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Basalt fiber - Determination of combustible matter content

玄武岩纤维 可燃物含量的测定

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Basalt fiber - Determination of combustible matter content

1 Scope

This document specifies the principles, instruments and materials, samples, operations, results presentation, precision, and test report for the determination of combustible matter content in basalt fibers.

This document describes two test methods for determining the combustible matter content of basalt fibers, namely:

- --- Method A: Muffle furnace burning method;
- --- Method B: Soxhlet extraction method.

Method A is the arbitration test method.

This document applies to basalt fiber yarns, chopped strands and milled fibers, fabrics, felts, and other products.

2 Normative references

The following documents contain the provisions which, through normative reference in this document, constitute the essential provisions of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

GB/T 18374 Terminology for reinforcements

3 Terms and definitions

Terms and definitions defined in GB/T 18374 apply to this document.

4 Principles

The principles of the methods are as follows:

--- Method A Muffle furnace burning method: Under the specified conditions, place the dry sample at a temperature of (425±20) °C and burn, and weigh the mass of the sample before and after burning.

--- Method B Soxhlet extraction method: When the sizing agent can be completely dissolved in a certain solvent, extract the sizing agent of the sample with a Soxhlet extractor, and weigh the mass of the sample before and after removing the sizing agent.

5 Instruments and materials

5.1 Method A: Muffle furnace burning method

- **5.1.1** Ventilated oven: The temperature can be controlled at (105 ± 3) °C.
- **5.1.2** Muffle furnace: The temperature can be controlled at (425 ± 20) °C, and the temperature shall be measured at the center of the Muffle furnace after the furnace door is closed.
- **5.1.3** Desiccator: It shall include a suitable desiccant inside, such as silica gel, calcium chloride, and phosphorus pentoxide.
- **5.1.4** Sample pan: It shall be made of heat-resistant materials such as ceramics or stainless steel, it can maximize the air circulation on the surface of the sample and prevent the loss of the sample.
- **5.1.5** Stainless steel clamp: It is used to hold the sample and the sample pan.
- **5.1.6** Balance: It shall be accurate to 0.1 mg.
- **5.1.7** Polished metal template: It is used to prepare the sample.
- **5.1.8** Appropriate cutting tools: They are used for cutting felt or fabrics, such as knives, scissors, disc knives, or punching devices.
- **5.1.9** Appropriate yarn winding machine: It is used to reel yarn samples.

5.2 Method B: Soxhlet extraction method

Warning: Extraction and handling of all organic solvents shall be performed in a fume hood.

- **5.2.1** Balance: It shall be accurate to 0.1 mg.
- **5.2.2** Ventilated oven: The temperature can be controlled at (105 ± 3) °C.
- **5.2.3** Desiccator: It shall include a suitable desiccant inside, such as silica gel, calcium chloride, and phosphorus pentoxide.
- 5.2.4 Soxhlet extractor: The capacity shall be 200 mL, and a 500 mL flask shall be

The size of the sample may be the same as the sample for which the mass per unit area is determined.

Woven fabric samples shall be cut at least 10 mm distance from the selvedge or selvage. If the sample must be folded, it shall not obstruct the passage of air over the entire surface of the sample. It is recommended to use a template (5.1.7) and a cutting tool or punching device (5.1.8) to cut out the sample to avoid material loss.

6.1.4 Felt

The mass of the sample shall not be less than 5 g. The recommended sample is a square (0.1 m²) of 316 mm×316 mm, and other shapes with a surface area close to 0.1 m² can also be used. If other shapes are used, appropriate modifications shall be made to the following sample preparation procedures.

If more than one square must be used to achieve the minimum mass of the sample, these square samples shall be cut along the same line in the length direction of the felt roll.

Cut a strip of felt at least 316 mm long across the width of the felt, and use a template and cutting tools to cut the samples from the strip:

- a) Cut a 316 mm×316 mm sample from each end of the felt strip (for the felt strip with trimmed edges, at least 10 mm from the edge);
- b) Between the samples at both ends, cut as many 316 mm×316 mm samples as possible (these samples shall be evenly distributed).

6.2 Preparation of samples

6.2.1 Yarn

In order to allow the air to circulate freely as much as possible and allow the samples to dry and burn completely, the samples shall not be subjected to excessive compression. Tie each sample into a loose knot so that it can be weighed on the balance (5.1.6).

6.2.2 Chopped strands and milled fibers

Put them into the sample pan (5.1.4), stack them naturally, do not compact them, and they cannot be higher than the sample pan.

6.2.3 Fabrics and felts

The samples can only be cut and stacked, not folded, so as to be placed in a sample pan suitable for the equipment [such as ovens (5.1.1), Muffle furnaces (5.1.2), balances]; for felt samples, the height of placement cannot be higher than the sample pan.

6.3 Number of samples

sample mass is constant.

- **7.1.3.2** Take the sample pan and the sample out of the oven together, and place them in a desiccator to cool to room temperature.
- **7.1.3.3** Weigh the total mass of the sample pan and the dried sample, and record it as m_1 ; the weight shall be accurate to 0.1 mg.
- **7.1.3.4** Repeat heating, cooling and weighing until the mass is constant.

7.1.4 Burning of the sample and mass weighing

- **7.1.4.1** Use stainless steel clamps (5.1.5) to hold the sample pan, put the sample pan and the dried sample into the Muffle furnace, ensure that the sample does not touch the furnace wall, and control the furnace temperature at (425 ± 20) °C.
- **7.1.4.2** Open the furnace door and let the sample burn for 5 min. Then, close the furnace door and burn for 60 min.

Note: Open the furnace door to allow volatiles to escape out of the furnace to prevent condensation from depositing on the sample or sample pan. If using a ventilated Muffle furnace, no need to open the furnace door.

- **7.1.4.3** Transfer the sample and sample pan from the Muffle furnace into a desiccator and cool to room temperature.
- **7.1.4.4** Weigh the total mass of the sample pan and the sample after burning, and record it as m_2 ; the weight shall be accurate to 0.1 mg.
- 7.1.4.5 Repeat heating, cooling and weighing until the mass is constant.

7.1.5 Result representation

Calculate the combustible matter content of the sample according to formula (1), and express it by the mass fraction of the dried product:

Where:

C --- The combustible matter content;

 m_0 --- The mass of the sample pan, in grams (g);

 m_1 --- The mass of the dried sample and the sample pan, in grams (g);

 m_2 --- The mass of the sample and the sample dish after burning, in grams (g).

Take the arithmetic mean of 3 measured values as the test result of combustible matter content.

7.2 Method B: Soxhlet extraction method

7.2.1 Operation

- **7.2.1.1** Put the filter cartridge (5.2.5) into a ventilated oven (5.2.2) with a temperature of (105 ± 3) °C to dry for 1 hour, and then put it into a desiccator (5.2.3) to cool to room temperature.
- **7.2.1.2** Put the sample into a ventilated oven with a temperature of (105 ± 3) °C to dry for at least 1 h until the mass of the sample is constant, and then put it into a desiccator to cool to room temperature.
- **7.2.1.3** Weigh the mass of the filter cartridge (m_3) , and the weight shall be accurate to 0.1 mg.
- **7.2.1.4** Put the dried sample into the filter cartridge, and weigh the mass (m_4) of the sample and the filter cartridge; the weight shall be accurate to 0.1 mg.
- **7.2.1.5** Put the filter cartridge and sample into a Soxhlet extractor (5.2.4), and put enough organic solvent (5.2.9) in the flask to ensure the circulation of reflux.

Note: If the fibers are not loose and do not fall off during the extraction, it is not necessary to use a filter cartridge.

- **7.2.1.6** Adjust the power of the heating furnace (5.2.8) to ensure that at least 8 refluxes are completed within 2 h, and the extraction is performed for 2 h (if it can be determined that the extraction is complete, the extraction time can be shortened).
- **7.2.1.7** Turn off the heating furnace, cool for 10 min; take out the filter cartridge and the sample, and leave them for 10 min to evaporate the excess solvent on the surface.
- **7.2.1.8** Put the filter cartridge and the sample into a ventilated oven at (105 ± 3) °C to dry for 1 hour, then put them into a desiccator to cool; weigh the mass (m_5) of the sample and the filter cartridge, and the weight shall be accurate to 0.1 mg.
- **7.2.1.9** Repeat the above drying and weighing until the difference between the two consecutive weighing results (m_5) is not greater than ± 0.2 mg. This step can be omitted if the minimum time for the sample to dry to a constant mass can be determined.

7.2.2 Result representation

Calculate the combustible matter content of the sample according to formula (2), and

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