

TESTING METHODS FOR POZZOLANAS

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Introduction

Pozzolanas are materials containing reactive silica and / or alumina which on their own have little or no binding property but, when mixed with lime in the presence of water, will set and harden like a cement. They are an important ingredient in the production of an alternative cementing material to ordinary Portland cement (OPC).

The Greeks and the Romans were the first civilisations known to use pozzolanas in lime mortars. The Romans used not only crushed pottery, bricks and tiles which formed the first artificial pozzolanas but also found that some volcanic soils were excellent for producing a hydraulic mortar. Nowadays, a wide variety of siliceous or aluminous materials are used for producing pozzolanas, the common materials being calcined clays, pulverised fly ash, volcanic ash and ash from agricultural residues such as rice husks. (For more general information on pozzolanas see the leaflet in the 'Low-cost cements' series entitled Pozzolanas).

Cement or some form of binding agent is a vital element in all types of construction and in recent years the cement market has been dominated by one product, OPC. In many countries of the developing world, OPC is an expensive and sometimes scarce commodity and factor this has severely limited the construction of affordable housing. Many of these countries have large supplies of pozzolanic materials readily available. In volcanic areas such as Central Africa, Central America and Indonesia there are enormous deposits of volcanic ash. In India and other Asian countries rice husk ash (RHA), (the husks can often be difficult to dispose of), provides a good source of pozzolana for use in masonry cements. Although still limited in comparison with the growth of the use of pozzolanas in Europe and the USA, there are a growing number of developing countries now using more and more pozzolanas in combination with lime or OPC.

Pozzolanas, by their diverse and varied nature, tend to have widely varying characteristics. The chemical composition of pozzolanas varies considerably, depending on the source and the preparation technique. Generally, a pozzolana will contain silica, alumina, iron oxide and a variety of oxides and alkalis, each in varying degrees. This presents problems for small-scale manufacturers wishing to use pozzolanas in a lime or OPC - pozzolana mix. Where there are no laboratory facilities available for testing the raw materials, then it is difficult to maintain standards and produce a consistent product.

It is also generally agreed that although the chemical content of a raw material will determine whether or not it is pozzolanic and will react when mixed with lime or OPC, the degree of reaction and subsequent strength of the hydrated mixture cannot be accurately deduced from just the chemical composition (except for a small number of known pozzolanas - see reference 5). In most cases no direct correlation can be found between chemical content and reactivity. Other characteristics of the pozzolana also affect its reactivity, such as fineness and crystalline structure.

It is also argued that because pozzolanas are used for a variety of different applications, such as in mortars, concretes, block manufacture, etc., and mixed with a variety of other materials such as lime, OPC, sand, etc., (which can also radically affect the reaction of the pozzolana), then perhaps it is better to develop a test to determine the desired properties of the mixture in the context for which it is intended. This provides valuable information for specific project applications and can also help determine the general characteristics of a pozzolana for cases where the application of the pozzolana is not specified. This approach, along with that of fineness testing, forms the basis for most field tests.

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Tests are required for a number of reasons;

1. to assess the viability of a new potential pozzolanic deposit
2. to provide quality control on a day-to-day basis as part of a production process
3. to provide long term quality control of the pozzolanic resource

Many of the standard tests specified in the relevant literature and in the national Standards which cover testing of pozzolanas (in India and USA for example), require sophisticated and expensive laboratory equipment to evaluate the pozzolanicity of a particular material. In developing countries where such equipment is beyond the reach of small-scale producers and where such laboratory facilities are often non-existent and many of the consumables hard to source, determining pozzolanicity can be a major problem. The time required to carry out such tests is also often very lengthy, often requiring a month or more for curing samples. This is not such a problem where the aim is to cover points 1 or 3 from the list above, but for short-term day-to-day analysis of the raw materials, a faster, simpler test is often required.

In this brief the aim is to note the various test methods available for determining the reactivity of pozzolanas, highlighting those which are particularly suitable for use by small-scale users of pozzolanas in developing countries. The brief will look quickly at the Standards available and then describe a simple, practical, rapid field tests which has been developed by practitioners in the field.

Test methods

The following tests are an example of Standards developed by countries to allow accurate characterisation of pozzolanic materials. Other countries apart from those mentioned below publish such Standards and these should be referred to where applicable.

There are also even more sophisticated procedures used, such as x-ray diffraction or electron microscopy to determine whether the structure of a pozzolana is amorphous (more reactive with lime) or crystalline, but such tests are beyond the scope of this brief.

Indian standards

The Indian Standard for methods of Test for Pozzolanic Materials (1967) gives a variety of tests for determining various characteristics of pozzolanas. They are briefly described below. The address for the Indian Standards Institution is given in the resource section of this brief for those who wish to study the Standard in more detail (see Reference 3.).

1. *Chemical analysis*

The chemical analysis will determine the following characteristics for pozzolanas:

- Loss on ignition. This is the loss of weight due to release of volatiles on ignition. A sample is ignited in a furnace under controlled conditions and the weight loss measured. This applies to pozzolanas which have to be calcined for use. Given as a weight percentage loss.
- Silica content
- Combined ferric oxide and alumina content
- Ferric oxide content
- Alumina content
- Calcium oxide content
- Magnesia content
- Sulphuric anhydride content
- Determination of soluble salts

These chemical tests are performed using specified reagents. Results of chemical content are given as a weight percentage.

2. *Fineness*

- To determine the specific surface of the pozzolana - given in cm^2/g
- To determine the fineness by sieving

3. *Soundness*

Soundness of a sample (see below) is a measurement of its tendency to crack, distort, pit or disintegrate. Either of the two following tests can be used to establish soundness:

- 'Le Chatelier' method uses a simple expandable ring to indicate the expansion of a sample over a set period of time
- The autoclave method. This method tests for expansion after a certain period of time at elevated temperature in an autoclave.

The sample is prepared in accordance with the Indian Standard for testing cement, but in place of cement a mixture of pozzolana and cement in the ratio 0.2N: 0.8 by weight is used, where,

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

4. *Initial and final setting time*

Simple tests are carried out to determine the setting times for a lime-pozzolana and lime-cement sample. Apparatus used is called the Vicat set

5. *Lime reactivity*

The test for lime reactivity as given in these standards is very similar to the test for compressive strength. A series of 50mm cubes are prepared using a lime : pozzolana : sand mixture. They are allowed to cure for 8 days in an incubator and the compressive strength of the cubes is measured. Results are given in kg/cm².

6. *Compressive strength*

A similar test is carried out as for the lime reactivity given above, but the mix contains cement in place of lime. Compressive strength tests are carried out on specimens which have been incubated for 7, 28 and 90 days. A control test is also carried out using a pozzolana-free mixture. Three 50mm cubes are tested and the average figure used. Again the result is given in kg/cm².

7. *Transverse strength*

The transverse strength test is again similar to the compression strength test but in place of cubes rectangular block (160 x 40 x 40mm) are prepared and tested using specially designed equipment. The ratio of the pozzolana: cement: standard sand mix is 0.2N : 0.8 : 3 by weight (N is given above in section 3).

8. *Drying shrinkage*

A simple test on a 250 x 10 x 10mm block to deduce the shrinkage over a 7 and 35 day period.

9. *Permeability*

A specially designed 'permeability unit' is used to test a series of specimens for porosity. Water is forced under pressure through cured specimens and the passage of water measured. The resulting coefficient of permeability is given in cm/second/unit gradient.

10. *Reduction in alkalinity and silica release*

Only applicable to certain pozzolanas, this test helps to ascertain the effectiveness of some pozzolanas in reducing the harmful effects of alkali-aggregate reaction in concrete. It is a chemical test using reagents to determine the reduction in alkalinity, given in millimoles/litre.

11. *Specific gravity*

A simple measurement of the specific gravity of the raw pozzolana using a piece of apparatus known as the 'Le Chatelier' flask. Given in g/ml.

The above test procedure will give a comprehensive characterisation of any pozzolana. The equipment required to perform such testing is, however, very costly and sophisticated. It is simply not possible in some regions of the world to carry out such tests, and where the application of the pozzolana is such that knowledge of the characteristics on such a level is not critical, then tests of this complexity are not suitable or necessary.

The Indian Standard 'Specification for Lime-Pozzolana Mixture' (IS 4098 - 1967) stipulates specific characteristics of different grades of such a mixture in terms of maximum free moisture content, loss on ignition and proportion retained on a 150 micron sieve; initial and final setting times; compressive strength and moisture retention. The minimum 28 day compressive strength, for example, is specified at between 7 and 40 kg/cm² depending on grade of material.

Characteristic	Requirements		
	Type LP40*	Type LP20*	Type LP7*
1. Free moisture percent, maximum	5	5	5
2. Loss on ignition percent, maximum	20	20	20
3. Fineness, percent retained on 150 micron IS sieve	10	10	-
4. Setting time a/ initial, minimum b/ final, maximum	2 h 24 h	2 h 36 h	2 h 48 h
5. Compressive strength (kg/cm ²) a/ 7 days, minimum b/ 28 days, minimum	20 40	10 20	3 7
6. Water retention (percent of original flow, minimum)**	70	70	70

*Type LP40 for masonry mortars up to GradeMM5
 Type LP20 for Masonry mortars up to Grade MM2 and for foundation concrete
 Type LP7 for masonry mortars up to Grade MM0.5 and for foundation concrete
 **No test is specified for this characteristic, however, in the Standard above.

American Society for Testing and Materials (ASTM) Standard
 The ASTM Standard (ASTM C311 - 77) varies slightly in content from the Indian Standard. Below is given a brief summary of the test methods of the ASTM standard.

Chemical analysis

1. *Moisture content*
 This is determined by drying a sample in an oven and weighing to determine the percentage weight loss.
2. *Loss on ignition*
 The method is similar to that used in the Indian Standard
3. *Chemical content*
 - Silicon dioxide
 - Aluminium oxide and iron oxide
 - Calcium oxide
 - Magnesium oxide
 - Sulfur trioxide
 - Available alkalis

As with the Indian Standard, these tests are carried out using specified reagents and the result is given as a percentage of the total weight.

technical brief

Physical tests

4. *Specific gravity*
Specific gravity is measured using the 'Le Chatelier' flask.
5. *Finenes*
Fineness is calculated after wet sieving a sample of pozzolana on a No. 325 (45µm) sieve.
6. *Soundness*
Soundness of a sample is a measurement of its tendency to crack, distort, pit or disintegrate. The autoclave method is used. This method tests for expansion after a certain period of time at elevated temperature in an autoclave.
7. *Drying shrinkage*
Again 3 specimens (3 of mortar and 3 of concrete) are incubated and measured for shrinkage after 8, 16, 32 and 64 weeks. Length change data, reported as percent increase or decrease in linear dimension to the nearest 0.001% is based on an initial measurement made at the time of removal from the moulds.
8. *Limits on amount of air-entraining admixture in concrete*
Tests are carried out on hardened concrete containing a specified (neutralised Vinsol resin) air-entraining admixtures for compressive strength, flexural strength, resistance to freezing and thawing and length change.
9. *Air entrainment of mortar*
Calculation of the amount of air-entraining admixture required to produce a specified air content in mortar.
10. *Pozzolanic activity index with Portland cement*
The pozzolanicity index is a number based on the compressive strength of sample cubes such that:

$$\text{Pozzolanicity activity index with Portland cement} = A/B \times 100$$

where, A is the average compressive strength of test mix cubes containing pozzolana (psi / kPa), and
B is the average compressive strength of pozzolana free test cube mix (psi / kPa)
11. *Water requirement*
Determination of water required to produce a specified flow in a pozzolana mix.
12. *Pozzolanic activity index with lime*
Similar to the test for pozzolanicity index with Portland cement but using lime. Based on the compressive strength of the cured lime-pozzolana mix.
13. *Reactivity with cement alkalies*
Tests to determine the expansion of mortar due to the alkali-aggregate reaction.

British standards

The British Standards Institute has no specific test to determine the reactivity of pozzolanic material. There is however a test for determining the pozzolanicity of pozzolanic cements (BS EN 196-5: 1995).

Using the Rio-Fratini method, the pozzolanicity is assessed by comparing the quantity of calcium hydroxide in the aqueous solution in contact with the hydrated cement, after a fixed period of time, with the quantity of calcium hydroxide capable of saturating a solution of the same alkalinity. The test is considered positive if the concentration of calcium hydroxide in the solution is lower than the saturation concentration.

technical brief

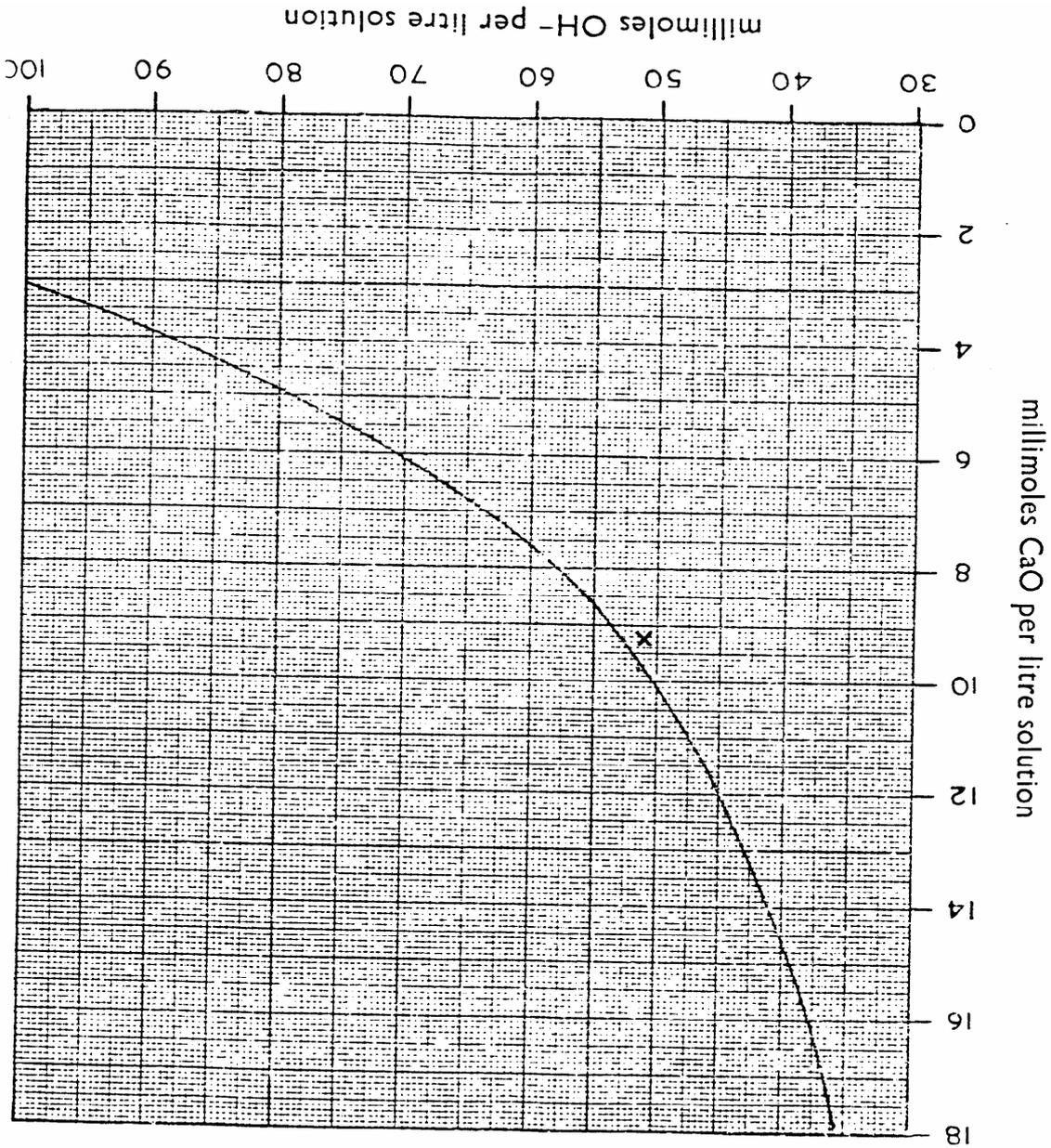


Figure 1. Example of British standard pozzolanicity test - for heap burned rice husk ash sample

Simple field tests

Over the years many field workers have developed simple tests, often based on the tests set out above in the national Standards (and vice versa), to allow rapid evaluation of pozzolanas in the field. Such tests need to be carried out in basic conditions, without sophisticated equipment, in field laboratories, and allow for a reasonably accurate determination of the working characteristics of a pozzolana or cement/lime - pozzolana mixture. The tests are almost universally based on the cube compressive strength and are accelerated to allow rapid testing to be carried out. The fineness test is also useful to meet day-to-day quality control needs as the fineness for a given pozzolana is one of the determinants of strength development. Allen (6) proposes the following criteria for a suitable compressive strength test for pozzolanic mixes:

- Equipment must be simple and test methods should be easy to use in all environments
- Reproducibility should be within acceptable limits
- The relationship between accelerated strength and 28 day strength should hold good for a variety of mixes and a variety of materials
- The results should be suitable in their own right for compliance with specifications and quality control
- Results should be available soon after mixing the test cubes

Robin Spence and J.W. Allen (see reference 1) have suggested a performance related test for pozzolanic materials based on their compressive strength when mixed with lime or Portland cement. The tests were developed in response to the need for testing of pozzolanic materials at a site near Arusha in Tanzania in an area where a large deposit of volcanic pozzolana is found. The test is meant mainly for comparison and quality control purposes. It is suggested that these tests could be used not as legally binding, rigid commitments, but as a guide to the manufacturer where there is no means of carrying out full tests. It is not recommended that this test be adopted in place of the standards which legally apply. It is also recommended, based on the experience of some practitioners, that the 28 day strength test be carried out on a regular basis even where the 7 day test has been carried out after curing at normal temperatures, as the 7 day test might not give a particularly accurate indication of eventual compressive strength with some types of materials.

The test procedure

The rapid field test for pozzolana based mixtures is based on the cube compressive strength test, since it is fairly easy to perform. The first step is the standardisation of the variables involved in each of the steps of the test. These are:

- Size of specimen
- Curing regime
- Type of lime and lime-pozzolana ratio
- Type of aggregate and aggregate cement ratio
- Water content and mixing procedure
- Casting and compaction procedure
- Method of performing compression test

Each of these is considered and a standard procedure adopted.

1. *Size of specimen*
This test uses 25mm cubes which are cast in 9 compartment gang moulds. The cube size is small to allow for compact transportable equipment.
2. *Curing regime*
The curing is carried out over an accelerated period of 7 days. The rate of strength development is sensitive to temperature and results of samples cured at elevated temperature have been found to correlate well with medium and long-term strength

development. The samples are cured in the following manner:

First 48 hours	Simple curing in moulds at 20°C in air
48 - 96 hours	Samples cured under water at 50°C
96 hours to 1 week	Samples cured under water at 20°C

A control sample is also prepared which is not subjected to elevated temperature during curing.

3. *Type of lime and lime-pozzolana ratio*
High-calcium hydrated lime is used, satisfying BS 890. A lime pozzolana ratio of 1:2 is used. This uses slightly more lime than usual for obtaining optimum strength but this allows for incomplete mixing.
4. *Type of aggregate and aggregate cement ratio*
A graded sand satisfying BS 4551 is used with an aggregate: cement ratio of 1:3.
5. *Water content*
The effect of water content on compressive strength is significant and therefore the water content of the sample has to be carefully controlled. To standardise samples, the workability of the sample is measured using the flow test outlined in ASTM C593 - 4.
6. *Mixing procedure, casting and compaction procedure and method of performing compression test*
The mixing procedure is given below:

Dry mix lime and pozzolana	2 minutes
Add estimated water content	2 minutes
Add sand and mix	1 minute
Clean sides of mixing bowl and mix	1 minute
Add any additional water needed (to obtain required flow) and mix	30 seconds

Moulds are filled in three layers, each layer tamped 30 times with a 1 cm² rod and the surface levelled with a trowel. Compression testing is carried out on a strain controlled testing machine at a constant strain rate of 1mm per minute and at least 3 cubes are crushed for each plotted point. A portable machine has been developed for field compression testing.

Flow experiment

As mentioned earlier, the water content of the sample cannot be fixed due to variations in materials being used. More useful as a working parameter is the workability of the mortar mix. To quantify the workability the ‘flow’ of the mortar is measured. A flow of between 65% and 75% is recommended in the ASTM standard C593.

Test equipment

The equipment required for the test is given below.

- *Laboratory mill*
A small laboratory mill able to crush hard rocks to a size of 100µm is required. A machine with a throughput of approximately 10kg/minute is suitable.

technical brief

- *Mixing machine*
A good quality domestic paddle mixer (used in food preparation) will serve the purpose (reference 6).
- *Laboratory burette*
For measuring water quantities. 50cm³ capacity is sufficient.
- *Flow table*
A flow table conforming to ASTM C230 - 68 should be available.
- *Mould*
A mould which gives standard 25mm cubes which can be demoulded without causing damage to the cubes is required.
- *Curing bath*
A curing bath with thermostatic control and circulation pump (for even distribution of water temperature) is required. Temperatures of 50°C should be achieved.
- *Testing machine*
A strain controlled compression test machine is used capable of applying uniaxial compression with a strain rate of mm/minute.

As mentioned earlier the test is purely for means of comparison and quality control and therefore the test results should not be used for satisfying the legal requirements. As the cube size is smaller than those stipulated in the Standards, the results tend to be of a higher value. For more information about this test and the procedures for analysis contact the Intermediate Technology Development Group (see resources section).

References and resources

References

1. BS EN 196-5:1996, *Methods of testing Cement. Pozzolanicity test for pozzolanic cements*, British Standards Institute, 389 Chiswick high Road, London, W4 4AL.
2. IS:1727:1967, *Indian Standard - Methods of Test for Pozzolanic Materials*, Indian Standards Institute, Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Dehli 1, India.
3. ASTM C311 - 77, *Standard methods of sampling and testing fly ash or natural pozzolanas for use as a mineral admixture in Portland cement concrete*. American Society for testing and Materials, 1916 Race St., Philadelphia, Pa. 19103, USA.
4. Allen, W. J., *The evaluation and testing of volcanic pozzolana*, Thesis, 1981.
5. Lea, F. M., *The Chemistry of Cement and Concrete*, Edward Arnold Publishers Ltd., 1937 (Reprinted 1970).
6. Sybertz, F., *Comparisons of different methods of testing the Pozzolanic activity of fly ashes*, from the Proceedings of the Third International Conference on Fly Ash, Silica Fume and Natural Pozzolans in Concrete, Trondheim, Norway, 1989, American Conference Institute, Detroit, 1989.
7. Spence, R., and Allen, W. J., *Lime Pozzolana Cements; testing and small scale production*, The Martin Centre for Architectural and urban Studies, Building technical File, Number 28, 1990.

Useful websites

1. <http://www.aipcr.lcpc.fr/pub-09-e.htm>
PIARC (World Roads Association). Publications related to testing of hydraulic and pozzolanic binder treated materials.
2. <http://www.gtz.de/basin>