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Test methods for discrete polymer fibre for fibre-reinforced cementitious composites

Méthodes d'essai des fibres polymères distinctes pour les composites à base de ciment renforcés par des fibres

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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 documents 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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 71, *Concrete, reinforced concrete and pre-stressed concrete*, Subcommittee SC 6, *Non-traditional reinforcing materials for concrete structures*.

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

Polymer fibre in this document means a fibre made with macromolecule substances as raw material such as aramid fibre, polyamide fibre, polyester fibre, polyethylene fibre, polypropylene fibre, polyvinylalcohol fibre. For fibre-reinforced cementitious composites (FRCC), many kinds of and types of polymer fibres are designed and produced on various demands. However, standards of discrete polymer fibres for FRCC in the civil engineering field are not specified yet despite the need for it.

If the fibre suppliers can show the principal fibre properties such as geometry and standardized basic mechanical properties, the engineer can design, manufacture and practice more effectively. Therefore, although the standard of the fibre itself is useful for users, construction engineers and others, it is expected to be used primarily by more fibre suppliers than those.

The status of the existing standards is as follows;

1) Test methods for composites exist, i.e. ISO 19044, ISO 21022 and ISO 21914. However, they are not for polymer fibre itself. Breaking force and elongation at break for the fibre itself are specified in ISO 2062, but other material properties of fibres, such as initial modulus of elasticity and thermal properties, are not specified.

2) Existing standards for fibres are intended for clothing textiles, ropes or strips. Test methods and unit system are different from those in the civil engineering field. The traditional unit system for textile is the Tex system, in which sectional size of fibre is expressed by weight per length. The unit system is different from that used in the civil engineering field. It would be very convenient to express them in SI units such as Newtons – millimetres.

The purpose of each testing item is described below.

For a fibre design, the fibre shape and mechanical properties are important for selection. The fibre length is selected upon the matrix composition. For example, a 4 mm to 12 mm length fibre is suitable for a uniform matrix such as cement mortar, and 20 mm or longer is required for concrete that includes coarse aggregates. The fibre diameter is also important because it influences the fibre dispersion through the fibre aspect ratio (length/diameter). The tensile strength and initial modulus of elasticity are key parameters that influence the reinforcing performance of the fibre through the fibre-to-matrix bond. On the other hand, the bonding strength, friction and surface treatment of fibre, in spite of their importance, are not included in this document as they are strongly related to the matrix properties and are generally difficult to estimate. In addition, creep and fatigue properties are not included in this document either because the needs of these properties depend on the application situations of the FRCC.

In terms of fibre usage, the fibre reinforcement performance in the FRCC is related to the fibre volume fraction, which is calculated from the fibre weight according to the fibre density. In the use of moisturized fibre products for a uniform fibre dispersion, the existence of water can have a significant effect on the hydration of the cementitious matrix. Thus, the fibre moisture content needs to be accurately estimated.

For the operation stage of the FRCC, their thermal properties and durability against chemicals are of particular concern. For instance, in case of high strength cementitious composites, the polymer fibre can melt during a fire to introduce small cavity so as to release the high internal pressure and consequently reduce the risk of an explosive failure of the cement matrix. Therefore, for fire protection applications, a relatively low melting point of the fibre is considered as apriority. In addition, fibres for the FRCC need to have high durability against alkaline conditions. Thus, the melting point and alkaline durability are two important parameters of the fibres.

The fibre properties are defined in this document as the properties of the smallest fibre unit that disperses in the FRCC. In actual application, fibres can also exist in the form of bundle even within the FRCC.

Test methods for discrete polymer fibre for fibre-reinforced cementitious composites

1 Scope

This document specifies the test methods for discrete polymer fibre for fibre-reinforced cementitious composites (FRCC).

This document defines the test methods for discrete polymer fibre, such as diameter, length, tensile strength, initial modulus of elasticity, density, melting point, moisture content and alkaline durability as basic items. These are test methods intended for certification of a fibre and not for quality control or field acceptance.

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 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1183-2, *Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method*

ISO 1183-3, *Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method*

ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*

3 Terms and definitions

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

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

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

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

3.1
fibre-reinforced cementitious composite
FRCC
concrete or mortar containing short discrete fibres that are distributed in the matrix

3.2
standard atmosphere
condition of an atmosphere with temperature of (20 ± 2) °C and relative humidity of (65 ± 4) %

3.3
standard condition
condition in *standard atmosphere* (3.2) for a period of at least 24 h

3.4
tenacity
tensile strength of fibre in cN/dtex

3.5
filament yarn
long and continuous fibre which has not been cut for fibre diameter determination or cut to the product length

3.6
consecutive fibre
long and continuous fibre for fibre diameter determination which is longer than the *chopped fibre* (3.7) products

3.7
chopped fibre
short fibre which is cut to the product length

3.8
individual fibre
smallest unit of fibre structure in fibre-reinforced cementitious composite

Note 1 to entry: "tex" expresses the unit that shows the sectional size of the individual fibre in weight (grams) per 1 000 m of fibre.

4 Symbols and abbreviated terms

c	cN/dtex	tenacity	7.4
d	mm	nominal value of individual fibre diameter	5.1
d_f	mm	measured value of individual fibre diameter	5.1, 5.2, 7.4, 7.5
E_{ini}	N/mm^2	initial modulus of elasticity	7.5
$\overline{f_{f0}}$	N/mm^2	average value of tensile strength of non-exposure specimen	11.7
$\overline{f_{f1}}$	N/mm^2	average value of tensile strength of alkaline exposed specimen	11.7
f_f	N/mm^2	tensile strength	7.4
l	m	length of specimen	5.1
L_f	mm	measured value of fibre length	5.2
L_F	mm	winding frame length in Figure 2	11.2
l_g	mm	grip length in tensile test	11.2
l_{test}	mm	gauge length in tensile test	7.5, 11.2
m	g	mass of specimen	5.1, 5.2, 10.1
m'	g	sample mass in standard condition	10.1
M_w	%	moisture content	10.1
n_f		number of fibres in yarn	5.1, 7.4, 7.5
P_A	N	load at point A	7.5
P_{max}	N	maximum tensile load	7.4
R_{et}	%	tensile strength retention	11.7
T_m	°C	melting point	9.1
δ_{ini}	mm	elongation at initial slope in tensile load – elongation curve	7.5
ρ	g/cm^3	density	5.1, 5.2, 7.4

5 Test methods for determining fibre diameter

5.1 Consecutive fibre method

a) Cut out filament yarns to make specimen referring to Table 2. Specimens are prepared by cutting out from each package without first layer. Take five specimens per package, each with a length according to Table 2 with an accuracy of 0,1 %. Use an appropriate metering device under a tension of 0,05 cN/dtex, with a tolerance of ±10 %. For fibres with diameters less than 0,5 mm, use a wrap reel. For diameters 0,5 mm and above, use a scale.

Table 2 — Specimen length

Nominal diameter, *d* mm	**Length, *l*** m
$d < 0{,}08$	250
$0{,}08 \leq d < 0{,}30$	100
$0{,}3 \leq d < 0{,}5$	10
$0{,}5 \leq d$	5

b) Condition the specimens before testing in the standard condition.

c) Measure the mass of the conditioned specimens, m, with an accuracy of 0,1 % in a standard atmosphere.

d) Calculate the fibre diameter with Formula (1):

$$d_f = 2\sqrt{\frac{m}{\pi \times l \times \rho \times n_f}} \qquad (1)$$

Formula (1) is derived from Formula (2), which determines m:

$$m = \pi\left(\frac{d_f}{2}\right)^2 \times 1\,000 \times l \times \frac{\rho}{1\,000} \times n_f \qquad (2)$$

Calculate the average value of the five or more results. The results shall be rounded off with a precision of three significant digits. If required, calculate the standard deviation and a 95 % confidence interval.

5.2 Chopped fibre method

a) Pick up 200 chopped fibre specimens for five or more times from the same package.

b) Condition the specimens before testing in the standard condition.

c) Measure the mass of the conditioned specimens, m, with an accuracy of 0,1 % in the standard atmosphere.

d) Calculate the fibre diameter with Formula (3):

$$d_f = 2\sqrt{\frac{5 \times m}{\pi \times L_f \times \rho}} \qquad (3)$$

Formula (3) is derived from Formula (4), which determines m:

$$m = \pi\left(\frac{d_f}{2}\right)^2 \times L_f \times \frac{\rho}{1\,000} \times 200 \qquad (4)$$

Calculate the average value of the five or more results. The results shall be rounded off with a precision of three significant digits. If required, calculate the standard deviation and a 95 % confidence interval.

NOTE The vibroscope method in ISO 1973 can be used under the agreement of the parties concerned.

5.3 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, and supplier);

b) conditioning status;

c) number of specimens (consecutive fibre method) or number of specimens set (chopped fibre method);

d) length of specimen (consecutive fibre method) or measured value of fibre length (chopped fibre method);

e) mass of specimen;

f) density used in calculation;

g) measured value of fibre diameter;

h) any deviations from the procedure;

i) any unusual features observed;

j) the date of the test;

k) a reference to this document (i.e. ISO 23523:2021).

6 Test method for determining fibre length

6.1 Procedure

a) Pick up 200 or more chopped fibre specimens from the same package.

b) Condition the specimens before testing in the standard condition.

c) Measure the length of the specimen by a scale in millimetres.

d) Calculate the average value of the length, L_f. The results shall be rounded off with a precision of 1 mm;

6.2 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, supplier);

b) conditioning status;

c) number of specimens;

d) measured value of fibre length;

e) any deviations from the procedure;

f) any unusual features observed;

g) the date of the test;

h) a reference to this document (i.e. ISO 23523:2021).

7 Test method for determining tensile strength and initial modulus of elasticity

7.1 Testing machine

Testing machine that can provide the extension to an individual fibre specimen at a constant rate shall be used. The testing machine is for consecutive fibre before cutting. It shall be capable of being set at test lengths of 200 mm to 500 mm using clamps to grip the specimen.

The clamps for gripping the specimens shall prevent slippage and fracture of specimen at the jaws. Flat-faced unlined jaws shall be the normal type but, if these cannot prevent slippage, then other types of clamps may be used, such as lined jaws, bollard clamps or other types of snubbing devices.

The pinching type clamps are generally used in a tensile test and can be used for a relatively short specimen. But if the pressure or frictional force for pinching the specimen is increased, the specimen is easily damaged at the gripping portion. Therefore, it is preferable to use lined jaws such as thin rubber sheet, man-made leather or other deformable and gripping thin materials.

The bollard-type clamps are also used in a tensile test of fibre. These are a type in which a specimen is wound to be fixed, and then it requires a long specimen.

As the type of clamp can influence the reading of the elongation, all interested parties shall use the same type.

The testing machine shall be equipped with an automatic load/elongation recording devices of sufficiently fast response, or with a system directly recording the breaking load and elongation at break.

The testing machine shall be capable of setting a pretension either by means of a set of pre-tensioning weights or by using the load-measuring device.

7.2 Sampling of specimens

In principle, specimens shall be a consecutive individual fibre. Specimens are prepared by cutting out from each package without first layer in consideration of the test length and grip length. The number of specimens shall be no fewer than 10.

If fibres of sufficient length cannot be obtained, the test length shall be at least 20 mm. In this case, the number of specimens shall be no fewer than 30.

In the case of bundled fibre products decomposed in FRCC, specimens with at least 8 turn bundles per 10 cm shall be used.

7.3 Procedure

a) Condition the specimens before testing in a standard condition.

b) Elongation rate shall be set in the range from 50 % of test length to 100 % of a test length per minute. Lower rate can be applied if agreed by all interested parties according to elongation capacity of fibre or facileness of initial modulus of elasticity calculation.

c) Set the specimen on the clamp. Move the crosshead until the specimen is taut, then start testing. Record tensile load and displacement during testing. Stop testing after the rupture of the specimen.

d) Omit the results obtained from the specimens as follows, and additional tests shall be carried out using the specimens from the same package:

— specimen slipped out from clamp;

— specimen ruptured at inside of clamp or at the edge of clamp.

7.4 Tensile strength

Calculate tensile strength with Formula (5):

$$f_f = \frac{P_{max}}{\pi \left(\frac{d_f}{2} \right)^2 \times n_f} \tag{5}$$

NOTE In the case of individual fibre, $n_f = 1$. In the case of multiple filament yarn, n_f is the number of fibres in the yarn.

According to the measuring equipment, tenacity may be automatically calculated. In this case, calculate the tensile strength with Formula (6):

$$f_f = 100 \rho c \tag{6}$$

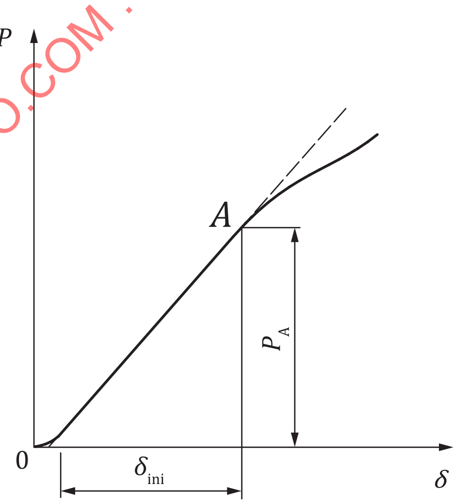
The results shall be rounded off with a precision of three significant digits. If required, calculate the standard deviation.

7.5 Initial modulus of elasticity

At the initial stage of load-elongation curve as shown in Figure 1, estimate the maximum tangent angle that is calculated by load at point A divided by elongation at initial slope. Calculate the initial modulus of elasticity with Formula (7):

$$E_{ini} = \frac{P_A}{\left(\frac{\delta_{ini}}{l_{test}} \right)} \times \frac{1}{\pi \left(\frac{d_f}{2} \right)^2 \times n_f} \tag{7}$$

NOTE In the case of individual fibre, $n_f = 1$. In the case of multiple filament yarn, n_f is the number of fibres in the yarn.



Key

P load, N

δ testing machine displacement, mm

Figure 1 — Tensile load – elongation curve at initial stage

The results shall be rounded off to the nearest hundred. If required, calculate the standard deviation.

7.6 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, supplier);

b) conditioning status;

c) test length;

d) number of specimens;

e) type of testing machine and load range;

f) tensile speed;

g) maximum tensile load;

h) tensile strength;

i) initial modulus of elasticity;

j) any deviations from the procedure;

k) any unusual features observed;

l) the date of the test;

m) a reference to this document (i.e. ISO 23523:2021).

8 Test method for determining fibre density

8.1 Procedure

Fibre density shall be determined in accordance with ISO 1183-1, ISO 1183-2 or ISO 1183-3.

Express the fibre density in grams per cubic centimetres with a precision of 0,01 g/cm^3.

8.2 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, supplier);

b) conditioning status of specimens;

c) a reference to this document (i.e. ISO 23523:2021);

d) test method;

e) number of tests and specimens;

f) density;

g) any deviations from the procedure;

h) any unusual features observed;

i) the date of the test.

9 Test method for determining thermal properties of fibre

9.1 Melting point

The melting point, T_m, shall be determined in accordance with ISO 11357-3.

Calculate the average value of the two or more results. Express the melting point in degree Celsius with a precision of 0,1 °C.

NOTE The glass transition temperature, T_g, is one of the thermal properties of polymer fibres. Hard and glassy-brittle phase transforms reversibly into a viscous or rubbery amorphous phase as the temperature changes. This transition temperature is said to be the glass transition temperature, T_g. T_g can be determined in accordance with ISO 11357-2. Therefore, phase transition is often unclear in polymer fibres because of low amorphous region content. If test results are considered to be unclear by using fibre specimen, raw material resin can be used for the specimen.

9.2 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, and supplier);

b) conditioning status of specimens;

c) number of specimens;

d) a reference to this document (i.e. ISO 23523:2021);

e) melting point;

f) any deviations from the procedure;

g) any unusual features observed;

h) the date of the test.

10 Test method for determining moisture content of fibre

10.1 Procedure

a) Pick up 5 g of samples from the same package.

b) Measure the mass of the sample, m.

c) Condition the sample in a standard atmosphere until the mass stops changing.

d) Measure the mass of the conditioned sample, m'. This is the sample mass at standard condition.

e) Calculate the moisture content with Formula (8):

$$M_w = \frac{m - m'}{m'} \times 100 \quad (8)$$

NOTE Values of m and m' are given with 3 significant digits.

Calculate the average value of the two or more results. Express the moisture content in percentages with a precision of 0,1 %.

10.2 Test report

The test report shall include the following items:

a) fibre profile (type of fibre, nominal value of fibre diameter, nominal value of fibre length, and supplier);

b) conditioning status of samples prior to preparation under standard conditions;

c) number of tests;

d) moisture content;

e) any deviations from the procedure;

f) any unusual features observed;

g) the date of the test;

h) a reference to this document (i.e. ISO 23523:2021).

11 Test method for determining alkaline durability

11.1 General

The alkaline durability is expressed by tensile strength retention in percentages after exposure in alkaline condition.

11.2 Apparatus and reagents

11.2.1 Reel, used for preparing test specimens.

11.2.2 Winding frame (Figure 2) **and receptacle,** used for immersing the specimens in aqueous alkaline solution.

The winding frame shall be made by alkaline resistant and high strength metal, such as SUS304, SUS316 or others. Frame length shall be larger than test length plus double of grip length. Receptacles shall be made by alkaline resistant materials, such as HDPE, SUS304 or others.

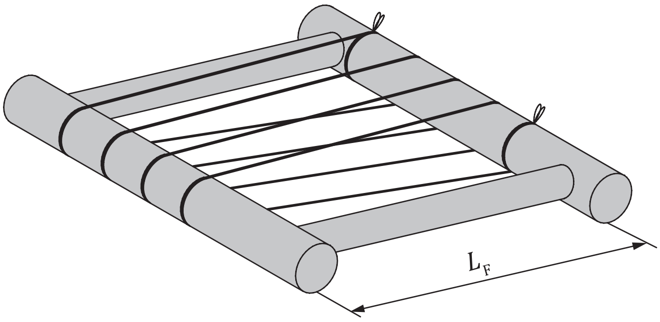
11.2.3 Distilled water, or water purified in same fashion as distilled water.

11.2.4 Potassium hydroxide.

11.2.5 Calcium hydroxide.

11.2.6 Sodium hydroxide.

11.2.7 pH sensor, used for measuring pH of aqueous alkaline solution.



Key

L_F $l_{test}+ l_g \times 2$ or more length is preferable

Figure 2 — Example of winding frame

11.3 Sampling and conditioning of test specimens

a) Choose samples from the same package and keep them away from heat, ultra violet, stress or other environmental influences to avoid changes in quality. Sample length shall be enough for tensile test (see 7.2).

b) Prepare samples for alkaline condition exposure and untreated. Samples shall be fixed to keep fibre not to move. Fixed on winding frame is preferable.

c) In the case of thin fibres, for example less than 0,08 mm, they are used in the test in a manufactured state, that is, in the state of uncut fibre bundles.

11.4 Preparation of alkaline solution

Alkaline solution shall be prepared by mixing the following aqueous solutions.

a) 14 g/l of aqueous potassium hydroxide, prepared in ambient temperature;

b) 2 g/l of aqueous calcium hydroxide, prepared in ambient temperature;

c) 10 g/l of aqueous sodium hydroxide, prepared in ambient temperature;

11.5 Exposure of alkaline condition

a) Sample aqueous alkaline solution before soaking and allow to cool to ambient temperature. Measure pH of the alkaline solution sample and check pH shall be 13,0 ± 0,5.

b) Soak samples in the alkaline solution of 60 °C while fixed on the winding frame. Samples shall be set and held 30 mm above the bottom of the receptacle and 30 mm below the liquid surface. The distance between the samples shall be 30 mm or more (Figure 3). Keep alkaline solution at 60 °C ± 2 °C. Care must be taken to ensure that the water in the solution does not evaporate and that carbon dioxide in the air is not absorbed during immersion.

c) Soaking time shall be 28 days.

d) Pick up samples at 28 days of soaking and wash by tap water. Washed samples shall be dried for tensile test.

e) Sample aqueous alkaline solution after soaking and allow to cool to ambient temperature. Measure pH of the alkaline solution sample.