

Introduction

Optical properties are key parameters for the quality of deinked pulp as well as for determining the efficiency of deinking operations. Parameters and details of the measurement procedures defined and described in this method are reflectance factors, light absorption and scattering coefficients, ERIC, colour values and dirt specks.

The method describes the determination of the Ink Elimination IE based either on the light absorption coefficients or on ERIC 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 assessing possible pollution levels which may occur in the water systems of deinking plants.

1 Scope

This INGEDE method describes procedures for measuring and calculating various optical characteristics of pulps and filtrates from deinking processes by means of filter pads and handsheets. The method is applicable for industrial as well as for laboratory samples.

2 Terms and definitions

IE: Ink Elimination, calculated as the ratio of the difference of the absorption coefficient k of the undeinked and deinked samples to the difference of the absorption coefficient k of undeinked and unprinted samples.

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 $10\,000\text{ m}^2/\text{kg}$ may be used. For further details please refer to TAPPI T 567 om-09 or ISO 22754.

3 Principle

Industrial or laboratory samples of pulp and filtrates in deinking processes are transformed to filter pads and handsheets by means of INGEDE Method 1. This INGEDE Method 2 describes and defines the parameters and the settings of the measurement devices to obtain results for optical characterisation of the samples. The calculation of the Ink Elimination is also part of this method and allows an assessment of the deinking process.

4 Equipment and auxiliaries

4.1 Equipment calibration for reflectance measurements

Any measuring equipment set-up which meets the ISO 2470-1 and ISO 5631-2 (colour) requirements may be used for measuring.

- **Zero point initialisation**

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

- **Upper limit initialisation**

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

4.2 Dirt particle measurement (A)

For the determination of the dirt particle area A, a scanner based image analysis system is needed for optical analysis. The scanner has to be calibrated to ensure reproducibility of the measurements.

Technical requirements of flatbed scanner:

- Scanning area \geq ISO A4
- Optical scan resolution \geq 2000 dpi,
- Colour depth 48 bit,
- Optical density $D_{MAX} \geq 4,0$

Requirements on measuring accuracy of flatbed scanner after warm-up period (see scanner manual) and under scanning conditions (see chapter 5.3)

- Reproducibility of mean grey value (8 Bit) ± 1 (A ISO A4 sample has to be scanned 10 times without any movement of the sample. All mean grey value of total sample area should be within 2 grey values.)
- Deviation of colour value (RGB-8 Bit) ≤ 5 (After calibration a scanned image of IT8-Target shouldn't have more deviation to associated reference file than ± 5 values in every colour channel – R,G,B.)

Suitable scanners: DOMAS Scanner*Advanced*, Techpap proposed scanner

NOTE:

"Scanner*Advanced*" is a name given by PTS to a commercial scanner that was accredited by PTS. This scanner device is delivered with the DOMAS 3.0 version.

The image analysis software is to be parameterised according to the specifications described in chapter 5.9.

Suitable software packages are: DOMAS 3.0 and above image analysis software, SIMPALAB Image Analysis Software.

5 Analysis

5.1 General

5.1.1 Sample preparation

The sample preparation is described in INGEDE Method 1. According to table 1 use either a filter pad or a handsheet to determine the optical parameter; then state in the report, which specimen was measured. The samples have to be air-conditioned in accordance with ISO 187.

5.1.2 Sample illumination for reflectance measurements – edge filter

For deinkability testing according to INGEDE Method 11, the samples are illuminated with C/2° conditions while using the edge filter of 420 nm (UV filter)¹. This applies to all reflectance measurements. Other tests are conducted according to the stated standards.

5.1.3 Measuring points and number of measurements for reflectance measurements

Both sides of the test sheets should be measured (filter pads and laboratory handsheets). Best care and attention is given to avoid measurements too near to edges, kinks or on visible non-uniformities of the test sheets.

Respectively two samples should be measured with four measurements on each side of filter pads and laboratory handsheets. Just one measurement is made on the top side of membrane filter samples.

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

5.2 Overview of measurements

The luminosity Y , R_{457} and the reflectance factors R_{∞} and R_0 , the CIE Lab colour coordinates (L^* , a^* , b^* values) are measured from the samples. The absorption coefficient k and scattering coefficient s and ERIC are determined based on reflectance factors, commonly provided by the measuring device. The Ink Elimination IE is calculated from the absorption coefficients or ERIC of the undeinked, deinked and unprinted samples. The dirt specks particle area A is analysed using a scanner based image analysis system. For more details see the following chapters.

¹ It has been shown that the results with light source C/2° with an edge filter of 420 nm and D65/10° with an edge filter of 420 nm are nearly identical. For this reason and because the method measured originally C/2° it was decided that the measurement is performed with C/2°.

Table 1: Overview of sample types, parameters to be measured and corresponding chapter in INGEDE Method 1

Sample	Chapter in INGEDE Method 1	Parameters
Filter pad	5.1.	Y, R ₄₅₇ , ERIC, IE ₇₀₀ , IE _{ERIC} , L*, a*, b*
Handsheet without recirculated white water	5.3	Dirt particle area A
Handsheet with re-circulated water	5.4	ERIC, s, k, IE ₇₀₀ , IE _{ERIC}
Filtrate	5.5	Y, ΔY

5.3 Reflectance factors

The reflectance R_{∞} is measured with a device according to ISO 2469 at a wavelength of 700 nm and 950 nm. R_{∞} is the reflectance factor of a layer handsheets or pad that is thick enough to be opaque.

R_{∞} at a wavelength of 457 nm (brightness) is measured with a device in accordance with the standard ISO 2470-1 ISO brightness.

The reflectance of a single sheet R_0 is measured at 700 and 950 nm with the stated conditions above. In accordance to ISO 9416, the single sheet must meet the requirements, that the opacity must not exceed 95 %. R_0 is the reflectance factor of a single sheet of paper with a black cavity as backing.

5.4 Y, L*, a*, b* and opacity

The luminosity Y is determined in accordance with DIN 6174. The CIELab colour coordinates L*, a* and b* are determined according to ISO 5631-2. From handsheets, the opacity following ISO 2471 is determined.

5.5 Determination of the light absorption coefficient k and the light scattering coefficient s

The light absorption coefficient k in m^2/kg and the light scattering coefficient s in m^2/kg are obtained by means of the measured reflectance factors R_0 and R_{∞} as well as basis weight according to Kubelka-Munk in compliance with ISO 9416. In addition to ISO 9416 where k and s are obtained by means of the tristimulus filter used to determine reflectance factor, the reflectance factor in the case of Ink Elimination has to be determined at a wavelength of either 700 nm or 950 nm.

The use of the method ISO 9416 is restricted to samples with an opacity lower than 95 %, otherwise inaccuracy in calculation of s will occur, for more details see ISO 9416. Therefore it is not

possible to determine s from filter pads or thick handsheets. Handsheets with recirculated water meet possibly the requirements of ISO 9416.

Equation 1: Light absorption coefficient in m^2/kg

$$k = s \cdot \left(\frac{(1 - R_{\infty})^2}{2 R_{\infty}} \right)$$

R_{∞} is expressed as decimal.

Equation 2: Light scattering coefficient in m^2/kg

$$s = \left(\frac{1000}{w} \right) \cdot \left(\frac{R_{\infty}}{1 - R_{\infty}^2} \right) \cdot \ln \frac{R_{\infty} (1 - R_0 \cdot R_{\infty})}{R_{\infty} - R_0}$$

w = grammage (g/m^2)

R_{∞} and R_0 are expressed as decimal.

5.6 Ink Elimination IE

Generally speaking, Ink Elimination is obtained using the light absorption coefficient k of the undeinked, deinked and unprinted samples. For known scattering coefficient s , Equation 1 gives the determination of k . Ink Elimination (IE) is calculated as follows:

Equation 3: Ink Elimination in %

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

Where:

UP = undeinked pulp

K_{UP} = light absorption coefficient k of undeinked sample

DP = deinked pulp

K_{DP} = light absorption coefficient k of deinked sample

$unpr$ = unprinted sample

K_{unpr} = light absorption coefficient k of unprinted sample

It is assumed that the difference in s of filter pads before and after the flotation is as magnitude as the losses of pulp components during hand sheet preparation. Assuming $s = \text{const}$, Equation 3 can be used simplified by neglecting the light scattering coefficient s , calculating IE only with R_{∞} from filter pads (Equation 4). Alternatively, the scattering coefficient s of the deinked pulp in the investigated plant or of the investigated sample has to be determined for the specific sampling point in the process and its value used as approximation.

Equation 4: Ink Elimination in %

$$IE = \frac{\left(\frac{(1 - R_{\infty,UP})^2}{R_{\infty,UP}} \right) - \left(\frac{(1 - R_{\infty,DP})^2}{R_{\infty,DP}} \right)}{\left(\frac{(1 - R_{\infty,UP})^2}{R_{\infty,UP}} \right) - \left(\frac{(1 - R_{\infty,unpr})^2}{R_{\infty,unpr}} \right)} \cdot 100$$

where:

$R_{\infty,UP}$ = reflectance factor R_{∞} of undeinked sample

$R_{\infty,DP}$ = reflectance factor R_{∞} of deinked sample

$R_{\infty,unpr}$ = reflectance factor R_{∞} of unprinted sample

R_{∞} is obtained either at a wavelength of 700 nm (for IE_{700}) or at 950 nm (for IE_{ERIC}).

- **IE_{700}**

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.

Equation 5: IE_{700} in %

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

- **IE_{ERIC}**

The ERIC values (chapter 5.7) 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.

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

Equation 6: IE_{ERIC} in %

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

5.7 ERIC

The calculation for ERIC follows ISO 22754 and TAPPI T567 om-09:

Equation 7: ERIC

$$ERIC = (k_{Sheet} / k_{ink}) \cdot 10^6$$

R_0 and R_∞ are determined at a wavelength of 950 nm of specimen, which meet the requirements to ISO 9416. If a lightweight sample is not available, make certain that the scattering coefficient s is truthfully representative of the sample being tested. For this case, ERIC value and the value for s has to be stated together in the report.

5.8 Filtrate Darkening

The filtrate darkening ΔY is the difference in luminosity Y between membrane filter pads made from filter pad filtrate and tap water as reference. The preparation of membrane filter pads is described in INGEDE Method 1.

The luminosity Y of the filtrate membrane filter pad ($Y_{filtrate}$) and of the reference membrane filter pad ($Y_{reference}$) are determined at identical conditions according to chapter 5.4. $Y_{filtrate}$ is the average value of the two membrane filter pads (see INGEDE Method 1). By subtracting $Y_{filtrate}$ from $Y_{reference}$, ($\Delta Y = Y_{reference} - Y_{filtrate}$), all factors affecting filtrate quality and not attributable to the pulp are eliminated.

5.9 Procedure for dirt particle measurement (A)

A scanner based image analysis system is to be used for the determination of the dirt specks area "A".

The top and the bottom side of at least two laboratory handsheets per specimen are to be assessed by the image analysis system. The arithmetic mean of min. 4 measured values is to be calculated. This mean value is to be taken as the dirt specks area "A".

Scanning conditions:

The sheets should be free of crinkles and waves to lie flat on the scanner. The sheets are to be scanned individually. As background an opaque batch of woodfree copy paper (min. 5 sheets with a luminosity of $Y = 84 \pm 2$ measured with illumination D65/10° and 420 nm edge filter) should be used. Every handsheet should be scanned one time from top and from the bottom with 8-bit grey modus, 600 dpi and reflective light.

If the scanner is idle for more than 15 minutes a blank scan has to be made in advance of any new measurement.

Image analysis software parameterisation: The threshold values and the size classification are to be defined as described in the appendix.

In case of using DOMAS image analysis system the following parameters are recommended:

- The threshold of measurement is determined by file "ingede2.sw"
- The size classification is determined by file "ingede2.kls"
- Select: "Circular specimen with border" if the specimen is circular
- Select threshold method "file" and select "ingede2.sw"
- Select size classification "circle equivalent diameter" and select "ingede2.kls"
- Select: image source "scan series" and select "specks_1.scn"
- Set "No of specimens" to "4"
- Select: "Average series of results"

In case of using SIMPALAB software (Techpap SAS):

Select the family "Ingede2.cfg", to take pre-installed parameters for the measurement of optical characteristics into account. The threshold of measurement, sizes for classification (50–100 μm , 100–150 μm , ...) and other parameters are already determined in the file "ingede2.cfg" and set automatically.

6 Report

When measuring laboratory handsheets and filter pads, where top and bottom are measured separately, always the mean of the two values should be reported. If the top and bottom values differ considerably, the individual values should also be reported.

The following should be noted in the test report:

- Type of test specimens the optical measurements refer to (laboratory handsheets or filter pads),
- Type of light and the inspection angle for which the values were calculated,
- The light absorption coefficient k in m^2/kg , the light scattering coefficient s in m^2/kg , ERIC or R_∞ at 700 nm of the undeinked and deinked pulp samples and the Ink Elimination derived in %,
- For filtrate samples, the test report should list the mean of both optical measurements (Y_{filtrate} and $Y_{\text{reference}}$).

7 References

7.1 Cited standards and methods

- IFRA Newsshade 2003, IFRA Special Report 1.11.2.
- INGEDE Method 1: Test sheet preparation 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 2469: Paper, board and pulps – Measurement of diffuse radiance factor (diffuse reflectance factor) (2014)
- ISO 2470-1: Paper, board and pulps – Measurement of diffuse blue reflectance factor – Part 1: Indoor daylight conditions (ISO brightness) (2009)
- ISO 2471: Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method (2008)
- ISO 4119: Pulps: Determination of stock concentration (1995)
- ISO 5269-2: Pulps – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method (2004)
- ISO 5631-2: Paper and board – Determination of colour by diffuse reflectance – Part 2: Outdoor daylight conditions (D65/10 degrees) (2014)
- ISO 9416: Determination of light scattering and absorption coefficients (using Kubelka-Munk theory) (2009)
- ISO 22754: Pulp and paper – Determination of the effective residual ink concentration (ERIC number) by infrared reflectance measurement (2008)
- TAPPI T 567 om-09: Determination of effective residual ink concentration by infrared reflectance measurement (2009)

7.2 Sources of the material used

DOMAS

- Files:
“ingede2.sw” and “ingede2.kls”: www.INGEDE.org
- Software:
“DOMAS Calibration Tester”, PTS Heidenau and Munich: www.ptspaper.de
- Scanner:
DOMAS Scanner*Advanced*, PTS Heidenau and Munich
- Image analysis software:
DOMAS 3.0, PTS Heidenau and Munich

SIMPALAB

- Files:
"ingede2.cfg"
- Software:
SIMPALAB_[]_3.00.[1x], Techpap SAS Grenoble
- Scanner:
A list of compatible scanners is available from Techpap SAS Grenoble
(www.techpap.com, sales@techpap.com)

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Annex

Threshold value determination

Use this formula: Threshold = mean – k_factor

Linear interpolation between two pairs of values gives the threshold value demanded.

mean (8-bit grey value)	k_factor
167,42	35,81
202,01	30,43
221,37	30,91
239,17	35,38
248,16	33,75

For use within DOMAS software the threshold of measurement is determined by the file "ingede2.sw"(see software attachment).

For use within Techpap SIMPALAB Software the threshold of measurement is determined by file "ingede2.cfg".

Size classification

Definition of the size classes of an equivalent diameter of a circle:

from (µm)	to (µm)
> 50	≤ 100
> 100	≤ 150
> 150	≤ 200
> 200	≤ 250
> 250	≤ 500
> 500	≤ 50 000

For use within DOMAS software the size classification is determined by file "ingede2.kls" (see software attachment).

For use within Techpap SIMPALAB Software the size classification is determined by the file "ingede2.cfg".