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उपयोग के लिए मेटाकेओलिन —
विशिष्टि

**Metakaolin for Use in Cement,
Cement Mortar and Concrete —
Specification**

ICS 91.100.10;91.100.15;91.100.30

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

Today, cement concrete is not just a mix of cement, aggregates and water but may also contain other materials like mineral and chemical admixtures in general aimed at improving the properties of concrete and sometimes to achieve certain specific properties like higher strength and durability.

Metakaolin, a mineral admixture, is a pozzolanic material is obtained from pure or refined kaolintic clay after controlled calcination and grinding, and is available in the country. Experimental results have shown that properties of concrete can be suitably improved with addition of appropriate amounts of metakaolin. Its use in concrete has already been covered, in IS 456 : 2000 'Code of practice for plain and reinforced concrete (*fourth revision*)'. However, a detailed Indian Standard specification was not yet available. This standard has, therefore, been brought out to provide necessary guidance in the manufacture and use of this material.

The composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that specified value in this standard.

Indian Standard

METAKAOLIN FOR USE IN CEMENT, CEMENT MORTAR AND CONCRETE — SPECIFICATION

1 SCOPE

This standard covers the chemical and physical requirements of metakaolin for use in cement, cement mortar and concrete.

2 REFERENCES

The following standards contain provision which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1727 : 1967	Methods of test for pozzolanic materials (<i>first revision</i>)
4032 : 1985	Method of chemical analysis of hydraulic cement (<i>first revision</i>)
4082 : 1996	Recommendations on stacking and storage of construction materials and components at site (<i>second revision</i>)
4305 : 1967	Glossary of terms relating to Pozzolana
6491 : 1972	Methods of sampling fly ash
11578 : 1986	Method for determination of specific surface area of powder and porous particle using low temperature gas adsorption techniques

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4305 and the following shall apply.

3.1 Metakaolin – Pozzolanic material is obtained by calcination of pure or refined kaolintic clay at a temperature between 650°C and 850°C to achieve complete dehydroxylation followed by grinding to a fineness of more than 9 000 m²/kg, when tested by BET method.

3.2 Densified Metakaolin – Metakaolin that has been treated to increase the bulk density by particle agglomeration.

4 CHEMICAL REQUIREMENTS

Metakaolin shall conform to the chemical requirements given in Table 1.

5 PHYSICAL REQUIREMENTS

Metakaolin shall conform to the physical requirements given in Table 2.

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Sampling

6.1.1 The methods and procedure of sampling of metakaolin shall be same as the method given for fly ash in IS 6491. The mixing of the increments to obtain

Table 1 Chemical Requirements
(Clause 4)

Sl No. (1)	Characteristic (2)	Requirements (3)	Method of Test, Ref to (4)
i)	SiO ₂ + Al ₂ O ₃ , percent by mass, <i>Min</i>	94.0	IS 1727
ii)	SiO ₂ / Al ₂ O ₃ , percent by mass, <i>Min</i>	1.15	IS 1727
iii)	Fe ₂ O ₃ +TiO ₂ , percent by mass, <i>Max</i>	3.0	IS 1727 (see also Note 1)
iv)	Al ₂ O ₃ , percent by mass, <i>Min</i>	40.0	IS 1727
v)	Moisture content, percent by mass, <i>Max</i>	3.0	(see Note 2)
vi)	Loss on ignition, percent by mass, <i>Max</i>	2.0	IS 1727
vii)	Alkalies as Na ₂ O equivalent, percent, <i>Max</i>	1.5	(see Notes 3 and 4)
viii)	Pozzolanicity by modified Chapelle test, mg CaO g ⁻¹ mK, <i>Min</i>	800	Annex A

NOTES

- 1 Any standard test method may be used for estimation of TiO₂.
- 2 For determination of moisture content, dry a weighed sample as received, to constant mass in an oven at 105°C to 110°C. Express the loss in mass in percentage as moisture content.
- 3 Requirement of limiting alkali shall be applicable in case metakaolin is to be used in concrete containing reactive aggregate.
- 4 For determination of alkalies, method of test used for determination of alkalies in cement given in IS 4032 may be adopted.

Table 2 Physical Requirements
(Clause 5)

Sl No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Specific surface by BET method, m ² /kg, <i>Min</i>	9 000	IS 11578
ii)	Oversize percent retained on 45 micron IS Sieve (wet sieving), <i>Max</i>	1.5	IS 1727
iii)	Compressive strength at 7 days as percent of control sample, <i>Min</i> (<i>see</i> Note)	100.0	IS 1727

NOTE — In the test method for determination of comprehensive strength of metakaolin cement mortar in accordance with IS 1727, the value of factor N may be taken as one and cement replacement by metakaolin shall be 10 percent.

gross sample and of the laboratory samples to obtain composite sample shall be done thoroughly. A clean and dry laboratory concrete drum mixer provides adequate mixing for the purpose. The amount of metakaolin shall be 10 to 50 percent of the volume capacity of the mixer. The mixing time shall be 5 ± 1 min. A polyethylene film shall be secured on the drum to keep the material in the drum during mixing of the sample.

6.1.2 The sample or samples for the purpose of testing may be taken by the purchaser or his representative or by any person appointed to supervise the work for the purpose of which the metakaolin is required of by the latter's representative.

6.2 Criteria for Conformity

6.2.1 The samples of metakaolin drawn in accordance with 6.1 shall be tested as per 4 and 5. For this, all laboratory samples shall be tested for all physical requirements, moisture content and loss on ignition; and the composite sample shall be tested for all the remaining chemical requirements.

6.2.2 The lot shall be considered passing, if samples meet in all the requirements. The metakaolin may be rejected, if it fails to meet any of the requirements of this standard.

7 STORAGE AND INSPECTION

7.1 The metakaolin shall be stored in such a manner so as to permit easy access for proper inspection and identification of each consignment.

7.2 Adequate facilities shall be provided to the purchaser for careful sampling and inspection, either at the source or at the site of work, as may be specified by the purchaser. For guidance on storage of metakaolin at site, IS 4082 may be referred to. In general, the material shall be stored similar to cement/ fly ash storage depending upon the storage requirement in bags/bulk form.

8 DELIVERY

The supply of metakaolin shall be made in suitable quantities mutually agreed to between the purchaser and the supplier. Where so required by the purchaser, the material shall be supplied in bags (jute laminated, multiply paper, polyethylene lined) or in drum/ tankers.

9 MANUFACTURER'S CERTIFICATE

The supplier/manufacturer shall satisfy himself that the metakaolin conforms to the requirements of this standard and, if requested by the purchaser, shall furnish a certificate to this effect, indicating the results of the tests carried out on the samples of metakaolin.

10 MARKING

10.1 Each bag/consignment of metakaolin shall be clearly and permanently marked with the following information:

- Manufacturer's name and his registered trade-mark, if any;
- The word 'Metakaolin';
- Net quantity of metakaolin, in kg;
- Batch/control unit number;
- Month and year of packing; and
- Address of the manufacturer.

10.2 BIS Certification Marking

The bag/consignment of metakaolin may also be marked with the Standard Mark.

10.2.1 The use of the Standard Mark is governed by the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which a license for the use of the Standard Mark may be granted to manufacturers or producers may be granted from the Bureau of Indian Standards.

ANNEX A

(Table 1)

DETERMINATION OF POZZOLANICITY BY MODIFIED CHAPALLE TEST

A-1 PRINCIPLE

The measure of the quantity of calcium hydroxide reacting with pozzolanic materials defines their pozzolanic activity. One gram of metakaolin is reacted with 2 g of CaO in 250 ml of water. The non-reacted lime is then measured and the result expressed as calcium hydroxide (in mg) fixed by metakaolin. A blank experiment is done similarly without metakaolin to correct the result.

A-2 APPARATUS

The apparatus used (*see* Fig. 1) shall be:

- Stirrer with a teflon magnet;
- Thermometer (0 to 100 °C range);
- Cooling column;
- Erlenmeyer flask with smooth inner surface (volume 750 ml); and
- Weighing balance (accuracy ± 0.0002 g).

A-3 REAGENTS

A-3.1 The reagents used shall be:

- Calcium carbonate for analysis;

- Distilled water;
- Saccharose for analysis;
- Hydrochloric acid (decinormal); and
- Indicator solution: 0.1 percent phenolphthalein in 50 percent alcohol solution.

A-3.2 Preparation of CaO

Prepare CaO as follows:

- Weigh accurately about 3.6 g of pure CaCO_3 in a platinum crucible.
- Calcine for about 30 min at 1 000°C.
- Weigh and calculate the loss of ignition.

NOTE — Verify that the loss on ignition is 44 percent.

A-4 PROCEDURE

Add exactly weighed 1g of metakaolin in powder form into the dry and clean Erlenmeyer flask (*see* Fig. 1). Add 250 ml of distilled water using a pipette. Then add exactly weighed about 2 g of CaO. Place the stirrer in the flask. Place the thermometer and the cooling column. Homogenize the content of the flask and start heating

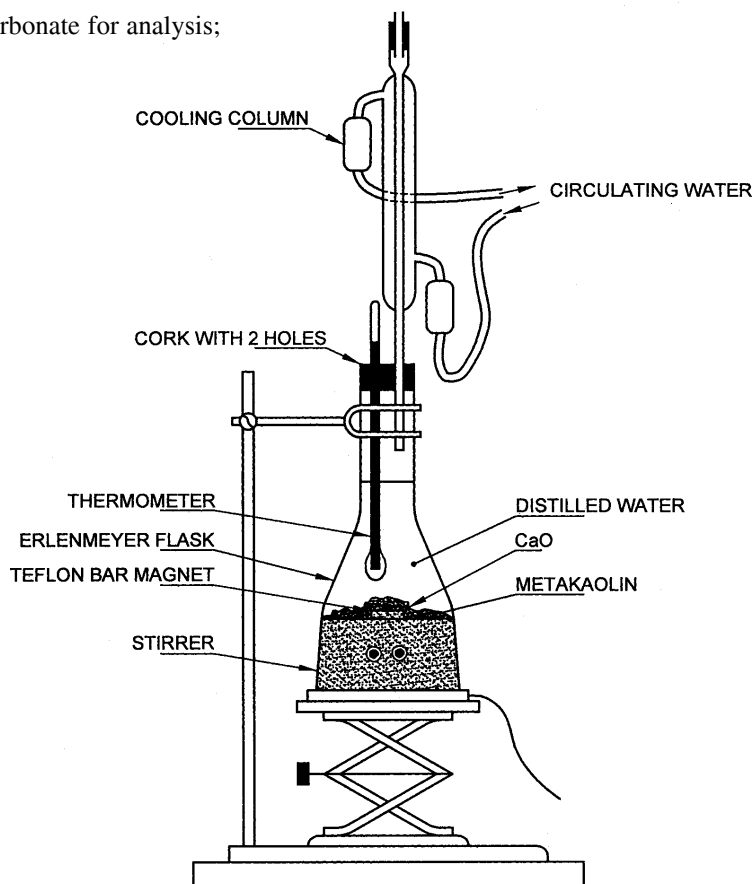


FIG. 1 ARRANGEMENT FOR POZZOLANICITY TEST

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to $85 \pm 5^\circ\text{C}$. Stir and heat for 16 h and then cool to room temperature. Add 250 ml freshly prepared saccharose solution (60 g saccharose dissolved in exactly 250 ml water). Stir for 15 min using magnetic stirrer. Filter about 200 ml and take out 25 ml with a pipette. Repeat the procedure without metakaolin. Titrate with 0.1N HCl solution using the phenolphthalein as indicator and correct the results obtained in the analysis.

A- 5 CALCULATION

$$\text{Ca(OH)}_2 \text{ fixed (in mg)} = 2 \times \frac{V_1 - V_2}{V_1} \times \frac{74}{56} \times 1000$$

where

V_1 = volume of 0.1 N HCl, necessary for the 25 ml of solution obtained by the blank sample in ml; and

V_2 = volume of 0.1 N HCl, necessary for the 25 ml of solution obtained by the reaction of metakaolin in ml.

A-6 RESULTS

The results are expressed in mg Ca(OH)_2 fixed by the metakaolin.

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