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Coating powders —

Part 15: Rheology

Poudres pour revêtement —

Partie 15: Rhéologie

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

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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.

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Introduction

The powder coating process is dependent on several material variables, whose presence within the powder mixture is critical for ensuring smooth operation. A precise understanding of the fluidization behaviour is important for optimal processing parameters in the fluidized bed, during pneumatic conveyance and the spraying process. In addition, understanding the long-term stability during storage or behavioural change during processing can also help to improve the reuse of powder coatings. Furthermore, melt rheology is introduced to understand the melt flow properties of a given powder coating system, to provide information after spraying, on the powder melt viscosity and gel point. The methods in this document can be used for quality-control and detailed studies.

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Coating powders —

Part 15: Rheology

1 Scope

This document specifies methods for the determination of the rheological behaviour of a coating powder both in particulate and molten form.

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 3219-1, *Rheology — Part 1: Vocabulary and symbols for rotational and oscillatory rheometry*

ISO 3219-2, *Rheology — Part 2: General principles of rotational and oscillatory rheometry*

ISO 8130-14, *Coating powders — Part 14: Vocabulary*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

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3 Terms and definitions

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For the purposes of this document, the terms and definitions given in ISO 3219-1, ISO 8130-14 and the following apply.

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

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

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

3.1
apparent viscosity
flow resistance of the fluidized powder bed

4 Principle

4.1 Powder

The different fluidization properties are determined in a rheometer. In addition to rotational speed and torque, the airflow rate through the powder bed can be precisely controlled and the pressure drop determined. This allows complete analysis of all fluidization parameters that influence the powder coating process.

4.2 Melt

In order to characterize the powder in its molten form, a rheometer in accordance with ISO 3219-2 shall be used to determine the melt and polymerization properties.

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5 Apparatus

Use ordinary laboratory apparatus, together with the following.

5.1 Rheometer, in accordance with ISO 3219-2, equipped with a cylindrical measurement cell with a minimum diameter of 50 mm and a minimum height of 100 mm, ideally transparent and coated with conductive material to prevent electrostatic charging to maintain visibility. As a measurement system, a rectangular stirrer of minimum of 10 mm × 30 mm measurement surface is recommended unless otherwise specified.

For particulate measurements, a mass flow controller capable of generating an airflow from 0,05 l/min to at least 5 l/min is connected to ensure a uniform fluidization. A pressure transducer is required if pressure drop measurements shall be gathered.

For molten measurements, a disposable plate-plate geometry in a rheometer according to ISO 3219-2 with a heated plate and hood is advised.

5.2 Measurement geometry

For fluidization, deaeration and apparent viscosity measurements, use a profiled cylinder. Alternatively, measure in an undisturbed bed, without any measurement system inserted.

For cohesion strength measurements, a rectangular stirrer with a minimum measurement surface of 10 mm × 30 mm is recommended.

Use of a disposable plate of at least 25 mm diameter is advised in molten measurements.

NOTE A plate of smaller diameter can give spurious results.

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6 Sampling

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Take a representative sample of the product under test as specified in ISO 15528.

A minimum sample volume for powder particulate rheology measurements of 80 ml is recommended.

For further measurements of the same powder, it is recommended to keep a consistent mass.

For melt rheology measurement, it is advised to fill the plate-plate gap with the excess particulate powder, e.g. × 15 the volume, and then compress it prior to melting.

7 Experimental

7.1 Powder measurements

7.1.1 Fluidization measurements

Carry out the fluidization measurement using the profiled cylinder and perform it in two modes, i.e. unstirred and stirred, e.g. rotational speed 8 min^{-1}. First, decrease the air flow from 5 l/min to 0 l/min continuously, within a minimum of 30 s and then increase again from 0 l/min to 5 l/min.

This measurement is carried out to find the volumetric flow rate i.e. the volume of gas per time necessary for subsequent testing, as well as the volumetric flow rates for incipient and full fluidization of the sample.

NOTE For explanations of transitions such as incipient and full fluidization, see A.1.

The measurements shall be performed first unstirred, then stirred.

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A pressure transducer is required for pressure drop measurements by taking a baseline measurement with the same parameters in the empty cell immediately prior to measurement giving absolute results.

7.1.2 Fluidized bed rheology measurements

Carry out fluidized bed rheology measurements with a profiled cylinder system. The rotational speed shall be increased from 0,1 min^{-1} to 1 200 min^{-1}, corresponding to an applied shear rate increase from 0,02 s^{-1} to 320 s^{-1}.

Calculate the powder's apparent viscosity in a manner analogous to that of a non-Newtonian fluid. To this end, the dimensions of the measuring chamber (e.g. 50 mm diameter) and the profiled cylinder (e.g. 17 mm diameter, no cone at the bottom) shall be measured in accordance with ISO 3219-2. Set the data gathered so that each data point is equivalent to at least one full revolution of the cylinder.

7.1.3 Deaeration measurements

Measure bed deaeration by first stirring and fluidizing the powders with an airflow of 5 l/min for 5 min at 8 min^{-1}, then stop the airflow and obtain the pressure data under the powder bed.

7.1.4 Cohesion strength measurements

Cohesion strength describes the internal resistance of the powder to flow, and thereby a measure of powder flowability. It is defined as a measure for the strength of the bonding forces between powder particles.

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Carry out cohesion strength measurements as a function of torque with a two-blade stirrer after a steady-state flow is achieved. Precondition the coting powder to remove residual tension by stirring at 8 min^{-1} with an airflow retrieved from full fluidization measurements of the powder.

The cohesion strength can be calibrated using a constant geometry factor, e.g. using a certified reference material (CRM) such as CRM-116 limestone powder at a 3 kPa preconditioning (see A.2 for further details).

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The calculation method relies on torque measurements made only when a steady-state flow is achieved.

NOTE The final 20 points of an analysis are usually averaged and used to compare the different samples. Steady-state flow is usually reached within the given time of 100 s.

7.2 Melt rheology measurements

Use a rheometer using a disposable plate-plate geometry with a heated hood and plate.

Set the gap to 0,5 mm, the heating rate to 3,5 K/min with a temperature range between 50 °C and 180 °C, constant oscillatory parameters to 1 Hz and minimum 0,032 5° in amplitude.

8 Expression of results

Apparent viscosity of the fluidized bed is measured over different shear rates according to the characteristics of the powder.

For information on the interpretation of test results, see Annex A.

9 Precision

For fluidized bed rheology measurement, a torque accuracy of 1 nNm and under is recommended.

For cohesion strength measurements in powder coatings, a torque accuracy of 1 µNm and under is recommended.

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The powder illustrated in Figure A.2 (fresh) has a repeatability and reproducibility of 1 %.

If the same powder is repeatedly measured and the cohesion strength decreases, this is an indication of segregation. For consistent results, use fresh powder for each measurement.

A reference material is not available for fluidized measurements since the only available certified reference material is too cohesive to be fluidized (see A.2 for details).

10 Test report

The report of results shall contain at least the following information:

a) all details necessary to identify the powder product tested;

b) a reference to this document, i.e. ISO 8130-15:2023;

c) the test method used;

d) the initial mass of the sample;

e) the result for the test sieve used, as indicated in Clause 8;

f) any deviation from the test method specified;

g) any unusual features (anomalies) observed during the test;

h) the date of the test.

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