BS EN 16806-1:2016

BSI Standards Publication

Textiles and textile products — Textiles containing phase change materials (PCM)

Part 1: Determination of the heat storage and release capacity

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Textiles and textile products - Textiles containing phase change materials (PCM) - Part 1: Determination of the heat storage and release capacity

Textiles et produits textiles - Textiles contenant des matériaux à changement de phase (PCM) - Partie 1: Détermination de la capacité de stockage et de dégagement de chaleur

Textilien und textile Erzeugnisse - Phasenwechselmaterialien enthaltende Textilien (PCM) - Teil 1: Bestimmung der Wärmespeicherungs- und Wärmefreisetzungskapazität

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European foreword

This document (EN 16806-1:2016) has been prepared by Technical Committee CEN/TC 248 "Textiles and textile products", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

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EN 16806, Textiles and textile products — Textiles containing phase change materials (PCM), consists of the following parts:

— *Textiles and textile products — Textiles containing phase change materials (PCM) — Part 1: Determination of the heat storage and release capacity*

— *Textiles and textile products — Textiles containing phase change materials (PCM) — Part 2: Determination of the heat transfer using a dynamic method*

— *Textiles and textile products — Textiles containing phase change materials (PCM) — Part 3: Determination of the heat transfer between the user and the product*.

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1 Scope

This part of EN 16806 specifies a test method for the determination of the heat storage and heat release capacity and the phase change temperatures of textile fibres, yarns and fabrics (woven and knitted fabrics, nonwovens) containing phase change materials (PCM). The test method can also be applied for pure or micro-encapsulated PCM.

This part of EN 16806 does not apply to the determination of the heat transfer properties of textile fabrics (woven and knitted fabrics, nonwovens) containing phase change materials, for which part 2 of EN 16806 applies.

This part of EN 16806 does not apply to determining the heat transfer between the user and the product for textile products, e.g. garments, mattresses, etc. made with PCM containing materials, for which part 3 of EN 16806 applies.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12127, *Textiles - Fabrics - Determination of mass per unit area using small samples*

EN ISO 139, *Textiles - Standard atmospheres for conditioning and testing (ISO 139)*

EN ISO 11357-1, *Plastics - Differential scanning calorimetry (DSC) - Part 1: General principles (ISO 11357-1)*

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

3 Terms and definitions

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

3.1
phase change material
PCM
a substance which is capable of storing and releasing thermal energy in the form of latent heat (enthalpy), over a specific temperature range during which the material changes phase (from solid to liquid or from liquid to solid), thus buffering external temperature changes

Note 1 to entry: These temperature ranges are usually close to skin temperature.

3.2
pure PCM
PCM in its free form

3.3
micro-encapsulated PCM
PCM contained in a spherically shaped particle with a closed shell and having a diameter in the range of 1 µm to 1 mm

3.4
textile containing PCM
textile containing the phase change material inside the fibres or yarns; or having the PCM applied to the fibre, yarn or textile surface

3.5
phase change temperature
peak temperature at which the PCM changes either from the solid to the liquid (melting temperature) or from the liquid to the solid state (crystallisation temperature)

Note 1 to entry: Ideally, the two phase change temperatures should be identical, but in reality a difference will be observed as often material mixtures are used.

4 Principle of test

This test determines the enthalpy of fusion and of crystallization, as well as the phase change temperatures of fibres, yarns and fabrics containing PCM.

A differential scanning calorimetry (DSC) method is used. The difference between the rate of heat flow into a specimen and that into a reference crucible is measured as a function of temperature and/or time while the specimen and the reference are subjected to the same temperature control programme.

5 Test equipment, reagents and materials

The DSC test equipment described in EN ISO 11357-1 shall be used.

Open aluminium crucibles shall be used to ensure an isobaric measurement. It is recommended to put a lid on the crucible but without sealing it hermetically.

The test equipment shall be calibrated periodically in accordance with the instructions and recommendations of the equipment manufacturer, i.e. with respect to the use and shelf life of calibration standards. The calibration procedure of EN ISO 11357-1 shall be followed. Calibration materials should be chosen with a transition temperature as close as possible to phase change temperature of the PCM. The heating rate shall be 5 °C/min.

NOTE A suitable calibration material is phenyl salicylate (CAS 118–55–8), with a melting temperature of 41,79 °C and an enthalpy of fusion of (89,4 ± 0,4) J/g. Other reference materials can be recommended by the suppliers.

6 Test specimens

6.1 Sample conditioning

Samples shall be conditioned in the standard atmosphere according to EN ISO 139 (20 °C ± 2 °C and 65 % RH ± 4 % RH) for at least 24 h before determining the mass per unit area.

This conditioning only applies to textile materials containing PCM and the dried micro-encapsulated PCM. It does not apply to the pure PCM.

6.2 Sampling and specimen preparation

6.2.1 General

The specimen shall be representative of the sample being examined and shall be prepared and handled with care. Particular care shall be taken to avoid any contamination of the specimen. If the specimen crucible is closed with a lid, this shall not cause any deformation of the bottom of the crucible. Good

thermal contact between the specimen and crucible and between the crucible and holder shall be ensured.

The mass of the DSC test specimens shall be between 10 and 40 mg, with 10 mg being recommended. It may be necessary for low weight fabrics to put several layers in the same crucible to get the required mass. The tolerance on the weight should be as described in EN ISO 11357-1. A lid shall be pressed on the crucible to ensure a good contact between the textile specimen and the bottom of the crucible. For very thin fabrics or for bulky, fluffy materials it may be necessary to stack, fold or compress the textile material to obtain a good thermal contact with the bottom of the crucible. Care shall be taken to allow pressure release during the measurement (isobaric measurement condition).

A new crucible and lid shall be used for each specimen.

NOTE Each DSC instrument has its own shape of crucibles and its own press to put the lid on the crucible. Crucibles are not interchangeable.

6.2.2 Pure PCM

Three specimens shall be prepared.

Pre-weigh the empty crucible and its lid.

Pre-heat the PCM sample to 60 °C so that it is in the liquid state. Collect one representative specimen of pure PCM through a micro-pipette with a volume range between 10 to 20 µL. Carefully deposit the PCM into the crucible so that the bottom of the crucible is completely covered, without overfilling. Carefully place the lid when sealing the crucible in order to avoid spilling of the PCM.

Leave the crucible at room temperature in a desiccator for 20 min to solidify the PCM.

Weigh the filled crucible and determine the weight of the PCM.

6.2.3 Micro-encapsulated PCM

The micro-encapsulated PCM product shall be dried at 60 °C for at least 24 h to remove all water. The micro-encapsulated PCM product shall then be conditioned as described in 6.1.

The sample size should be sufficient to prepare five DSC specimens. Three of them shall be tested; if the coefficient of variation is too high (see 7.2) the other two shall also be tested.

Prepare five DSC specimens in accordance with 6.2.1.

6.2.4 Fibres and yarns

Five representative test specimens shall be taken from the fibre or yarn sample and prepared in accordance with 6.2.1. Three of them shall be tested; if the coefficient of variation is too high (see clause 7.2) the other two specimen shall also be tested.

6.2.5 Fabrics

Test specimens shall be taken from a fabric sample of at least 50 cm length over the entire width of the fabric.

From this fabric sample, five specimens shall be taken to determine the mass per unit area of the fabric in accordance with EN 12127.

Representative test specimens for the DSC analysis shall be taken from three of the five specimens used for the determination of mass per unit area. One DSC specimen shall be taken from each of these three mass specimens. The two remaining mass specimens shall be kept for additional tests in case of excessive scattering of test results or if test results are discarded.

Care should be taken to ensure representativeness of the specimens. In the case of multi-layered (e.g. coated, laminated or stacked) substrates, specimen shall be taken in a manner that includes all layers of

the sample. In the case of heterogeneous samples, specimens with different composition shall be taken. As many different zones as possible shall be sampled taking into account the relative surface area of the fabric.

Deviations from the above sampling procedures may be agreed on by the involved parties. This deviating sampling procedure then shall be clearly described in the report.

7 DSC test procedure

7.1 General

Test specimens and reference specimens are subjected to a temperature control programme, which contains a heating-cooling-heating thermal cycle at a rate of 5 °C/min under a controlled inert atmosphere.

The cycling shall be performed between a low temperature, T_{low}, of −20 °C and a high temperature, T_{high}, of 60 °C. The base line (as defined in 7.2) before and after the phase change transition peak shall be stable over a range of 20 °C. If this condition is not met, the interval between T_{low} and T_{high} shall be extended.

7.2 Test sequence

The test procedures as defined in EN ISO 11357-1 and EN ISO 11357-3 shall be applied unless otherwise specified hereafter.

A heating and cooling rate of 5 °C/min shall be used.

The test shall be performed on three specimens.

The test procedure shall be performed as follows:

— Choose the testing range as defined in 7.1.

— Perform a first heating step from T_{low} to T_{high}, followed by an isotherm at T_{high} for 2 min.

— Then perform a first cooling step from T_{high} to T_{low}, followed by an isotherm at T_{low} for 2 min.

— Then perform a second heating step from T_{low} to T_{high}.

If the coefficient of variation on these three test results exceeds 20 % (see Clause 8), two additional measurements shall be performed.

NOTE Inhomogeneous samples as well as samples containing significant amounts of water or that show artefacts in the temperature range of the measurement cycle (e.g. glass transitions or phase transitions in the substrate material) can result in a high coefficient of variation.

8 Calculation of test results

8.1 General

For each DSC specimen:

— based on the recorded second heating cycle, determine the enthalpy of fusion in J/g (as defined in EN ISO 11357-3);

— based on the recorded cooling cycle, determine the enthalpy of crystallization in J/g (as defined in EN ISO 11357-3);

— based on the recorded second heating cycle, determine the extrapolated onset melting temperature, the peak melting temperature (phase change temperature on heating) and the extrapolated end melting temperature (as defined in EN ISO 11357-3);

— based on the recorded cooling cycle, determine the extrapolated onset crystallization temperature, the peak crystallization temperature (phase change temperature on cooling) and the extrapolated end crystallization temperature (as defined in EN ISO 11357-3).

The heat of fusion and heat of crystallization should be similar (within 10 %). If this is not the case, the area of integration shall be checked and adjusted.

Calculation of the average enthalpy of fusion and crystallization:

For each of the three specimens, calculate the average enthalpy per unit mass for crystallization and of fusion.

8.2 Calculation of the average enthalpy of fusion and crystallization per unit area (Fabrics)

Record the mass per unit area (g/m^2) to three significant digits for each individual DSC specimen, as determined in 6.2.5, as well as the mean value and the coefficient of variation, if required. Calculate the average mass per unit area for the sample from these individual values.

Calculate the average enthalpy of fusion/ crystallization per unit area as the ratio of the average enthalpy of fusion/ crystallization per unit mass and the average mass per unit area for the sample.

9 Test report

The test report shall contain the following elements:

a) reference to this part of the standard;

b) identification of the test lab;

c) identification of the sample and the material;

d) test conditions, including;

   1) date;

   2) particulars of specimen preparation;

   3) reference of calibration standard and frequency of calibration.

e) type of DSC instrument used (heat flux or power compensated), as well as the instrument model and manufacturer;

f) number of specimens;

g) test results, in the correct number of significant digits, as determined by the standard deviation;

   1) the enthalpy of fusion, the enthalpy of crystallization, the average results and standard deviations for this;

   2) the mass of the fabric in g/m^2 and the calculated average result for the heat storage and release capacity in J/m^2;

For high standard deviations it is useful to report 3 significant digits, even though it should be less according to the standard deviation.

3) the measurement curve with interpretation including:

   i) the extrapolated onset melting temperature (in °C);

   ii) the peak melting temperature (phase change temperature) (in °C);

   iii) the extrapolated onset crystallization temperature (in °C);

   iv) the peak crystallization temperature (in °C).

h) other remarks and observations;

i) deviations from this standard;

The test report may additionally include the following results:

j) individual results for the enthalpy of fusion;

k) individual results enthalpy of crystallization;

l) individual results for the fabric weight;

m) for extended peaks: to report the enthalpy for temperature intervals within the complete peak, as agreed with the customer.

Bibliography

[1] "Standards, Calibration, and Guidelines in Microcalorimetry – Part 2: Calibration Standards for Differential Scanning Calorimetry (IUPAC Technical Report – Della Gatta et Al) © 2006 IUPAC, Pure and Applied Chemistry 78, 1455–1476, Table 3

[2] ISO/TR 11827, *Textiles — Composition testing — Identification of fibres*

[3] CEN/TR 16298, *Textiles and textile products - Smart textiles - Definitions, categorisation, applications and standardization needs*

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