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Recycled pulps — Estimation of stickies and plastics —

Part 3: Determination and identification by applying near-infrared measurement

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

A list of all parts in the ISO 15360 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Tacky particles, commonly named stickies, are a major issue in paper recycling since they can affect the processes as well as the quality of the recycled products. Therefore, a variety of test methods have been developed over decades. Existing laboratory test procedures for sticky contaminants in pulps made from paper for recycling or in recycled papers require an elaborate sample preparation to increase the concentration of these sticky contaminants by screening the pulp through a slotted plate and analysing the rejects. With the test procedure and equipment based on this document, the content of sticky contaminants can be determined at a laboratory handsheet or at the paper by means of near-infrared (NIR) measurement but also on filter paper where stickies have been concentrated and deposited with existing test procedures.

The approach to the measurements specified in this document is different from established test methods because the amount and chemical composition of polymeric substances are determined by applying NIR measurements. The content of these substances, which are typical constituents of adhesives are assigned as stickies. This is a further major difference from existing methods, which typically analyse the sticky behaviour, but not the chemical composition. The results determined by applying the method specified in this document correlate very well with established sticky measurement techniques, e.g. ISO 15360-2 or INGEDE Method 4.[1] The measurement procedure in this document also enables to simultaneously analyse polymeric substances without tacky behaviour, typically called “plastics” in other methods.

The method described here can be implemented by any system that combines particle size measurement and NIR analysis. For example, “3DStick”[2,3] involves laser triangulation to determine size and location of particles and subsequent NIR to determine chemical composition. Alternatively, direct camera imaging in the NIR spectrum has been used to both locate and identify the chemical composition of particles.[4] This method can prove valuable for online measurement in systems that generate dry sheet samples[5].

Recycled pulps — Estimation of stickies and plastics —

Part 3:
Determination and identification by applying near-infrared measurement

1 Scope

This document specifies a method for the determination of stickies and non-tacky polymeric contaminants present in pulp or paper sheets near-infrared measurement. This document is applicable to recycled pulps and papers.

Sampling of pulp and paper as well as the preparation of handsheets are outside the scope of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 216, *Writing paper and certain classes of printed matter — Trimmed sizes — A and B series, and indication of machine direction*

ISO 534, *Paper and board — Determination of thickness, density and specific volume*

ISO 536, *Paper and board — Determination of grammage*

ISO 4119, *Pulps — Determination of stock concentration*

ISO 5263-2, *Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 degrees C*

ISO 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method*

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

ISO 5270, *Pulps — Laboratory sheets — Determination of physical properties*

ISO 15360-1, *Recycled pulps — Estimation of Stickies and Plastics — Part 1: Visual method*

ISO 15360-2, *Recycled pulps — Estimation of Stickies and Plastics — Part 2: Image analysis method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
stickies
diverse group of materials in disintegrated pulp that can adhere to objects at ambient temperature or can adopt adhesive characteristics when subjected to elevated temperature, elevated pressure or change of pH

[SOURCE: ISO 4046-2:2016, 2.61, modified — two instances of the word "may" replaced with "can".]

3.2
non-tacky polymeric contaminant
polymeric substance that is usually not found in adhesives, excluding cellulosic materials and inorganic substances

Note 1 to entry: ISO 15360-2 defines these substances as “plastics”.

4 Principle

This document specifies the determination of the chemical composition of an air dry paper sample applying a near-infrared (NIR) analysis system. The sample to be analysed can have various origins – pulp, paper machine, pilot plant, laboratory etc. – but shall be made using recycled fibres. The surface areas of particles, which are neither natural fibres nor inorganic substances are determined. The measurement differentiates between stickies or non-tacky polymeric contaminants according to their chemical nature.

5 Apparatus (near-infrared analysis system)

5.1 Hardware (measuring device)

The measuring device is a hyperspectral NIR imaging system. The illumination device, optics and sensor shall cover at least the spectral range from 1 400 nm to 1 800 nm. The optical system has a lateral resolution of at least 127 DPI (i. e. object pixel size ≤200 µm). The optical system may be of the wide-field type, or of the point-, line- or wavelength-scanning type.

Annex A details the characteristics of such compatible hyperspectral NIR systems.

It is recommended that the system allows analysing paper sheets of ISO A4 according to ISO 216 format or larger.

5.2 Software

The required software is a NIR evaluation system for qualitative and quantitative assessment of polymeric substances by size and number. The software shall consist of an NIR imaging algorithm, including chemometrics and classification.

For examples, see Annex B.

5.3 Penetration test reference piece

A film or plate with an approximate size of ISO A4 in accordance with ISO 216 and a maximum thickness of 300 µm consisting of at least one polymeric substance, which is assigned to the sticky or non-tacky polymeric contaminants group (see 8.1).

5.4 Calibration of the NIR sensor

Calibration shall be done according to the manual of the respective device. The calibration shall be checked in accordance with Annex A.

6 Preparation of specimen

6.1 General

In the case of determination of plastics and stickies deposited on filter paper, the measurement can be performed directly, without the need of the penetration test defined in 7.4.

In the case of determination on paper sample, the NIR signal can be submitted through a limited specimen thickness only. For pulp or paper sheets, this specimen thickness is depending on the fibre type, the degree of densification, the filler content and the like. Therefore, a penetration test shall be performed to determine whether the thickness of the specimen is below the maximum allowable specimen thickness δ_{max} (see 7.4).

6.2 Pulp samples

6.2.1 Measurement of stickies and non-tacky contaminants deposited on filter

The contaminants shall be isolated in accordance to ISO 15360-1 or ISO 15360-2 and deposited on filter paper before air drying. The oven dry mass of pulp used to concentrate the contaminants shall be determined.

6.2.2 Measurement of stickies and non-tacky contaminants directly on paper or handsheets

Determine the oven-dry mass of the stock used for preparing the handsheets in accordance with ISO 4119.

Prepare handsheets having a thickness of maximum δ_{max} in accordance with ISO 534 by using a sheet former in accordance with ISO 5269-1 or ISO 5269-2. The cumulative area of multiple handsheets shall be a minimum of 0,1 m^2 and a maximum of 0,5 m^2. When statistical results are reported, they should be based on measurements of at least 50 particles, which can require scanning multiple handsheets or paper samples.

EXAMPLE If using a Rapid-Köthen sheet former according to ISO 5269-2, usually four handsheets are prepared.

The polymeric substances, which have a detectable size for the measurement, but are not retained in the handsheet are negligible.

If the penetration test cannot be performed in advance (see 7.4), and therefore δ_{max} is unknown, it is recommended to prepare handsheet samples having a thickness of max. 100 µm for recycled pulps.

When preparing several handsheets from the same pulp sample, the weight of the suspension used for the handsheet forming should be the same for each handsheet.

Determine the oven-dry grammage of the handsheets in accordance with ISO 5270.

When statistical results are reported, they should be based on measurements of at least 50 particles, which can require scanning multiple handsheets or paper samples, the grammage of individual handsheets made from stock samples taken at different positions within the process should not vary more than ±10 % from their average grammage.

6.3 Paper samples

Determine the oven-dry grammage of the paper samples in accordance with ISO 536. The cumulative area of multiple paper samples shall be a minimum of 0,1 m^2 and a maximum of 0,5 m^2. When statistical results are reported, they should be based on measurements of at least 50 particles, which can require scanning multiple handsheets or paper samples. The paper samples can be cut to paper sheets, suitable for the measurements.

To determine the stickie area in mm^2/kg when analyzing paper samples, the following cases shall be distinguished:

— If the paper sheet thickness is $\leq \delta_{max}$, place the paper sheets under the NIR sensor and perform the NIR analysis;

— If the paper sheet thickness is > δ_{max}, pulp the paper samples on laboratory scale in accordance with ISO 5263-2, and prepare handsheets as specified in 6.2. The procedure that shall be followed is the same as specified for pulp samples.

NOTE The pulping can cause size reduction of the particles (stickies non-tacky polymeric contaminants) thus effecting the detectable amount and the size distribution.

7 Procedure

7.1 Parameters for the analysis

Define the size classes for the particles and select the available substances.

Size classes in circle equivalent diameter in accordance with INGEDE Method 4[1] see Table 1.

Table 1 — Size classes in circle equivalent diameter

Dimensions in µm

Size classes	Lower limit	Upper limit
K1	100	200
K2	201	300
K3	301	400
K4	401	500
K5	501	600
K6	601	1 000
K7	1 001	1 500
K8	1 501	2 000
K9	2 001	3 000
K10	3 001	5 000
K11	5 001	10 000
K12	10 001	20 000
K13	20 001	50 000
K14	50 001	200 000

7.2 Placement of specimen

Place the specimen under the NIR sensor and fix it. When analysing laboratory handsheets, place them with the wire side facing up.

If analysing paper sheets ≤ δ_{max}, the side of the paper sample having the first contact with a heated drying cylinder should be facing up. Use the same side facing up for all paper sheets of one paper sample. When several paper samples from the same paper machine are measured, ensure that always the same side of the paper samples are measured by applying the NIR analysis.

If the paper sheet thickness is > δ_{max} the NIR measurements can be performed for only one side facing up, preferably the paper side having the first contact with a heated drying cylinder. The specific stickie area can be determined in relation to the total surface area of the sample.

7.3 Measurement

After placing the specimen under the NIR sensor, start the measurement. The specimen shall be scanned in individual stripes and the individual scans are assembled if using a line scan sensor. If using a matrix sensor, the entire sheet may either be analysed by one shot or by individual rectangular scans that are assembled. If using a single spot sensor, the entire sheet is analysed by 2D point scanning.