SLOVENSKI STANDARD
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Trdna biogoriva - Metoda za doloį anje taljenja pepela - 1. del: Karakteristiį na temperaturna metoda

Solid biofuels - Method for the determination of ash melting behaviour - Part 1: Characteristic temperatures method

Feste Biobrennstoffe - Verfahren zur Bestimmung des Schmelzverhaltens der Asche - Teil 1: Verfahren zur Bestimmung charakteristischer Temperaturen

Biocombustibles solides - Méthode de détermination de la fusibilité des cendres - Partie 1: Méthode des températures caractéristiques

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English Version

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Biocombustibles solides - Méthode de détermination de la fusibilité des cendres - Partie 1: Méthode des températures caractéristiques

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Foreword

This document (CEN/TS 15370-1:2006) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

Ash melting is a complex process where also sintering, shrinkage and swelling can occur.

The test method described in this Technical Specification provides information about fusion and melting behaviour of the composite inorganic constituents of the fuel ash at high temperatures.

The test method is empirical. The ash used for the test is a homogeneous material, prepared from the fuel, and the determination is performed at a controlled rate of heating in a controlled atmosphere. In contrast, under full-scale conditions, the complex processes of combustion and fusion involve heterogeneous mixtures of particles, variable heating rates and gas compositions.

The determined characteristic temperatures in the test can be used for comparison of the tendency of the ashes from different types and qualities of solid biofuels to form fused deposits or to cause bed agglomeration on heating.

The method is based on the methods described in ISO 540:1995 and DIN 51730:1998.

The terms ash fusibility and ash softening are synonyms to ash melting.

1 Scope

This document specifies a method for the determination of the characteristic temperatures for the ash melting behaviour of solid biofuels.

2 Normative references

The following referenced documents are indispensable for the application 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.

CEN/TS 14588:2003*, Solid Biofuels – Terminology, definitions and descriptions*

CEN/TS 14775:2004, *Solid Biofuels –Method for the determination of ash content*

3 Terms and Definitions

For the purposes of this Technical Specification, the terms and definitions given in CEN/TS 14588:2003 shall apply together with the following.

3.1
Shrinkage starting temperature
(abbreviation: SST): The temperature at which shrinking of the test piece occurs. This temperature is defined as when the area of the test piece falls below 95 % of the original test piece area at 550 °C.

NOTE Shrinking may be due to liberation of carbon dioxide, volatile alkali compounds, and/or sintering.

3.2
Deformation temperature
(abbreviation: DT): The temperature at which the first signs of rounding of the edges of the test piece occurs due to melting.

NOTE For computerised evaluation a shape factor change of 15 % marks the deformation temperature. For definition of shape factor see normative Annex A.

3.3
Hemisphere temperature
(abbreviation: HT): The temperature at which the test piece forms approximately a hemisphere i.e. when the height becomes equal to half the base diameter.

3.4
Flow temperature
(abbreviation: FT): The temperature at which the ash is spread out over the supporting tile in a layer, the height of which is half of the height of the test piece at the hemisphere temperature.

NOTE Half of the height of the test piece has been defined due to frequently occurring bubbling effects. This is especially important for automatic image evaluation. Be aware that this definition is different to other standards.

4 Principle

A test piece made from a prepared ash is heated up with constant rate and continuously observed. The temperatures at which characteristic changes of the shape occur are recorded. The characteristic temperatures are defined in Clause 3 (see also Figure 1).

5 Reagents

5.1 Demineralised water.

5.2 Dextrin, 100 g/l solution.

Dissolve 10 g of dextrin in 100 ml of water.

5.3 Ethanol, with a purity ≥ 95 %.

5.4 Gold wire, of diameter 0,5 mm or larger, or

gold plate, of thickness 0,5 mm to 1,0 mm with a purity of 99,99 % or a certified melting point (e.g. 1064 °C).

5.5 Nickel wire, of diameter 0,5 mm or larger, or

nickel plate, of thickness 0,5 mm to 1,0 mm, with a purity of 99,9 % or a certified melting point (e.g. 1455 °C).

Nickel is used for reducing atmosphere.

5.6 Palladium wire, of diameter 0,5 mm or larger, or

palladium plate, of thickness 0,5 mm to 1,0 mm with a purity of 99,9 % or a certified melting point (e.g.1554 °C).

5.7 Carbon dioxide, **carbon monoxide, hydrogen** or

ready mixture of **carbon dioxide** and **carbon monoxide** with 55 % (V/V) to 65 % (V/V) carbon monoxide and 35 % (V/V) to 45 % (V/V) carbon dioxide or

ready mixture of **hydrogen** and **carbon dioxide** with 45 % (V/V) to 55 % (V/V) hydrogen and 45 % (V/V) to 55 % (V/V) carbon dioxide.

6 Apparatus

6.1 Furnace, electrically heated, which satisfies the following conditions:

a) it shall be capable of reaching the maximum temperature at which the properties of the ash are to be determined (a temperature of 1500 °C or more may be required);

b) it shall provide an adequate zone of uniform temperature in which to heat the test piece(s);

c) it shall provide means of heating the test piece(s) at a uniform rate from 550 °C upwards;

d) it shall be capable of maintaining the required test atmosphere (see 7.1) around the test piece(s);

e) it shall provide means of observing the change of shape of the test piece(s) during heating.

6.2 Pyrometer, comprising a platinum/platinum-rhodium thermocouple.

6.3 Mould, of brass, stainless steel or other suitable material for preparing the test piece.

6.4 Hand press with spring pressure compression, to produce the test piece. The required spring pressure is about 1,5 N/mm^2.

6.5 **Support for the test piece,** of such a material that it becomes neither distorted, not reacts with nor absorbs the ash during the determination. Support of sintered alumina or fine textured mullite are generally satisfactory, but difficulties may arise with individual ashes, in which case zirconium supports or a non-absorbent interface such as platinum foil may be used between the original support and the test piece.

6.6 **Flowmeters**, for measuring the components of the reducing gases (See 7.1) and/or for measuring the flow rate of the oxidizing gas.

6.7 **Agate mortar and pestle** or other adequate grinding instruments.

6.8 **Test sieve**, of aperture 0,075 mm (e.g. diameter of 100 mm or smaller, preferable completed with lid and receiver).

6.9 **Optical instrument**, which enables the profile of the test piece to be observed throughout the determination, by using a camera or video equipment.

7 Test conditions

7.1 Test atmosphere

An oxidizing or reducing atmosphere depending on the application can be used.

An oxidizing atmosphere is obtained with air or carbon dioxide.

The reducing atmosphere is obtained by introducing a mixture of

a) 55 % (V/V) to 65 % (V/V) carbon monoxide with 35 % (V/V) to 45 % (V/V) carbon dioxide or

b) 45 % (V/V) to 55 % (V/V) hydrogen with 45 % (V/V) to 55 % (V/V) carbon dioxide into the furnace at a minimum linear rate of flow past the test piece between 100 – 250 mm/min, calculated at ambient temperature.

NOTE The flow rate is not very critical, provided that in the case of reducing atmosphere it is sufficient to prevent any leakage of air into the furnace. However, the same flow rate level is recommended also for oxidizing atmosphere. For furnaces with larger diameter a flow around 400 mm/min for reducing atmosphere may be needed. In all cases refer also to manufacturer instructions. The flow rate for rotameter adjustment can be calculated by multiplying the flow rate in mm/min with the inside cross-section area of the furnace tube and converting to units litres/min.

WARNING: When using the reducing atmosphere given above, the gases emerging from the furnace will contain a proportion of carbon monoxide; it is essential, therefore, to ensure that these gases are vented to the outside atmosphere, preferably by means of a hood or an efficient fan system. If hydrogen is used in the reducing atmosphere, great care shall be taken to prevent an explosion occurring, by purging with carbon dioxide both prior to the introduction of the hydrogen and after the hydrogen supply is shut off.

7.2 Shape of test piece

The test piece shall have sharp edges to facilitate observation.

The mass of the test piece shall be such as to ensure equalization of the temperature within the test body. Hence, dimensions that are too large shall be avoided.