

The complete filtrate obtained by dewatering the pulp for one filter pad is homogenised. 100 ml filtrate are completely drained using a cellulose nitrate membrane filter in a vacuum filtration unit. Any fibrous material found on the membrane filter may indicate that some pulp bypassed the filter paper when preparing the filter pad. In such a case the membrane filter and filtrate have to be discarded. Prepare a new filter pad and filtrate as described in chapter 5.1.

The filtrate of two filter pads (chapter 5.1) is filtered respectively. Generally, the filtration is done without any retention aids. The result of this filtration must be a discoloured, clear liquid.

Exception:

In case of still having a colourised filtrate after membrane filtration, repeat the procedure with a new sample (100 ml). Add retention aid solution (start with 5 ml) before membrane filtration, possibly alum or cationic polyacrylamide (CPAM) with high molecular weight and low cationic charge. State in the report whether the membrane filtrate was coloured and a retention aid was used; if yes, how much.

The membrane filters are removed from the filtration unit and dried in a desiccator.

Reference membrane filters are made in the same way, but using exclusively 100 ml of tap water without pulp. Prepare a membrane filter for each test series or on a daily basis at least.

6 Report

- The type of handsheets prepared
- Büchner funnel diameter
- Photography of handsheets and filter pads, membrane filtrate and membrane filter
- Filtrate sample preparation with or without retention aid, dosage
- All deviations from the method

7 References

7.1 Cited standards and methods

- INGEDE Method 2: Measurement of optical characteristics of pulps and filtrates from deinking processes.
- ISO 187: Paper, board and pulps – Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (1990).
- ISO 4119: Pulps – Determination of stock concentration (1995).
- ISO 5263-2: Pulps – Laboratory wet disintegration – Part 2: Disintegration of mechanical pulps at 20 °C (2004)
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing, Part 2: Rapid-Köthen method.
- ISO 9416: Paper – Determination of light scattering and absorption coefficients (using Kubelka-Munk theory) (2009)

7.2 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. In 2014 a filter paper was defined based on the results of INGEDE Project 140 13.

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