



# The standard reactivity test as a measure of lime's quality

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## Synopsis

The method used to assess the reactivity of lime was evaluated to gauge its usefulness as a quality control tool. The effect of particle sizes, different lime and water masses for a fixed s:l ratio and the same for various s:l ratios on the reactivity values obtained, were investigated. An attempt to introduce the amount of grit produced during the slaking reaction as an additional measure of lime quality and reactivity, proved to be unsuccessful. It was also found that the relative amount of lime slaked during the different tests was independent of the reactivity value of the lime. The investigation confirmed the usefulness of the accepted test procedure as a reliable method to measure the reactivity of lime, despite various reservations expressed from a theoretical point of view.

## Introduction

Lime and limestone/dolomite are materials often used in a large number of industries, among them the water treatment as well as steel and ferro-alloy production industries. Lime is obtained by calcining limestone and its quality must be controlled in order to satisfy customer expectations. Two industry-accepted tests are normally conducted on unslaked lime to ensure its compliance with specifications, namely the available lime content determination according to SABS standard method 824<sup>1</sup> and the reactivity test described in a German DIN procedure contained in the European Norm 12485 of 1996<sup>2</sup>. The first test mentioned above measures the acid neutralization capacity of the unslaked lime, while the second one describes the kinetics of how fast the material will react with water to produce a calculated  $R_{DIN}$  value or reactivity value. This latter test supposedly gives an indication of how 'reactive' the unslaked lime is.

The classifications of lime's reactivity is as follows<sup>3</sup>:

$R_{DIN}$ value > 30	- highly reactive lime
$10 < R_{DIN}$ value < 30	- reactive lime
$R_{DIN}$ value < 10	- unreactive lime

For steel-making processes and effluent treatment applications, lime of at least a

reactive grade is required. This choice stems from the assumption that high reactivity lime possesses the necessary chemical reactivity to react fast and also to slake well in water.

One of the main problems encountered by customers buying lime revolves around its reactivity. Although logic predicts that there is a relationship between available lime content and reactivity, its extent cannot be calculated from basic principles. This is further aggravated by conditions during the production process and the varying chemical nature and origin of the materials themselves and South African commercial lime in particular.

However, an often forgotten fact that can in many cases explain poor reactivity results obtained in tests, is the test method itself. The standard procedure calls for an amount of 150 g of unslaked lime with a particle size of between 1 mm and 4.75 mm to be added to 600 grams of water at 20°C in a thermostatically isolated container and for the time to be measured that it took the water's temperature to rise to 60°C as a result of the exothermic slaking reaction between the water and the lime. The required  $R_{DIN}$  value is then calculated by dividing 2400 (40°C temperature rise × 60 sec/min) by the time in seconds in which the temperature rise occurred. This technical note will examine the effect of different variables of the method, e.g. particle size, total water and lime amounts and various solid:liquid ratios, on the reactivity values obtained.

## Experimental procedure

Commercial unslaked lime was crushed and sieved in various size fractions, ranging from < 75 μm to 4.75 mm. These various size fractions, or portions from it, were calcined at

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950°C for an hour to ensure that only CaO was present in the material. The necessary mass for each test was then weighed and added to the selected amount of water at 20°C. The mixture was stirred with a mechanical stirrer at 300 rpm and the temperature rise monitored with an ordinary thermometer and stopwatch. At least three repetitions of each test run were conducted to obtain reasonably repeatable values.  $R_{DIN}$  values were then calculated in each case as prescribed by the European Norm<sup>2</sup>. In selected instances the per cent available lime at the start of the test and after completion was also determined. The procedure for this was reported earlier in detail in literature<sup>4</sup>.

An additional test introduced in this investigation in order to try and distinguish between reactivities of different limes, is a determination of the amount of grit produced during the slaking procedure. For this purpose, the lime-water reaction mixture was transferred to a 0.5 mm pore size sieve for screening after the reactivity test. The rationale behind this was that any slaked lime would form very fine calcium hydroxide particles that would easily pass through this sieve size, while the coarser, unhydrated parts and impurities would be trapped by this sieve size. The residue trapped on the sieve was dried in an oven for 3 hours at 100°C and then cooled. After cooling, the residue was gently brushed with a paintbrush until all the fine powder was removed and only the coarser and hard solid particles were left on the sieve. This final residue was weighed to calculate the per cent grit as a percentage of the original mass of lime used in the test.

### Results and discussion

The basis of the reactivity test is the exothermic reaction of unslaked lime with water. From elementary thermodynamic calculations one can deduce that the addition of 150 g of CaO to 600 g of water can raise the temperature of the water from 20°C to more than 80°C (neglecting the small amount of heat transfer and loss to the environment). However, as with all thermodynamic calculations, it does not provide any information on the kinetics of the slaking process, which defines the reactivity of the material. The latter is influenced by practical considerations such as the particle size of the material tested, its chemical composition, as well as production and storage conditions.

Regarding the test itself, the main point of criticism that can be levelled against it, is the wide range of allowable particle sizes that can be used. However, the total amounts of materials used in the test and the recommended liquid:solid ratio can also potentially influence results obtained. Given consistency among these previously mentioned parameters, one should be able to distinguish between limes of different reactivities, regardless of whether these differences are due to their chemical compositions, or production and storage conditions.

Logic would predict that particle size will have an influence on the slaking rate and thus measured reactivity of lime. If so, the large particle size range allowed for in the test procedure should produce various reactivity values if the particle size distributions varies largely between tested samples. Therefore the first variable investigated was the effect of particle size on the measured reactivity. The results

obtained are summarized in Table I.

The results summarized in Table I clearly indicates that the particle size has a major influence on the measured reactivity. In the size range of 1.0 to 4.75 mm, however, the reactivity measured stays more or less constant. One can therefore deduce that in the particle size range called for in the test procedure, the reactivity should not be influenced by a varying particle size distribution between the various samples evaluated. This is in agreement with previous work done by Frank<sup>5</sup> who has found that there is no significant difference between the reactivities of 5 mm and 3 mm lime particles. Furthermore, it seems that the total amount of lime slaked during the test stay more or less the same in all particle ranges. This emphasizes once again that there is not necessarily a correlation between the reactivity or slaking rate of lime and the amount of available lime theoretically available for slaking. The percentage grit does not seem to be as sensitive a measure to gauge reactivity and is therefore not recommended for use to distinguish between reactivity of various limes.

Commercial lime slakers in industry normally operate by using 3 to 4 parts of water for 1 part of unslaked lime. In the test procedure a 4:1 ratio of water to lime is recommended. This led to a decision to include an assessment of different water:solid ratios on reactivity in the investigation. Table II summarized the results obtained in this regard.

The results shown in Table II indicate that there is a direct relationship in the decrease of the reactivity values with an increase in the amount of water used in the test. This can easily be explained from thermodynamic equations that predict a direct proportionality between the amount of heat required and the mass of water whose temperature must be increased. What is interesting though, is that the amount of grit produced in each case is independent of the reactivity values measured. This confirms previous indications that the amount of grit produced is a poor measure of the reactivity of the lime. The tests using 150 g of lime and 300 ml of water

Table I

#### The effect of particle size on lime reactivity

Particle size (mm)	Reactivity/ $R_{DIN}$ value	% Grit (m/m)	$\Delta$ AvCaO (%)
2.0 to 4.75	152 ± 2.0	2.5 ± 0.4	24.2 ± 0.6
1.0 to 2.0	148 ± 2.5	1.3 ± 0.3	24.0 ± 0.4
0.5 to 1.0	113 ± 3.0	1.1 ± 0.1	26.1 ± 0.2
0.075 to 0.5	75 ± 1.5	0.03 ± 0.01	27.6 ± 0.3
< 0.075	26 ± 3.5	0.02 ± 0.01	26.1 ± 0.4

Table II

#### The effect of water:solids ratio on the reactivity of lime

Lime:water ratio	$R_{DIN}$ value	% Grit (m/m)
1:2 (150g/300 ml)	334 ± 15.5	Not determ.
1:3 (150g/450 ml)	244 ± 4.9	0.8 ± 0.2
1:4 (150g/600 ml)	160 ± 2.2	0.7 ± 0.1
1:5 (150g/750 ml)	85 ± 0.6	0.7 ± 0.3
1:6 (150g/900 ml)	Not determ.	0.7 ± 0.3

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resulted in inefficient slaking of the lime and produced an explosive and violent reaction. The difficulty in measuring the reactivity in such cases is reflected in the large standard deviation obtained.

A temptation often arising in laboratories, especially when insufficient material is available for evaluation purposes, is to reduce the required quantities of reagents used in a prescribed method by pro-rata amounts. This could lead to unreliable results being obtained. For this purpose modifications in the amounts of lime and water used in the reactivity test were made while keeping the ratio between them constant at 1:4. The results obtained in this exercise are shown graphically in Figure 1.

The data indicate that the reactivity values stay more or less the same within experimental error. There seems to be a tendency to higher reactivity values when lesser amounts than those prescribed by the standard test method are used, although too little work has been done to make conclusive deductions about this. However, the limited data is enough to caution that using lesser amounts when limited material for testing is available can produce misleading figures reflecting a higher reactivity of the lime than is truly the case.

### Conclusions

The reliability of the industry accepted method for measuring lime reactivity was established within the prescribed parameters of the method. Possible sources of error when not using the prescribed particle sizes and correct amounts of water and lime were investigated and their influences established. This work shows conclusively that the reactivity test yields results reliable enough to use it as a measure to investigate the slaking process of lime. Further investigations in this regard will be reported in other communications<sup>6,7</sup>.

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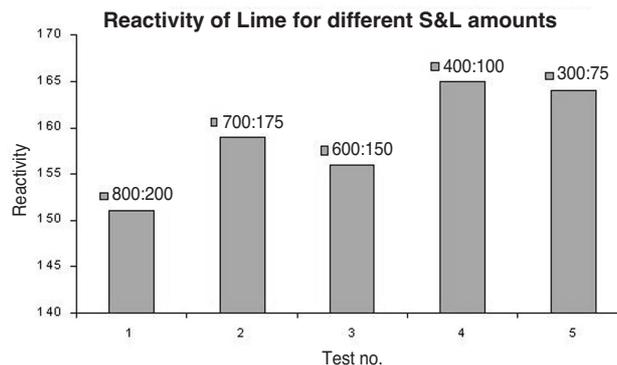


Figure 1—Measure reactivity with a constant water:lime ratio, but using different amounts of lime and water

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## Mineral industry Internet degrees avoid on-line information trap\*

The minerals engineering faculties of four of Australia's leading universities are jointly developing an Internet-based undergraduate degree in mineral processing.

The degree will draw on the best bits from courses offered at the University of Queensland, University of South Australia, Curtin, and Murdoch Universities, delivering material to students enrolled in each of the four institutions.

University of Queensland on-line course developer Ms Diana Drinkwater said that because various universities have expertise in different specialist areas, it means UQ students, for example, will get the best expertise available without having to travel to Adelaide or Perth, and vice versa.

'While elements of the Internet-based degree programme will be trialed over the next two years, any UQ student enrolling in the programme now will have access to all four institutions from first semester 2002.'

Ms Drinkwater said approach being taken by each of the four universities was not to offer an on-line degree as such: 'You don't have to offer an on-line degree to use Internet technology in a constructive way, and we're not saying that this will be an on-line degree.'

She said the degree programme would draw heavily from recent experience gained through Internet-based mining-related courses funded by the Minerals Council of Australia and DETYA, and developed through UQ's Julius Kruttschnitt Mineral Research Centre, Tertiary Education Development Institute, and Learning Resources Development Unit.

Ms Drinkwater's first taste of Internet success came through a simulation and control course designed at the JKMRc in Brisbane and delivered via the Internet to students at the University of Toronto during 2000. The course focused on the learning potential of using the Internet, avoiding the common mistake of uploading a wad of lecture notes, expecting students to download them and at the end of the day gain very little by way of a learning experience.

'We don't want to replace tedious lecture material with tedious digital material—we want to be much smarter about how we use the Internet.'

Ms Drinkwater admits that UQ and partners were not the only people doing it the right way, as there were some very good courses now being offered 'on-line'. But, as a prospective student, you need to exercise caution in your course selection.

'You could probably divide so-called on-line courses into two groups, ideally one which offers the use of the Internet as a medium for active communication,' she said.

'The other type takes lecture material and merely whacks it all on a website called 'on-line learning'—you tend to see this approach with some commercial short courses.'

Ms Drinkwater said the Toronto course was a useful first step towards creating a social constructivism learning environment, much the same way as in a traditional tutorial-based classroom setting. The course centred on a discussion board where students would come on-line during their day, receiving their responses overnight from Australia. As for course material, the study guide was printed and bound and set by mail some time before the course started—it was deliberately not made available on-

line—which meant students had everything there ready to interact as the Internet sessions started. The guide also clearly laid out the ground rules for assessment, which is the driving motivator behind study and learning—facilitated through the Internet.

The JKMRc course has been upgraded to a fourth-year undergraduate subject in mineral engineering, now part of the University of Toronto curriculum in 2002.

'It's as if I'm guest lecturing in the course, but without physically getting on a plane and going to Canada,' Ms Drinkwater said. 'The technology and the learning methodology we are using provides a way for students to access resources in an efficient way.'

UQ's Sustainable Minerals Institute Education Manager Dr Dominic Howarth said the aim with flexible delivery of mining-related courses through the Internet was to enhance the whole student learning process by creating a socially interactive environment using the web.

'A lot of people think that because you are going on-line you can somehow get away from what we used to call teaching, which is really directing student learning activities in a productive way,' he said. 'The Internet gives us many new ways of being able to do that, such as using websites to create quizzes for students, set up discussion groups, even small discussion groups as you would in a class room, so that students can interact with one another and the learning facilitator.'

Ms Drinkwater said one of the first activities students are usually asked to do is to introduce themselves over the 'net and talk a bit about where they come from. They then read everyone else's introductions and they get to know each other.

'This is what you would do in a normal teaching environment and there's some sound pedagogical theory behind all of that.'

Based on this approach, UQ's Sustainable Minerals Institute will offer three Internet-based courses at post-graduate certificate level in 2002:

- ▶ Coal operational management
- ▶ Risk management
- ▶ Mining environmental management.

'People keep talking about on-line degrees, but the benefits of on-line technology are getting pretty obvious now,' Dr Howarth said. 'We can deliver benefits for ourselves and for other universities across Australia and around the world by using internet technology, but it doesn't mean you have to have on-line degree as such.'

'We aim to source the highest quality content and deliver it conventionally or via the Internet—the student is the big winner.' ♦

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