

Methods for testing lime in the field

Introduction

If simple, field methods are to be used, it is easier to test the quality of 'lime' when it is in the form of quicklime, i.e. calcium oxide CaO . This would be the case if the lime were being bought direct from a kiln before the kiln operator had started to hydrate it. Usually, though, as quicklime will deteriorate if left exposed to the air and because it can be a hazardous material to handle, lime is more usually available and purchased as hydrated or 'slaked' lime, i.e. calcium hydroxide Ca(OH)_2 .



Figure 1: Loading stone/fuel through the hopper at the chimney level, lime kiln, Chegutu, Zimbabwe
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In either case the primary factor that determines the quality is the percentage of available lime which is the actual chemical 'lime', either as CaO or Ca(OH)_2 , though usually quoted in terms of the equivalent CaO content. Pure quicklime, CaO , will have 100% available CaO , whilst pure hydrated lime, Ca(OH)_2 , will contain 75.56% available CaO .

A minimum available lime content would be specified, for instance, by certain industrial consumers such as sugar refineries. A minimum available CaO , say 60%, specified for a hydrated lime means that it has nearly 80% of the maximum that would be possible. Where lime is to be used for soil stabilisation, such as in roadmaking or in soil blocks, it is still the chemical purity, as available lime, that is important. In this case impurities are less critical but having less available lime means that more 'lime' would have to be transported and consumed.

Soils with high clay content are more in need of high available lime for their stabilisation as the fine clay minerals react in two ways in warm climates – through cation exchange and some pozzolanic reaction.

For soils that are richer in silt and sand it may be preferable to stabilise with Portland cement rather than lime. If lime is to be used it could with advantage be a hydraulic lime. This is not a white lime but is usually grey and though the lime available for reaction may not be as high as with white lime, it has an active, moderately cementitious component to help bond the sand grains in the soil.

Other, simpler, methods for testing quicklime can give an indication of the available lime content, but they ideally need to be done in comparison with a sample of high grade material so as to establish a reference marker. For quicklime these include the measurement of the temperature reached during its hydration, usually in a thermos flask. Hydrated lime, or quicklime that has been slaked to make the dry hydrate, can be tested by determining the strength developed by a mix of lime, pozzolana and sand but this can take several days to complete.

Available lime

An acknowledged standard test for available lime is the ASTM Rapid Sugar Test which uses relatively simple, portable laboratory glassware and common reagents and can be performed wherever there is a small kitchen or table top in a quiet place. There are two procedures depending on whether the acid reagent available is hydrochloric acid or sulphuric acid. The methods are given in Annex 1.

Heat of hydration (for quicklime)

This simple test can also be performed 'in the field' with easily portable items. Though it is primarily for comparing the reactivity of quicklimes, especially for monitoring the burning conditions in a small lime kiln, the maximum temperature reached through the exothermic (heat producing) reaction of quicklime with water is a good indicator of the quality of the lime, at least in terms of the available CaO. The rate at which the temperature rises is an indicator of how reactive it is.

Apparatus

- No. 7 mesh sieve (2.83 mm)
- Thermos flask
- Thermometer reading to at least 100°C.
- Clock or watch with seconds hand.
- Scale to weigh 50 g to ± 0.5 g
- A pestle and mortar or other means of crushing the quicklime to pass the No.7 mesh.

Method

Take several lumps of fresh quicklime, break them with a hammer on a clean surface, cone and quarter to get a representative sample of small fragments. Grind 100–200 g of this with a pestle and mortar, so that it just passes through a No.7 mesh sieve.

Into a thermos flask put 170 ml of water at the normal prevailing water temperature, which in tropical countries may be 23°C. Carefully weigh out 50.0 g of the No.7 mesh quicklime, put it into the thermos flask, start the stop watch and begin gently stirring the mixture.

At one-minute intervals, record the temperature of the water and continue doing so for 24 minutes. Note the maximum temperature (and the time it was reached). By comparing the maximum temperature, and the hydration curve of temperature against time, with those obtained with samples of quicklime of known available lime content, the quality of the sample can be compared and an estimate made of its available CaO content, as well as its degree of reactivity.

Other field tests

Poor quality in hydrated lime can be due to either or all of three causes:

- The original limestone had a relatively low level of calcium carbonate (CaCO_3) and there were other components containing magnesia (MgO), silica (SiO_2), iron, alumina, etc.
- The limestone was poorly burned so that the lime contains some of the original limestone still as CaCO_3 .
- The lime has been left exposed to the atmosphere so that carbon dioxide has converted the calcium hydroxide, Ca(OH)_2 , back to calcium carbonate, CaCO_3 .

In the first two cases, the non-lime components will mostly have been removed by screening and cycloning. Hydrated lime itself, i.e. calcium hydroxide, is very much finer than those impurities, having a specific surface area of around $1.3 \text{ m}^2/\text{g}$, compared with Portland cement which is about $0.4 \text{ m}^2/\text{g}$. So a Fineness Test can be used, passing the lime through a fine mesh sieve, and a good quality lime hydrate should have not more than about 1% retained on a No.85 (0.18 mm) mesh. Note also that a good hydrated lime which does not contain significant quantities of impurities and has not carbonated substantially would be expected to have a bulk density of around 0.5 g/ml (kg/litre), or less, so if the lime sampled has a bulk density higher than this (unless a hydraulic lime) then its quality would need to be questioned.

Where the lime has become carbonated, there may be no immediately obvious change in appearance. What tends to happen is the lime is no longer fine, light and soft but bags of lime become harder and the lime lumpy. Good quality lime should have not more than 6% calcium carbonate. A simple test is to see if there is a reaction with dilute hydrochloric acid. There should be no obvious effect if some dilute, 10%, hydrochloric acid is dropped onto a little of the lime in a watch glass or saucer, apart from some dissolution of the lime. If there is 'fizzing' and bubbles of carbon dioxide, then it suggests that the quality of the lime has deteriorated due to exposure, possibly in inadequate storage conditions over a period of time. Sometimes there is the risk of unsoundness causing disruption of plastered surfaces and possibly also weakness in stabilised soil blocks if the hydrated lime has in it any unhydrated oxide, especially magnesia, MgO. This may occur if the original raw material was a

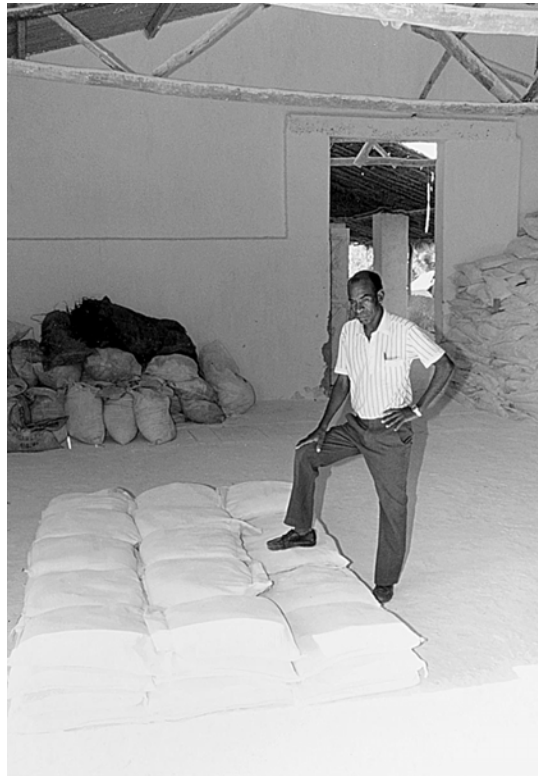


Figure 2: Bagged lime in Chenkumbi, Malawi
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dolomitic limestone. A simple test for soundness is the Pat Test which is one of the two tests for soundness in the British Standard for Portland Cement BS 12, and which can also be applied to testing the soundness of lime. In it three test pats are prepared each in the same way. 70 g of the hydrated lime is mixed with 70 ml of water, covered and allowed to stand for 2 hours. Then, 10 g of plaster of Paris is added and mixed with trowels for 2 minutes and a pat with a flat surface is formed in a ring mould resting on a flat base plate. The three pats are left for 30 minutes to set. They are then dried at 40°C for 12 hours and placed in a steamer and subjected to saturated steam at atmospheric pressure for 3 hours. After moving from the steamer and allowing to cool, the pats are examined for any disintegration, popping or pitting.

The British Standard Specification for Building Limes (BS 890: 1995) does not specify the CaO as 'available lime'. Instead, chemical quality is based mainly on the overall CaO + MgO and the CO₂ content. So, for instance, for Calcium Lime 90 (CL 90) equivalent to 'high-calcium lime', the chemical specification is:

CaO+MgO	MgO	CO ₂	SO ₃
>85 %	<6 %	<6 %	<2.5 %

Note that these values are applicable to all forms of lime. For quicklime these values correspond to the 'as delivered' condition; for all other forms of lime (hydrated lime and lime putty) the values are based on the water free and bound water free product.

There are two other tests which can be done, but these are rather inaccurate and subjective, so an evaluation based on these tests alone should not be taken on good authority. Firstly, both quicklime and hydrated lime are slightly soluble in water, but calcium carbonate is hardly soluble at all. So washing a small quantity (say 5 to 10 grammes) with excess warm sugar solution (the sugar helps the lime to dissolve quicker) for a few minutes would allow quick or hydrated lime to dissolve away. Any solid residue, especially if in the form of a white powder,

is likely to be calcium carbonate and a rough estimate of the proportion could be obtained. Additionally a sample of the quick or hydrated lime can be treated with phenolphthalein indicator. This will turn bright pink if the sample is substantially quick or hydrated lime, but become weakly pink or not change colour if the lime has substantially carbonated. Note that these two indicative tests would work best with high calcium lime. If the lime might be hydraulic, that is contain clay minerals, the tests could be misleading and indicate that you have a poor lime when in fact the lime is completely satisfactory.

Annex 1

Determination of available lime by the rapid sugar test (using hydrochloric acid)
The following procedure is that specified by the ASTM. Boynton considered it to be the simplest, most expeditious method and equal or superior in accuracy to other methods.

Apparatus

- 300 ml Erlenmeyer flask, i.e. a conical flask having approximate volumes marked at certain intervals.
- 100 ml burette, with stand. (A wooden stand can be made locally).
- Balance capable of weighing 0.85 g and 0.5 g to an accuracy of 2%, i.e. to 0.01 g.
- No.100 mesh sieve. (0.15 mm).

Materials

- CO₂ free distilled water, if available.
- Hydrochloric acid (specific gravity 1.18) 15.7 ml per litre of distilled water.
- Methyl orange indicator.
- Phenolphthalein indicator.
- Sucrose – granulated sugar is satisfactory – 15 g.

Method

Take 0.5 g of 100 mesh lime and brush it into a 300 ml Erlenmeyer flask containing 20 ml of CO₂ free distilled water and stopper the flask. Swirl and heat to boiling for 2 minutes. Add 150 ml of water and at least 15 g of sucrose. Stopper the flask, shake at intervals for 5 minutes and allow to stand for 30 minutes to 1 hour. Add 2 drops phenolphthalein, wash down stopper and sides of flask with distilled water, then titrate in the original flask with the standard HCl solution*. Add about 90% of the estimated amount of acid before shaking the flask and then complete titration, with the final acid being fed slowly until the pink colour disappears. Note the reading: 1 ml of the acid solution is equivalent to 1% available lime expressed as CaO.

* Standard HCl solution: 15.7 ml of HCl (specific gravity 1.18) per litre of CO₂ -free distilled water. The solution is standardised against 0.85 g of anhydrous Na₂CO₃ with methyl orange as indicator, so that this amount will neutralise exactly 90 ml of standard HCl solution. In adjusting for this, add more water if it is too strong or more acid if too weak.

Determination of available lime by the rapid sugar test (using sulphuric acid)

A variation on the ASTM test is that which has been used at the analytical chemistry laboratory of the Geological Survey Department in Zomba, Malawi. In place of HCl it uses sulphuric acid (battery acid), H₂SO₄, and gives values for the available CaO which are up to 3% more than those obtained when HCl is used.

Method

- Put 2.500 g of lime into a 250 ml flask, add 35–45 ml of CO₂ -free water and boil for 3 minutes.
- Cool to room temperature. Mix 20 g of sucrose (sugar) in 20 ml of water (= 50% solution) Add this solution to the lime in the flask and shake for 30 minutes. Add more water up to the 250 ml mark.
- Filter through a No.1 Whatman paper. Discard the first 15–30 ml of filtrate. Pipette 25 ml into a flask.
- Add 5 drops of phenolphthalein. Titrate with 0.357N sulphuric acid.

Calculation

The available lime, as CaO, = ml of 0.357N acid, on the burette, x 4.

References

- Boynton, Robert S. *Chemistry and Technology of Lime and Limestone*. Interscience Publishers, New York and London, 1966.
- Holmes, S. and Wingate, M.; *Building With Lime*; Intermediate Technology Publications, London, 1997.

Useful Addresses

basin

Website: <http://www.gtz.de/basin>

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