SLOVENSKI STANDARD
SIST-TS CEN/TS 17158:2018

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Kompoziti iz materialov na osnovi celuloze in termoplastov (običajno se imenujejo lesni polimerni kompoziti (WPC) ali kompoziti iz naravnih vlaken (NFC)) - Ugotavljanje velikosti delcev lignoceluloznega materiala

Composites made from cellulose based materials and thermoplastics (usually called wood polymer composites (WPC) or natural fibre composites (NFC)) - Determination of particle size of lignocellosic material

Verbundwerkstoffe aus cellulosehaltigen Materialien und Thermoplasten (üblicherweise Holz-Polymer-Werkstoffe (WPC) oder Naturfaserverbundwerkstoffe (NFC) genannt) - Bestimmung der Partikelgröße von lignocellulosehaltigem Material

Composites à base de matières cellulosiques et de thermoplastiques (communément appelés composites bois-polymères (WPC) ou composites fibres d'origine naturelle (NFC)) - Détermination des dimensions de particles de matières ligocellulosiques

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English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TS 17158:2018) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

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Introduction

Either optical systems or sieve analysis can be used for the determination of particle size of wood flour or flour based on alternative lignocellulosic materials for use in wood plastic composites. Microscopic analysis is also possible but requires a significant amount of time and effort to generate statistically viable results, hence, it is not suggested for this task. Particle size determination can be performed using raw material as well as processed and extracted material to observe the effects of processing on particle size. Optical systems and in addition, sieve analysis can be performed using a vibrating sieve or an air-jet sieve in order to match results from optical measurements with those from mechanical measurements and which are suitable to determine the mass of particles which are too fine or too coarse. Sieve analysis does not generate any information regarding the shape of the particles and leads only to a diameter-based, coarse size distribution. Therefore, size parameters supplied by optical measurements cannot directly be compared with those from sieve analysis. Methods using active air-jet dispersion are particularly suitable for material containing long natural fibres which tend to agglomerate. Optical methods derive very similar results regarding the type of distribution and its characteristic percentiles for the particle length and a shape factor (length/width ratio) if optical resolution and weighting method are comparable. For a comparison between these measurement principles see (Plinke et. al 2016 [2]).

This document is related to the determination of particle size of the lignocellulosic material used in the preparation of wood-polymer composites according to EN 15534-1 and prEN 15534-2 for compounds. Furthermore, the methods mentioned in this document can be used to determine the extent of particle degradation after processing, i.e. after compounding and/or extrusion and injection-moulding. Manufacturers of wood and other natural fibre producers and users of wood and natural fibres can use the methods described in this document to determine particle size of their lignocellulosic raw materials and to compare the particle size of their raw materials to alternative lignocellulosics. There are no requirements for particle size of the lignocellulosic fillers used in WPC, however, it is useful to know particle size for formulation development and to understand the relationship between particle size and WPC properties such as strength, water uptake and swelling.

1 Scope

This document specifies mechanical and optical test methods for the determination of particle size of lignocellulosic material for use in wood plastic composites (WPC) and natural fibre composites (NFC).

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.

EN ISO 4610, *Plastics - Vinyl chloride homopolymer and copolymer resins — Sieve analysis using air-jet sieve apparatus (ISO 4610)*

ISO 9276-6:2008, *Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology*

ISO 13322-2, *Particle size analysis — Image analysis methods — Part 2: Dynamic image analysis methods*

DIN 66165-1:2016, *Particle size analysis — Sieving analysis — Part 1: Fundamentals*

DIN 66165-2:2016, *Particle size analysis — Sieving analysis — Part 2: Procedure*

3 Principle

The determination of particle size is carried out either mechanically using the

— **vibrating sieve analysis;** or the

— **air jet sieve analysis;**

or optically based on automatic image analysis systems

— after manual dispersion on transparent foil and image acquisition using a flat-bed scanner (designated here as "**static image analysis system**");

— or after automatic dispersion by jet air and image acquisition using a CMOS-camera (designated here as "**dynamic image analysis system**").

4 Apparatus

4.1 Vibrating sieve analysis

The vibrating sieve analysis is carried out according to DIN 66165-1:2016, procedure F (vibrating screen in static air).

The number and opening widths of the sieve inserts shall be suitable for the material under investigation. It is recommended that this apparatus is used for an expected size range between 100 µm and 20 000 µm (particle length) and for material which does not tend to agglomerate. The opening widths of the sieve inserts shall be chosen from DIN 66165-1:2016, Table 2 so that the expected size range is covered.

4.2 Air jet sieve analysis

The air jet sieve analysis is carried out according to EN ISO 4610.

The number and opening widths of the sieve inserts shall be suitable for the material under investigation. It is recommended that this apparatus is used for an expected size range between 20 µm and 2 000 µm (particle length). The opening widths of the sieve inserts shall be chosen from DIN 66165-1:2016, Table 2 so that the expected size range is covered.

4.3 Static image analysis systems

An automatic image analysis system with evaluation software after manual dispersion on transparent foil and picture recording with flat-bed scanner using a flat-bed scanner with transparency unit and an automatic feeder[1].

NOTE Product versions without feeder are less suitable due to material handling.

4.4 Dynamic image analysis systems using automatic dispersion by jet air and image acquisition with a CMOS-camera

An automatic image analysis system with evaluation software after automatic dispersion by jet air using a dry disperser and a vibratory feeder and picture recording with a CMOS-camera[2].

5 Calibration

5.1 Vibrating sieve analysis

No calibration is necessary if the manufacturer's recommendations for operation are met.

5.2 Air jet sieve analysis

No calibration is necessary if the manufacturer's recommendations for operation are met.

5.3 Static image analysis systems

Perform a scanner test and calibrate the system with respect to image quality and gray value thresholds according to the manufacturer's instructions, using a set of spheres with a uniform diameter of 600 µm. No more calibration is necessary for the scanner's geometry. It is recommended to frequently verify the measurement and evaluation chain using synthetic flock fibres with a known and narrow length distribution (see Fischer et al. 2016 [3]).

5.4 Dynamic image analysis systems

Calibrate the system using a suitable reference material in order to verify the accuracy of the measurement. Verify the threshold values and optical sensors of the system. Calibrate, verify, and clean the system as frequently as necessary.

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[1] The product "FibreShape" (manufacturer: IST Ag, Vilters, Switzerland) in the version FibreShape Basic Automatic (using a flat-bed scanner with transparency unit and an automatic feeder) together with evaluation software supplied by the manufacturer is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

[2] The product "QICPIC" (manufactured by Sympatec, Clausthal, Germany) with dry disperser "RODOS/L" and vibratory feeder "VIBRI/L" together with evaluation software supplied by the manufacturer is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

6 Procedure

6.1 Sampling and preparation of test specimens

6.1.1 General

For all methods the sample has to be dry. For most materials mechanical sub-sampling is not possible. Therefore, the operator has to ensure that the sample is representative of the material. If the result appears questionable it is recommended to perform three measurements with independently drawn samples.

6.1.2 Vibrating sieve analysis

Perform the measurement according to DIN 66165-2. It is recommended to use an initial mass of at least 50 g.

6.1.3 Air jet sieve analysis

Perform the measurement according to EN ISO 4610, but in one run for each sieve insert. It is recommended to use an initial mass of at least 50 g. Start with the sieve insert with the smallest opening width, and determine the mass of the throughput fraction from the difference between the initial weight and the sieve retention. Repeat the procedure with the sieve retention for each fraction to be determined.

6.1.4 Static image analysis systems

Use a parameter set for measurement with maximum resolution but activate the option to exclude agglomerations. Disperse an appropriate quantity of particles manually on the foil of the sample feeder using a hand sieve with appropriate opening width. Make sure that that the area coverage of particles on the image field does not exceed ca. 2 %. If the material tends to agglomerate, those agglomerations cannot be avoided totally but try to minimize them as much as possible. Set the automatic sample feeding option to three image fields and start the measurement.

6.1.5 Dynamic image analysis systems

Disperse an appropriate quantity of particles on the vibratory feeder and/or inlet hopper. In accordance with ISO 13322-2, select a suitable measurement range between size range from approx. 20 µm to 5 000 µm (dry-dispersion). Depending on the size and shape of the particles, a total mass of particles between 2 g and 20 g is recommended.

6.2 Observation and measurement

6.2.1 Vibrating sieve analysis

Each particle of the samples passes one or more sieves of the stack, driven by vibration and their own weight. The mass of particles retained on each sieve insert and additionally the mass of fine particles are the readings to be used for the report.

6.2.2 Air jet sieve analysis

Each particle of the sample can pass the sieve in the current run, driven by air pressure and turbulence, or remain on the sieve. The mass of particles passing the sieve insert in each run and additionally the mass of coarse particles after the last run are the readings to be used for the report.

6.2.3 Static image analysis systems

The particles are dispersed manually on the transparent foil of the feeder. Keep the foil clear from dust and avoid air turbulence around the device. A stepper motor moves the foil stepwise to cover the scan