INGEDE Method 2 January 2007	Measurement of Optical Characteristics of Pulps and Filtrates from Deinking Processes	INGEDE International Association of the Deinking Industry

1 Purpose and scope of application

This INGEDE Method describes a procedure for measuring and calculating various optical characteristics of pulps and filtrates from deinking processes. Parameters defined and described in this method are reflectance factors, light absorption and scattering coefficients, ERIC, colour values and dirt. The method can be used for industrial as well as for laboratory samples.

The method contains the determination of the ink elimination IE based either on ERIC or on the light absorption coefficients of undeinked and deinked pulps.

Particularly when dealing with extremely fine dispersed printing ink particles (e.g. water based printing ink) in the deinked pulp, the filtrate analysis method allows to assess possible pollution levels which may occur in the water systems of deinking plants.

2 Terms and definitions

ERIC: Effective Residual Ink Concentration, calculated as the ratio of the absorption coefficient k of a pulp or paper sample divided by the absorption value of black printing ink and multiplied by 10^6. For black printing ink, a constant k-value of 10,000 m²/kg may be used. For further details please refer to TAPPI T 567 pm-97.

3 Equipment

Any measuring equipment set-up which meets the ISO 2470 (ISO brightness) and ISO 5631 (colour) requirements may be used for measuring.

For the determination of dirt particle area A, a scanner-based system is needed for optical analysis, e.g. DOMAS or Simpatic.

4 Sample preparation

The sample preparation is described in INGEDE Method 1.

The samples have to be conditioned in accordance with ISO 187.

Sample	Description in INGEDE Method 1 (chapter)	Parameters
Filter pad without flocculant	4.1.1	Y, R_{457}, ERIC, IE_{700}, IE_{ERIC}
Filter pad with flocculant	4.1.2	Y, R_{457}
Handsheet without recirculated white water	4.1.4	Dirt Particle Area A
Handsheet with recirculated white water	4.1.5	ERIC, s, k, IE_{700}, IE_{ERIC}
Filtrate sample	4.2	Y, ΔY

5 Procedure for reflectance measurements

5.1 Equipment calibration

5.1.1 Zero point initialisation

A black standard which meets the requirements which are specified in ISO 2469 is used to check the zero point.

5.1.2 Upper limit initialisation

A white standard which meets the criteria described in ISO 2469 is used to set the upper limit.

5.2 Measurement procedure

5.2.1 Sample illumination

The sample should be illuminated with low UV; C/2° conditions are recommended. Alternatively, place a UV blocking filter (420 nm) into the beam of the D65 light source, but this has to be stated as deviation. A second measurement is done with UV included (D65).

5.2.2 Measuring points

Both sides of the test sheets should be measured (filter pads and laboratory handsheets). Care should be taken not to take measurements too near to edges, kinks or on visible non-uniformities of the test sheets.

Membrane filter samples should be measured at the top side only.

5.2.3 Number of measurements

Two samples should be measured in each case. Four measurements are made on each side of filter pads and laboratory handsheets, and just one measurement on top is made on membrane filter samples.

Note: When measuring laboratory handsheets, these ought to be stacked in a way to guarantee an opaque pile of sheets.

5.2.4 Determination of the light absorption coefficient k

The light absorption coefficient k in m^2/kg is then obtained by means of the measured reflectance factors R_0 and R_∞ as well as basis weight according to Kubelka-Munk in compliance with the mathematical equation specified in ISO 9416. (Note: In addition to ISO 9416 where k is obtained by means of the tristimulus filter used to determine reflectance factor Y, the reflectance factor in the case of ink elimination has to be determined at a wavelength of either 700 nm or 950 nm).

5.2.5 ERIC

Strictly speaking, the exact determination of the ERIC value requires the measurement of the scattering coefficient s. For this, thin handsheets made with recirculated water are required. If samples with opacity below 97 % are not available, the ERIC value may be determined using opaque sheets. In that case it is not possible to calculate an exact scattering coefficient. Therefore a fixed value for $s = 42$ m²/kg may be used. Alternatively the scattering coefficient of the deinked pulp in the investigated plant or of the investigated sample may be determined and its value used.

5.2.6 Ink Elimination (IE)

After determining k of the undeinked pulp (UP = undeinked pulp), deinked pulp (DP = deinked pulp) and unprinted pulp (unpr = unprinted pulp) samples, ink elimination IE is calculated as follows:

$$IE \text{ in } \% = \frac{K_{UP} - K_{DP}}{K_{UP} - K_{unpr}} \cdot 100$$

In this equation, k_{unpr} is the specific light absorption coefficient k of a printing ink-free pulp sample. If no unprinted samples are available, the value for k_{unpr} may be set to 0.

5.2.6.1 IE_{700}

$$IE_{700} \text{ in } \% = \frac{\left(\frac{(1-R_{\infty,UP})^2}{R_{\infty,UP}}\right) - \left(\frac{(1-R_{\infty,DP})^2}{R_{\infty,DP}}\right)}{\left(\frac{(1-R_{\infty,UP})^2}{R_{\infty,UP}}\right) - \left(\frac{(1-R_{\infty,unpr})^2}{R_{\infty,unpr}}\right)} \cdot 100$$

The R_∞-values measured at 700 nm on filter pads of deinked pulp (DP) and undeinked pulp (UP) are not used in %, but as absolute values, e. g. 0.69. If no unprinted samples are available, the value for the term $(1–R_{\infty,unpr})^2/ R_{\infty,unpr}$ may be set to 0.

5.2.6.2 IE_{ERIC}

$$IE_{ERIC} \text{ in } \% = \frac{ERIC_{UP} - ERIC_{DP}}{ERIC_{UP} - ERIC_{unpr}} \cdot 100$$

The ERIC values are measured at 950 nm of DP and UP. If no unprinted samples are available, the value for $ERIC_{unpr}$ may be set to 0.

Remark referring to chapter 5.2.6:

If the deinkability of different paper grades is compared, the measurement of the unprinted papers is recommended.

5.2.7 Filtrate Darkening (ΔY)

The filtrate darkening is the difference between membrane filter pads made from filter pad filtrate and reference membrane filter pads (see chapter 4.2 of INGEDE Method 1).

The luminosity of the membrane filter pad and of the reference value is determined at identical conditions. By subtracting Y_{DP} from $Y_{reference}$, ($\Delta Y = Y_{reference} – Y_{DP}$), all factors affecting filtrate quality and not attributable to the pulp are eliminated.

6 Procedure for dirt particle measurement (A)

Evaluation of dirt specks can use TAPPI T 213, DIN 54362-1, ISO 5350-3 or TAPPI T 537. The equivalent black area (EBA) is assessed. As smallest size class 50 µm for equivalent circle diameter has to be set. The measured results of different scanner based image analysis systems are not mutu-

ally comparable. Therefore INGEDE recommends using the DOMAS or Simpatic equipment. Please follow the instructions of the equipment used.

Remark: This chapter will be revised according to the results of the scanner harmonisation project.

7 Analysis

The following values can be used for assessing deinked pulp for filter pads and laboratory handsheets:

- Diffuse blue reflectance factor (ISO brightness R_{457}) according to ISO 2470.
- Luminosity (Y value) according to DIN 53140.
- CIELAB colour coordinates (L*, a*, b* values) according to ISO 5631.
- R_∞ at 700 nm according to DIN 54500 or ERIC according to TAPPI T 567 pm-97.
- Dirt particle area A.

The values should be calculated for the top and bottom of the sheet.

Additionally for laboratory handsheets:

- Specific light scattering and specific light absorption coefficient (s, k) according to ISO 9416
- Opacity according to ISO 2471.

Membrane filter sample:

- ISO brightness R_{457} or luminosity Y for assessing filtrate quality according to ISO 2470 or DIN 53140 respectively.

Note: Samples with strong colour shade should be assessed using chromaticity coordinates rather than ISO brightness R_{457}.

8 Test report

When measuring laboratory handsheets and filter pads, where separate top and bottom side measurements are made, the mean of the two values should always be shown. If the top and bottom values differ considerably, the individual values should also be shown.

The following should be noted in the test report:

- The type of sheet formation the optical measurements refer to (laboratory handsheet or filter pad),
- the amount of UV with which the sample was illuminated,
- the inspection angle and the type of light for which the values were calculated,
- the light absorption coefficient k in m^2/kg, ERIC or R_∞ of the undeinked and deinked pulp samples and the ink elimination derived in percent.

For filtrate samples, the test report should list the mean of both optical measurements.

9 References

- DIN 53140: Paper and board testing: Determination of standard colour values using the three-range procedure (in German).
- DIN 54358-T01: Preparation of laboratory sheets for physical testing – Rapid-Köthen method (in German).
- DIN 54500: Paper testing: Measuring density-related light scatter and absorption coefficients in fibre materials and paper (in German).
- DIN EN 20 187: Paper, board and pulps: Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (in German).
- DIN EN ISO 4119: Pulps: Determination of stock concentration (in German).
- IFRA Newsshade 2003, IFRA Special Report 1.11.2.
- INGEDE Method 1: Test sheet preparation of pulps and filtrates from deinking processes.
- ISO 187: Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples.
- ISO 2469: Measurement of diffuse reflectance factor.
- ISO 2470: Measurement of diffuse blue reflectance factor (ISO brightness).
- ISO 5269/2: Pulps – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method.
- ISO 5631: Determination of colour (C/2 degrees) – Diffuse reflectance method.
- TAPPI T 567 pm-97: Determination of effective residual ink concentration by infrared reflectance measurement.

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