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Method for determination of bulk density, true density, true porosity and open porosity of carbon materials

炭素材料体积密度、真密度、真气孔率、显气孔率的测定方法

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Method for determination of bulk density, true density, true porosity and open porosity of carbon materials

1 Scope

This document specifies the method for determination of bulk density, true density, true porosity and open porosity of carbon materials.

This document is applicable to the determination of bulk density, true density, true porosity and open porosity of carbon materials at room temperature.

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.

GB/T 1427, Sample method of carbon material

GB/T 1997, Coke-sampling and preparation of samples

GB/T 8170, Rules of rounding off for numerical values & expression and judgment of limiting values

GB/T 8718, The terms of carbon materials

3 Terms and definitions

For the purpose of this document, terms and definitions given in GB/T 8718 apply.

4 Determination of bulk density

4.1 Instruments and apparatuses

4.1.1 Analytical balance: scale graduation 0.01 g.

4.1.2 Blast drying oven: equipped with an automatic temperature control device, capable of maintaining the temperature at (110±5) °C.

4.1.3 Vernier caliper: measuring range (0~200) mm, graduation value 0.02 mm.

$$V_b = abc \qquad \cdots\cdots(2)$$

Where:

a – length of the rectangular test sample, in millimeters (mm);

b – width of the rectangular test sample, in millimeters (mm);

c – height of the rectangular test sample, in millimeters (mm).

4.4.3 Calculate the bulk density of the test sample (D_b) according to Formula (3):

$$D_b = \frac{m_1}{V_b} \times 10^3 \qquad \cdots\cdots(3)$$

Where:

D_b – bulk density of the test sample, in grams per cubic centimeter (g/cm^3);

m_1 – mass of the test sample, in grams (g);

4.4.4 The calculated value shall be rounded to two decimal places and the numerical value shall be rounded off in accordance with the provisions of GB/T 8170.

4.5 Allowable error

The allowable error in bulk density determination shall not exceed 0.01 g/cm^3.

5 Determination of true density

5.1 General

This Chapter specifies two methods for determining true density - Method 1 (boiling method) and Method 2 (helium method).

5.2 Method 1: Boiling method

Warning – The absolute ethanol used in this method is flammable. When heated on an electric stove, an asbestos mesh must be used and the heating temperature shall be controlled.

5.2.1 Reagents

5.2.1.1 Absolute ethanol: not less than 99.7%, analytical grade or above.

5.2.1.2 Sulfuric acid: chemically pure or above.

5.2.1.3 Acetone: chemically pure or above.

5.2.2.5 Blast drying oven: equipped with automatic temperature control device, capable of heating to 200 ℃.

5.2.2.6 Desiccator: containing desiccant.

5.2.2.7 Filter paper.

5.2.2.8 Electric hot plate or thermostatically controlled electric stove.

5.2.2.9 Thermometer: (0 ~ 50) ℃, graduation value 0.1 ℃.

5.2.2.10 Beakers: 300 mL, 500 mL, 1000 mL.

5.2.2.11 Sand bath.

5.2.2.12 Jaw crusher.

5.2.2.13 Crusher.

5.2.2.14 Square-hole standard sieve: pore size 0.15 mm.

5.2.3 Sample preparation

5.2.3.1 Take samples of carbon materials in accordance with GB/T 1427; if necessary, crush the samples to less than 1 mm [If the samples are damp, they shall be first dried in a blast drying oven at (150 ± 10) °C for 20 min]; reduce the samples to (50 ~ 60) g by quartering; crush all to pass through a 0.15 mm square-hole standard sieve.

5.2.3.2 The number and mass of incremental samples of calcined coke and calcined anthracite shall be in accordance with the industrial analysis samples in GB/T 1997, and the laboratory preparation procedures shall be the same as above.

5.2.4 Test procedures

5.2.4.1 Calibration of density bottle

5.2.4.1.1 Determination of density bottle mass (m_2):

First, soak the density bottle in a saturated solution of potassium dichromate in concentrated sulfuric acid for (1 ~ 2) hours; then take it out (if it is a new density bottle or it has not been used many times, inject hydrochloric acid solution to 2/3 of the bottle and boil it on a sand bath for more than 30 minutes); use water to rinse it; then use absolute ethanol and acetone to wash it respectively; use detergent powder to clean the outer wall of the bottle. Finally, use distilled water to clean the inner wall of the bottle; place it in a drying oven and dry it at 120 ℃ ± 5 ℃ for 2 h; take it out and place it in a desiccator; cool it to room temperature; weigh it with the reading accurate to 0.000 1 g. Repeat the above operation by using distilled water to clean the inner and outer walls of the bottle several times and measure its mass. The weighing error shall be within

0.000 4 g for at least three times. Take the average value as the density bottle mass, which is m_2.

5.2.4.1.2 Determination of density bottle water value (mass m_3 of density bottle plus distilled water):

Fill bubble-free distilled water into the density bottle in 5.2.4.1.1 (distilled water can be boiled before use) and place it together with the dropping bottle in a constant temperature water bath. The water level in the water bath shall be slightly above the scale line of the density bottle or level with the bottle mouth of the capillary density bottle. Keep the temperature at (25 ± 0.2) ℃ for 20 minutes. When the scale line of the density bottle is not exposed above the water surface, use a filter paper roll or a dropping bottle to suck out or add distilled water in the density bottle so that the liquid level is accurately at the scale line; wipe the inner wall above the liquid level clean (if a capillary density bottle is used, the stopper shall be immediately covered). Take out the density bottle; use a clean absorbent towel to carefully wipe the outside of the density bottle; quickly weigh its mass with the reading accurate to 0.000 1 g. Repeatedly inject distilled water and measure its mass according to the above operation. The weighing error of the density bottle water value shall be no more than 0.002 4 g for at least 3 times. Take the average value, which is the density bottle water value, that is, m_3.

5.2.4.1.3 The density bottle water value shall be calibrated every three months. If the frequency of use is low, it can be delayed appropriately.

5.2.4.2 Determination of true density of test sample

5.2.4.2.1 Determination of true density of test sample wettable by distilled water

Weigh 3 g of the sample as m_4, with the reading accurate to 0.000 2 g; place it in a clean density bottle; fill bubble-free distilled water (distilled water can be boiled before use) to 2/3 of the bottle and boil for 3 minutes; do not allow the sample to splash out at this time. After removing the bottle, inject bubble-free distilled water (distilled water can be boiled before use) to a level slightly above the scale line or to the same level as the mouth of the capillary density bottle. Put it together with the dropping bottle injected with distilled water into a constant temperature water bath. The water surface of the water bath shall be slightly higher than the scale line of the density bottle. Keep at (25±0.2) ℃ for more than 30 minutes. When the scale line on the density bottle is not exposed above the water surface, use a filter paper roll or a dropping bottle to adjust the distilled water level to the scale line and wipe the inner wall above the liquid surface (if using a capillary density bottle, the stopper shall be covered immediately). After taking it out, use a clean and absorbent towel to wipe the outside of the bottle carefully and quickly weigh its mass, which is m_5.

5.2.4.2.2 Determination of true density of test sample wettable by absolute ethanol

Weigh 3 g of the test sample, which is m_4; read the value to the nearest 0.000 2 g; place it in a dry density bottle. Boil the absolute ethanol in the beaker and inject it into a blank

5.3.2.6 Square-hole standard sieve: pore size 0.075 mm.

5.3.2.7 Drying oven: equipped with automatic temperature control and air blowing device, and capable of maintaining the temperature within the range of (110±5) ℃.

5.3.2.8 Glass desiccator: containing color-changing silica gel or granular anhydrous calcium chloride.

5.3.2.9 Analytical balance: scale graduation value not less than 0.000 1 g.

5.3.3 Test sample

5.3.3.1 Take samples in accordance with the provisions of GB/T 1427 or relevant product standards.

5.3.3.2 Crush the samples to a size of less than 4 mm; mix thoroughly; use the quartering method to separate at least 100 g of samples; grind all the samples to a particle size of less than 0.075 mm.

5.3.3.3 Place the prepared samples in a drying oven and dry at (110 ± 5) °C for at least 2 h; remove the samples and place them in a desiccator to cool to room temperature before testing (the desiccator must be placed in the same place as the true density tester).

5.3.4 Test procedures

5.3.4.1 Turn on the true density tester; introduce helium; preheat for at least 30 minutes; check the air tightness of the gas line.

5.3.4.2 Calibrate the instrument using standard samples.

5.3.4.3 Place the samples in the sample cup, with the sample volume not less than 2/3 of the sample cup volume and not exceeding the scale line inside the sample cup. When filling the samples, vibrate the sample cup to ensure that the samples are filled tightly; remove the samples above the scale line on the inner wall of the sample cup and the sample adhering to the outer wall; accurately weigh the mass of the samples to an accuracy of 0.000 1 g.

5.3.4.4 Place the sample cup on the test stand and tighten it; enter the test parameters according to the instrument manual; let it stand for 5 ~ 10 minutes before measuring.

5.3.5 Test data processing

5.3.5.1 Calculate the true density of the sample (D_t) according to Formula (8):

$$D_t = \frac{m_4}{V_2} \qquad \cdots\cdots (8)$$

Where:

6 Determination of true porosity

Calculate the true porosity of the test sample according to Formula (10):

$$P_t = \frac{D_t - D_b}{D_t} \times 100\% \quad \cdots\cdots (10)$$

Where:

P_t – true porosity of the sample (volume fraction), %.

The result shall be rounded to two decimal places and the numerical value shall be rounded off in accordance with the provisions of GB/T 8170.

7 Determination of open porosity

7.1 General provisions

This Chapter specifies two methods for determining open porosity - Method 1 (vacuum method) and Method 2 (boiling method).

7.2 Method 1: Vacuum method

7.2.1 Instruments and apparatuses

7.2.1.1 Blast drying oven: equipped with automatic temperature control device, capable of heating to 200 ℃.

7.2.1.2 Vacuuming device: The vacuum degree can reach 0.098 MPa (see Figure 3).

7.2.1.3 Analytical balance: provided with hanging hook, scale value (0.01 ~ 0.000 1) g.

7.2.1.4 Beaker: 1000 mL.

7.2.1.5 Square plate.

7.2.1.6 Grid: woven from metal mesh.

7.2.2 Test sample

7.2.2.1 Samples shall be sampled and processed in accordance with GB/T 1427.

7.2.2.2 The sample size shall be Φ(45±0.1) mm × (40±0.1) mm, Φ(40±0.1) mm × (40±0.1) mm; or other regular cylindrical or rectangular samples whose mass shall not be less than 30 g.

7.2.3 Test procedures

7.2.3.1 Place the sample in a drying oven at (110 ± 5) °C and dry it to constant weight. The error between 2 weighings shall not exceed 0.02 g. After cooling to room temperature, weigh the sample to obtain m_8.

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