1 Determination of Apparent Porosity

The Evacuation Method

General:- The evacuation method of the determination of apparent porosity is applicable to all types of refractories.

Evacuating Apparatus:- An apparatus capable of reducing the pressure to a value not greater than 25 mm of mercury, shall be used.

Immersion Liquid:- Water may be used, unless the test material is unstable in contact with it. Liquid paraffin may be used with all types of refractory material but it shall be fractionated before use and the fraction boiling below 200°C shall be rejected.

Test specimen - Test specimen measuring 65×65×40 mm shall be cut form the brick or shape as to remove the original surface of the refractory under test. For this purpose a cut-off grinding wheel is recommended. Any loosely adhering grog or dust shall be removed from the test piece before use.

Procedure

- Dry the test specimen at 110°C and weigh after cooling to room temperature in a desiccator. This weighing and all subsequent weighings shall be made to an accuracy of 0.1 g.
- Place the test specimen after weighing in an empty vaccum desiccator. Reduce the pressure in the desiccator to a value not greater than 25 mm of mercury. Admit the immersion liquid slowly till the specimen is covered. Allow the specimen to stand under reduced pressure for at least 5 to 6 hours. Air shall then be allowed to enter the vessel and the test specimen held in a sling of fine thread, shall be weighed (S) while suspended in the immersion liquid of the same density as that in the desiccator.
- Lift the test piece slowly from the immersion liquid by means of a sling. The sides of a test piece will drain during this operation and the globules of liquid that form on the under-sided and between the sling and the top surface shall be removed by brief contact with the edge of a piece of filter paper. Care should be taken that the filter paper does not come into contact with the surface of the test piece since the removal of too much liquid will lead to large errors than the removal of insufficient liquid. Then weigh the soaked test piece suspended in air (W).
- Obtain the exterior volume (V) in cubic centimeters of the test specimen by subtracting the suspended weight (S) from the saturated weight (W) and the actual volume of open pores (V1) in cubic centimeters by subtracting the dry weight (D) from the saturated weight (W).

Calculation

Obtain the apparent porosity (P) in percent from either of the following formulae

$$P = \frac{V_1}{V} \times 100 \qquad\qquad P = \frac{W - D}{W - S} \times 100 \tag{1.1}$$

The Boiling Water Method

General - The boiling water method of the determination of apparent porosity is applicable to burnt bricks only.

Test Specimen - Test specimen as given under 9.1.4 shall be used.

Procedure

1. Dry the test specimen at 110°C and weigh after cooling to room temperature in a desiccator. This weighing and all subsequent weighings shall be made to an accuracy of 0.1 g.
2. Place the test specimen in distilled water and boil for two hours, and then allow to cool to room temperature while still immersed in water. During boiling, ensure that the test specimen is not in contact with the heated bottom of the container.
3. Heat the specimen in a container which does not give off scale. After boiling and cooling, weigh the test specimen (S) while suspended in water.
4. Immediately after obtaining the suspended weight remove the test specimen from water, blot lightly with a moistened towel and weigh in air (W).
5. the exterior volume of the test specimen (V) and true volume of open pores (V1) as in 9.1.5.4 and calculate the apparent porosity as given in 9.1.6.

Determination of true specific gravity and true density

Objective

This test determines the true specific gravity and true density of refractory materials under prescribed conditions. It is not applicable to materials attacked by water.

Apparatus

Analytical Balance and Weights
50-ml Pycnometer Bottle with Capillary Tube Stopper
Thermometer
Drying Oven
Weighing Bottle
Desicator
A suitable apparatus to produce a vaccum of 12 to 25 mm mercury pressure.

Preparation of Sample

1. Take two pieces of the size of a walnut from different positions of a solid specimen in such a way as to exclue any part of the original exterior surface (skin).
2. Crush the pieces between hardened steel surfaces to a maximum particle size of 3 mm thoroughly mix the crushed material and reduce by quartering to a test sample of 50 g.
3. If the material submitted for testing is already crushed or ground thoroughly mix a representative portion of at least 500 g and reduce by quartering to a test sample of 50 g.
4. Grind the entire 50-g sample in an agate mortar to such a fineness that it will pass through IS Sieve 15. Do the grinding either by hand or by mechanical sample grinder so constructed as to prevent the introduction of any impurity
5. Remove by a magnet any magnetic material introduced in crushing or grinding

6. Take care in all stages of preparation of the test sample not to exclude any portions that are difficult to grind, and avoid any selective sampling.

Procedure

1. Dry the 50 g sample to constant weight at 105°C to 110°C and place in a glass stoppered weighing bottle. Make duplicate tests on material from the 50-g sample. Record all weights to the nearest 0.001 g.

2. Dry the pycnometer and stopper at 105°C, cool in a desiccator, weigh (p) on an analytical balance. Then fill the pycnometer with distilled water at room temperature (t°C), and again weigh (W1) with the stopper in place. Then empty the pycnometer and again dry.

3. Place approximately 8 to 12 g of the sample in the dry pycnometer; weigh the pycnometer, stopper and sample (W). Fill the pycnometer to one-fourth to one half of its capacity with distilled water, and boil the water (See Note 1) at atmospheric or under reduced pressure for approximately 10 to 15 minutes. Afte boiling, fill the pycnometer with distilled water (See Note 2), cool to room temperature (t°C) in a water-bath, insert the stopper, wipe off excess water from the stopper and the pycnometer thoroughly with a lintless towel. Then weigh the pycnometer and contents (W2).

Note 1: Exercise caution during boiling so that it is not sufficiently vigorous to cause loss of the sample due to popping. If the boiling is done at atmospheric pressure, it is advisable to insert with the stopper a thin strip of paper before the boiling operation.

Note 2: Fill the pycnometer so that there is an overflow of water through the capillary tube when the stopper is inserted. When wiping the excess water from the tip of the stopper, do it in such a way as not to withdraw any water from the capillary tube. any variation in room temperature (t) when obtaining weights (W1) and (W2) will introduce an appreciable error. Therefore, obtain them at the same temperature within the limits of 0.3°C. Use a constant temperature bath controlled to 0.1°C.

Calculations

Calculate the true specific gravity in accordance with the following formula:

$$\text{Specific gravity} = \frac{W - P}{(W - P) - (W_1 - W_2)} \tag{1.2}$$

where t = temperature of the material and the water in °C, W = weight in g of the stoppered pycnometer and sample, P = weight in g of the stoppered pycnometer, W_2 = weight in g of the stoppered pycnometer, sample, and water, and W_1 = weight in g of the stoppered pycnometer filled with water.

The true density of the sample may be determined without additional measurement in accordance with the following formula :

$$\text{True Density} = \text{Specific gravity} \times (d_w - d_a) \tag{1.3}$$

where d_w = density of water at the temperature at which the test was carried out, and d_a = density of air at the temperature at which the test was made.

Report the determinations to the nearest 0.001. Calculate also the following from the obtained results.

Note: These are however, not included in the said I.S.Specification
i) Bulk density ii) Water absorption

Porosity

Porosity Effects:
Density (Bulk),
Thermal Conductivity
Thermal resistance of the body
Strength at room temperature as well as at high temperature
Permeability
Corrosion or Chemical attack
Surface properties (smoothness etc.),
Absorption

TYPES OF PORES Open or apparent,
Closed or sealed
Continuous or isolated
Size of pore : big, medium, small
Distribution : Well distributed or appearing as defects such as holes, cracks, etc.

Porosity is always expressed in percentage.
Apparent porosity (Open)% : (Open pore volume/Bulk volume)×100
Sealed porosity % : 100 × (Sealed pore volume/B Volume)
True porosity %: 100 × (Total pore volume/B Volume)

Open Porosity

Open porosity is found out from:
Wt. of the body in air: W
Wt. of the body in water: D
Saturated wt. of the body with water: S

Sealed porosity is known by finding out the density of the body (True density).

Total Bulk volume = Open pore volume + closed pore volume + Volume of the material

If water affects the material than other liquid is to be used, and then the density of the liquid has to be taken into account.

Porosity is affected by

- The nature of the material. Certain materials have micro porosity and cannot be easily reduced. Hence porosity of the body prepared from the material is higher, e.g. DIATOMACEOUS EARTH.

- Particle size, shape, arrangement of the particles from which the body is prepared.
- Method of preparation, Hand Moulding, machine or hydraulic moulding, slip-casting, extrusion and fusion etc.
- Amount of water used during fabrication
- Physical and chemical reactions that occur when materials is fired in the furnace to get the final shape such as phase change accompanied by expansion and contraction, bloating, moulding, frothing, decomposition of carbonates, sulphated etc., burning of carbon and organic materials, loss of water of crystallization etc.
- Temperature of firing and time. At higher temperatures the body reduces its porosity due to one or more of the following reasons:
  - Melt formation
  - Crystallisation or recrystallisation
  - Diffusion : Solid-solid, solid-liquid
- Fusion and cast or well sintered refractories may have very low porosities, even less than 1 per cent.
- Normal refractories which have been fired in the furnace to get the final shape have 10 to 30 per cent porosity
- Insulating refractories porosity may vary between 50 and 80 per cent.