SLOVENSKI STANDARD
SIST-TS CEN/TS 17510:2020

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Materiali, pridobljeni iz izrabljenih avtomobilskih gum - Določanje specifične površine prahu - Metoda, ki temelji na adsorpciji kriptona

Materials obtained from end-of-life tyres - Determination of the specific surface area of powders - Method based on krypton adsorption

Materialien aus Altreifen - Bestimmung der spezifischen Oberfläche von Granulaten und Mehlen - Verfahren basierend auf Kryptonadsorption

Matériaux produits à partir de pneus usagés non réutilisables - Détermination de la surface spécifique des poudrettes - Méthode fondée sur l'adsorption de krypton

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English Version

Materials obtained from end-of-life tyres - Determination of the specific surface area of powders - Method based on krypton adsorption

Matériaux produits à partir de pneus usagés non réutilisables - Détermination de la surface spécifique des poudrettes - Méthode fondée sur l'adsorption de krypton

Materialien aus Altreifen - Bestimmung der spezifischen Oberfläche von Mehlen - Verfahren basierend auf Kryptonadsorption

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Ref. No. CEN/TS 17510:2020 E

Contents

Page

European foreword

This document (CEN/TS 17510:2020) has been prepared by Technical Committee CEN/TC 366 “Materials obtained from End-of-Life Tyres (ELT)”, the secretariat of which is held by UNI.

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Introduction

Specific surface area (A_S) is a parameter of great importance when it comes to physical characterization of materials such as granulates and powders from rubber materials. Like other physical characteristics, specific surface area could influence the performance of materials in its different applications.

Depending on the type of sample to be characterized, several different methods can be used for the determination of the specific surface area, generally based on different physical principles. The most widespread and useful method used in materials characterization is gas adsorption, either through gravimetric or volumetric methods.

For very low surface area samples the traditional volumetric method of nitrogen adsorption at 77 K or Argon at 87 K shows some important limitations. Alternatively, for absolute areas as low as 0,05 m^2g^{-1} the suitable method for A_S determination is krypton adsorption at 77 K.

1 Scope

This document specifies a method for the determination of low specific surface area of powders ELTs rubber by measuring the amount of physically adsorbed krypton gas and applying the theoretical multipoint Brunauer, Emmett and Teller (BET) method.

This document defines a specific method for powders taking into account that, in order to obtain an accurate value of specific surface area, a representative sample of the material to be tested is taken according to the principle that every particle of the sample that represents the lot have an equal probability of being included in the sample.

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 14243-1:2019, *Materials obtained from end of life tyres — Part 1: General definitions related to the methods for determining their dimension(s) and impurities*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14243-1:2019 and the following apply.

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

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

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

3.1
adsorption
enrichment of the adsorptive gas at the external and accessible internal surfaces of a solid material

[SOURCE: ISO 15901-2:2006]

3.2
adsorbate
adsorbed gas

[SOURCE: 15901-2:2006]

3.3
adsorptive
gas or vapour to be adsorbed

[SOURCE: ISO 15901-2:2006]

3.4
adsorbent
solid material on which adsorption occurs

[SOURCE: ISO 15901-2:2006]

3.5
isotherm
relationship between the amount of gas adsorbed and the equilibrium pressure of the gas, at constant temperature

[SOURCE: ISO 15901-2:2006]

3.6
surface area
extent of available surface area as determined by a given method under stated conditions

[SOURCE: ISO 15901-1:2016]

3.7
specific surface area
absolute surface area of the sample divided by sample mass

3.8
relative pressure
ratio of the equilibrium adsorption pressure, *p*, to the saturation vapour pressure, *p*°, at analysis temperature

[SOURCE: ISO 15901-3:2007]

4 Principle

The determination of the specific surface area undergoes by the determination of the amount of adsorbate required to cover the external and accessible internal pore surface of the adsorbent with a complete monolayer of adsorbate. The monolayer capacity is then calculated by using the BET equation:

$$\frac{p / p^\circ}{n_a \left[1 - \left(p / p^\circ\right)\right]} = \frac{1}{n_m C} + \frac{C - 1}{n_m C} \times \left(p / p^\circ\right) \qquad (1)$$

where n_a is the amount adsorbed at the relative pressure p/p°, n_m is the monolayer capacity and C is a constant dependent on isotherm shape. By plotting $\frac{p / p^\circ}{n_a \left[1 - \left(p / p^\circ\right)\right]}$ against p / p° a linear relation is achieved and the n_m value determined from slope and y-intercept values. The range of linearity of BET is usually restricted between 0,05 and 0,35, depending on the materials this range can be wider or shorter.

For low specific surface areas, lower than 1 m^2g^{-1}, the recommended method is krypton adsorption at 77 K (liquid nitrogen temperature). Since at 77 K krypton is below its triple point temperature, there are some studies that suggest the adsorbate could be in a liquid-like state and therefore it is more adequate to use the value of p° for supercooled liquid when plotting the BET.

The test is performed by automated equipment following the principle that gas is admitted to the sample cell, which is at a constant temperature of 77 K, and the amount of adsorbed gas is recorded after equilibration is achieved and plotted as relative pressure, *p*/*p*°.

5 Apparatus

5.1 General

For the present test it is recommended to use an automated volumetric apparatus with the following features.

5.1.1 Vacuum system, with the capability of attaining pressures below 10-4 torr, preferable with a coupled system composed by a diaphragm and turbo molecular pump.

5.1.2 Constant pressure transducers, the automated apparatus should have three gages, one capable of measuring 0 torr to 1 000 torr to the nearest 1 torr, one capable of measuring 1 torr to 10 torr at nearest 0,001 torr and one of 1 torr or 0,1 torr at nearest 0,000 1.

5.1.3 Manifold, both main manifold and station manifold should have a fixed volume known to the nearest 0,001 cm^3.

5.1.4 Gas, krypton with purity greater than 99,99 %, helium with purity greater than 99,999 %.

5.1.5 Sample cell should be able to accommodate enough sample to achieve a total absolute area of at least 0,5 m^2 and reduce at the same time the length of the cold zone. Normally, a volume of 25 cm^3 is needed in order to fulfil the requirements of total absolute area.

5.1.6 Dewar flask, for immersion of sample cell and reference (p°) cell in liquid nitrogen, controlled by a RTD level indicator in order to ensure a constant height of liquid nitrogen in sample and p° cells.

5.1.7 Heating mantle or other type of outgassing device to degas samples before analysis.

5.1.8 Laboratory analytical balance, capable to read at the nearest 0,1 mg.

6 Procedure

6.1 Sample preparation

Sampling should always be carried out, when possible, according to CEN/TS 17188:2018 and EN 14243-2:2019, 5.2. The laboratory sample should then be split into smaller samples (test sample) of weight not higher than 10 g using a sample splitter or sample divider.

Prior to the analysis, the sample shall be prepared and outgassed (also called degassed) in order to remove all the physisorbed material, without causing any permanent modification in the sample to be analysed.

Weight the sample (in an analytical balance to the nearest 0,1 mg) into an adequate sample cell in order to achieve a total absolute area of at least 0,05 m^2. The sample should not be compressed to avoid problems in the diffusion of the supercooled krypton during the analysis.

The sample outgas should be performed at 60 °C during 8 h with a stepwise of 1 °C min^{-1} under ultra-high vacuum by using for this purpose a turbomolecular pump system or similar. The sample should be kept in vacuum or filled with nitrogen until the moment of analysis.