Development of a method for evaluating raw materials for use in iron ore sinter in terms of lime assimilation

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Synopsis

Steel is produced in a basic oxygen furnace from hot metal obtained from a blast furnace. A sintered iron ore with good high-temperature properties (strength and permeability) should be used as feed to the blast furnace. The quality of this sintered ore depends on the reactivity of the iron ore used as feed to the sinter plant during the lime assimilation step in the sintering process. The penetration test is the standard method for evaluating the reactivity of iron ore with lime. It is, however, difficult to determine the exact depth of penetration from the standard test. A new test method is proposed that allows automatic evaluation of iron ores in terms of lime assimilation with increasing temperature. A comparison of the coefficients of variation for the new and standard methods for each ore type demonstrates that the results of the new test are more reproducible and more precise than those of the standard method. The test is also less time-consuming and easier to implement.

Keywords
iron ore, sintering, properties, lime assimilation

Introductions

It is important to determine whether iron ore used as feed to a sinter plant will produce a sinter suitable for use in a blast furnace. The feed to the blast furnace should be permeable (slightly porous, but not too porous since this could have an adverse effect on sinter properties) and have a high strength. A few standard tests are available to evaluate raw materials for use in the sinter process. The current method used can be difficult to interpret, time-consuming, and provides mostly limited information. A new test method, which allows calculation of the reactivity of iron ore with lime with increasing temperatures, was investigated. The new method should be easy to implement, reproducible, quick, and give valid results.

Background

Kumba Iron Ore identified a need to develop a new test method to evaluate iron ore in terms of lime assimilation. Lime is added to iron ore to improve the porosity and to obtain the correct sinter strength and permeability for use in the blast furnace. If the sintered ore does not have adequate strength, a large amount of fines will be produced during stockpiling and handling of ore before use in the blast furnace. These fines will then be blown out by the off-gas from the top of the blast furnace. Insufficient porosity of the sintered ore, on the other hand, will not allow sufficient flow of gas through the ore, causing insufficient reduction of iron in the blast furnace and resulting in a product that is not suitable for steelmaking. The porosity of the sintered ore should, however, not be too high since this can have an adverse effect on the sinter properties.

Higuchi *et al.* (2003, p. 1388) found that different iron ore types react differently during lime assimilation, depending on surface morphology as well as chemical composition. The standard method for determining whether an ore type is suitable for use in the sinter plant is the penetration test. A 5 mm lime tablet is placed on top of a 10 mm iron ore tablet, both pressed from powder. The ore and lime tablets are heated in a furnace and the lime penetrates into the ore tablet. The depth of penetration is used as an indication of the reactivity of the ore type.

Steelmaking

Steel is produced by three processes, of which the blast furnace (BF) and basic oxygen furnace (BOF) combination has been most popular since the nineteenth century. Liquid pig iron (hot metal) produced by the BF is used as feed to the BOF. The solids feed to the BF include iron oxide, metallurgical coke, sintered ore, manganese ore, and dolomite (Buschow, 2001, pp. 4293–4296).

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Blast furnace

Solid material is fed into the BF from the top while hot gas is blown from the bottom upwards, as can be seen in Figure 1. This gas, which consists of a mixture of air and pure oxygen, is used to improve combustion efficiency to reduce the iron ore with metallurgical coke. Molten metal with a slag layer on top collects at the bottom of the BF and the liquid metal is tapped to be used as feed in the BOF.

In order to obtain sufficient contact between the gas and solid particles in the BF, a permeable burden is required to allow a high and uniform gas flow rate (Barker *et al* (2006, p.1393). The iron feed material should not contain excess fines, since the fines will be lost to the top gas.

Barker *et al* (2006, p.1393) also mention that sinter strength is an important characteristic since the sinter used as feed to the BF will be subjected to stockpiling, handling, and transportation. During all of these steps the sinter should not degrade and produce fines, which will be blown out of the BF with the top gas. To ensure that the iron ore feed has sufficient permeability, strength, and correct size, the fine ore is sintered.

Iron ore sintering

Iron ore sintering can be described as the controlled burning of a fuel mixed with iron ore (Barker *et al*, 2006, p. 1393). The process converts natural fine iron ore material, screened iron ore fines, coke, and lime into a fused clinker-like aggregate that can be effectively used in the BF. Iron ore fines are mixed with 5% anthracite, which acts as fuel and is conveyed in the sinter through the process. The mixture of sinter material is fed onto a moving grate, and at the feed end gas burners are used to ignite the top of the bed. As the mixture moves forward, the combustion zone progresses downward due to air flow through the permeable bed. This results in a temperature profile through the thickness of the bed. Temperatures as high as 1300°C to 1480°C are reached at the hottest spot in the bed, causing the particles to fuse together into porous clinker. At the discharge end, sintering would have occurred through the thickness of the bed. The material is then crushed and screened. The oversize material is sent to the BF stockpiling yard and the undersize is returned to the sinter process.

Sintering of the ore has further benefits to the BF operation since the flux is incorporated into the sinter mix instead of being added separately to the BF feed, and it also produces a sized sinter as feedstock with better high-temperature properties (Barker *et al*, 2006, p. 1394).

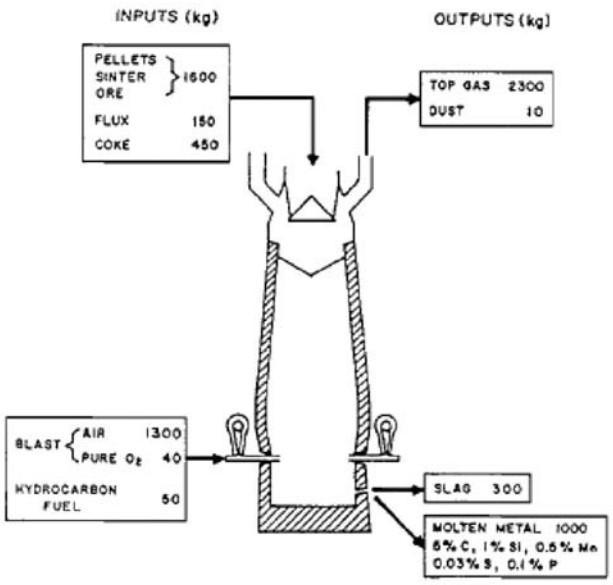

Figure 1—Blast furnace feed materials (Buschow *et al*. 2001, p. 4296)

Lime assimilation during iron ore sintering

With the world-wide increasing demand for iron ore, more lower-quality grades of iron ore are being produced than in the past. As sinter plants have to make use of the ore at hand, it is important to have a quick pre-production test to evaluate whether an ore will be suitable for use in the BF after sintering.

The iron ore sintering process consists of three stages. The first stage is the heating of the burden before melt formation takes place. The second stage is the primary melt formation of pseudo-particles with an adhering fines layer, and the last stage is the assimilation of lime with the nucleus ore (Hida and Nosaka, 2007, p. 103). Various tests are used to determine the properties of the ore during each step. In this project the focus will be on testing the properties obtained during the last stage of the sintering process.

Three tests currently are used in industry to evaluate iron ore in terms of lime assimilation. These include the small packed bed sintering test and the penetration test as described by Higuchi *et al* (2004, pp. 1385-1386), and also the variation of the small packed bed sintering test (Hida and Nosaka, 2007, p. 104).

Since the penetration test makes use of a tablet of iron ore and a second tablet of a combination of lime and iron ore, the conditions in this method are similar to those found in the sintering process. This method was therefore chosen as the standard to be used to evaluate the new developed test method, and is hence the only method described here in more detail.

The standard penetration test

The standard penetration test is used to evaluate the melting of fines into the adhering layer of pseudo-particles during sintering. The test uses two pressed tablets: an ore tablet consisting of an equivalent mass ratio of size fractions -0.25 mm and +0.25–0.5 mm and a primary melt tablet consisting of 26 mass% CaO-FeO). The ore of the two size fractions is mixed and pressed into a tablet 10 mm in diameter and 5 mm in height, while the primary melt reagents are hand-mixed for 20 minutes and pressed into a tablet with diameter and height of 5 mm. Both tablets are produced using an iron mould at a pressure of 0.314 kN.

The primary melt tablet is placed on top of the ore tablet in the centre of a nickel vessel. The sample is heated from ambient temperature to 800°C within 3 minutes, then from 800°C to 1300°C in 2.5 minutes. The temperature is maintained at 1300°C for 2 minutes, after which the furnace is then set to cool over 10 minutes.

A vertical section of the sample is mounted and polished. Macro images at 5× magnification are used to measure the depth of penetration, which is defined as the distance from

the top rim of the tablet to the tip of the reaction zone (Higuchi *et al*, 2004, p. 1386). Figure 2 shows the heated sample with the penetration depth indicated by the arrows. This measurement is used to compare the reactivity of different ore types (Higuchi and Okazaki, 2004, p. 432).

New length reducibility test method

Industry requires an easy test method to determine the assimilation of iron ore with lime during sintering, as there is a need to determine the reactivity of a given type of iron ore with lime and the subsequent porosity and sinter strength. Since the grade of iron ore being mined is decreasing due to the increase in demand, it is becoming even more important to establish whether sintering of a particular ore will result in a suitable product for use in the blast furnace.

A disadvantage of the penetration test is that there is no certainty on the measured depth of penetration, since penetration invariably does not proceed in the perfectly hemispherical fashion described in the literature. The depth often varies across the section and there is uncertainty whether to measure the deepest, the shallowest, or average depth of penetration. Furthermore, the samples sometimes react to such an extent that a completely molten mass is obtained, and no conclusion can be drawn from such results. This test is also not considered to be fully reproducible, since measurement of the penetration depth is done manually.

The alternative test method, which is aimed at achieving conditions similar to those found in the sintering process, makes use of a single cylindrical sample consisting of a mixture of 26 mass% lime and 74 mass% iron ore which is placed in the furnace.

Hewakandamby *et al.* (2013, p. 456) described a test method to determine the behaviour of ash from biomass and coal at elevated temperatures. A cylindrical sample is heated in an ash fusion furnace, and images of the sample obtained at fixed temperature intervals are used to determine the temperatures at certain states, for example the softening temperature.

In the new length reproducibility test method used here, the furnace is equipped with a digital camera to obtain images of the sample with increasing temperature. Matlab™ is then used to convert the digital images to a greyscale image to find a matrix equivalent to the size of each sample. As the samples are heated and started to react, the height of the samples decreases and can be measured as a function of temperature by the software program and a plot of height fraction *versus* temperature obtained. The samples are large enough to allow most samples to be submitted subsequently for additional testing such as sinter strength tests or optical examination to determine the porosity.

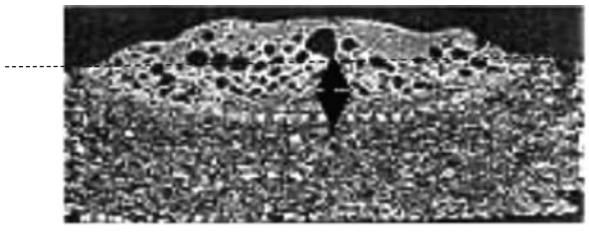

Figure 2—Vertical section through lime and iron ore pellet after sintering (Hida and Nosaka, 2007, p. 104)

Hewakandamby *et al* (2013, p. 454) concluded that the normal ash fusion test used in the past does not produce consistent results as it is based on visual inspection and the results are therefore dependent on the subjective judgement of the person carrying out the experiment. The original ash fusion test as applied to iron ore sintering cannot, therefore, be regarded as inherently reproducible. The method described here is an automated one, with a higher precision than that of the original visually evaluated ash fusion test.

Experimental procedure

The validity of the new length reducibility test method was evaluated by comparing the results with those from a standard penetration test, using various ore types.

Three ore types were tested: two haematite ores, a typical Northern Cape ore in South Africa, a West-African ore, and a goethitic iron ore from Marra Mamba in Australia. All three ore types were tested under the same conditions using both methods, including the quantities of ore and lime used to produce the samples. Eight samples of each ore type were tested in each of the two methods.

Standard test procedure

The standard penetration test procedure used was based on the conditions given by Higuchi *et al.* (2004, pp. 1385-1386). The test was carried out in an infrared furnace, which has a fast enough heating rate to simulate the sinter temperature profile.

Samples and sample preparation

All sampling methods and preparations were done according to ISO 10836 (ASTM part 12), which is equivalent to ASTM E877 (ASTM part 12). The two tablets were placed in the furnace simultaneously, one on top of the other. The top tablet was 5 mm in diameter and height and comprised 26 mass% lime and 74 mass% Fe ore. The bottom tablet, 10 mm in diameter and 5 mm in height, consisted of equal amounts of -0.25 mm and 0.25–0.5 mm pure iron ore.

Given the density of iron ore fines of 5 g/cm^3 (Fe ore MSDS), the total mass of iron ore needed for a 10 mm ore tablet was 1.96 g. In order to limit the number of variables in the test work, all the samples of each ore type were prepared from a single batch mixture. The total mass of iron ore required for the samples for each ore type was milled to the required particle size distribution (equal amounts of -0.5 +0.25 mm and -0.25 mm) and compressed at 0.314 kN in a hand-operated hydraulic press.

The melt tablet consisted of 0.113 g lime and 0.322 g iron ore (5x5 mm site consisting out of 26% lime and 74% iron ore, The ore and lime for all of the samples for each ore type was hand-mixed in a single batch and pressed into a green pellet. When the correct particle size distribution of equal amounts of -0.5 mm +0.25 mm and -0.25 mm, was obtained, the mixed product was pressed into pellets in a hand-operated hydraulic press at a compressive load of 0.314 kN.

The samples were heated in an infrared furnace for the standard penetration test. The ore sample was placed in the furnace with the 5 mm melt tablet on top of the 10 mm ore tablet. The furnace was set to simulate the sinter process. The temperature was increased to 800°C over 3 minutes, then to

1300°C in over 2.5 minutes, and maintained at 1300°C for 2 minutes. The temperature was then decreased to ambient over 10 minutes and the sample removed from the furnace.

The reacted samples were sectioned vertically through the axial centre and mounted in resin. The bottom half of the sample was removed to allow inspection of the centre of the sample by conventional and stereo-optical microscopy. Since the 5× magnification of the sample surface did not allow full inspection of the surface, the stereo microscope was used to take three successive images of the entire surface area, which were combined into a single collage. A distinct defect or grain was used as reference point to allow the entire penetration depth to be measured from the bottom of the sample (ore tablet) up to the point of maximum penetration from the top. This total was then subtracted from the original height of the ore tablet to find the depth of penetration, which could be identified as a darker uniform phase at the top of the tablet, as seen in Figure 3. The bottom granular part consisted of iron ore particles with size fractions of equal amounts -0.25 mm and -0.5 + 0.25 mm.

Length reducibility test procedure

These tests were carried out in an ash fusion furnace equipped with a digital camera to capture the change in height of the various samples as a function of temperature.

Samples and sample preparation

The 10 mm diameter by 5 mm height tablets for these tests consisted of single samples with 26 mass% CaO and 74 mass% iron ore, i.e. a lime to ore ratio equivalent to the penetration tests. The ore comprised equal amounts of -0.25 mm and +0.25 -0.5 mm material, as was the case for the penetration test. The procedure for preparing the powders, mixing them, and pressing them into green pellets was identical to that used in the penetration test.

Heating procedure

Up to four samples were placed simultaneously in the ash fusion furnace by balancing them on the sample carrier in the furnace, with the camera focused on all four samples. The furnace was set to increase the temperature at 7°C/minute and the computer set to take an image of the four samples with each 2°C increase in temperature, starting at 1150°C.

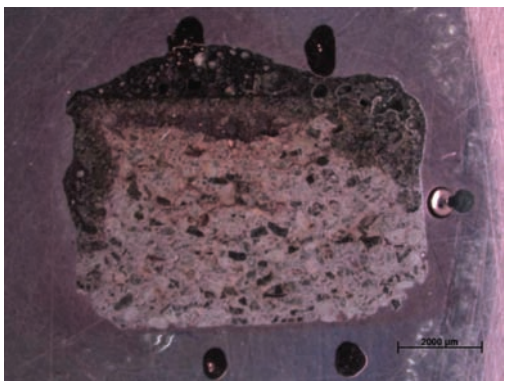

Figure 3—Marra Mamba pellet with darker melt phase penetrating into granular ore phase

Once the furnace reached 1204°C it was switched off and allowed to cool to 400°C before the samples were removed. The temperature of 1204°C was chosen to ensure that the samples did not melt completely and molten material leak onto the furnace tube, thereby cracking it.

Analyses of the samples

The digital images of the samples in the furnace were analysed using a program written in ImageJ specifically for this project. The program requires a manual but accurate rectangular selection of each sample in the image to be drawn based on the initial width and height of the sample. The selection allows the program to set boundaries on where these measurements need to be taken, which allows it to focus exactly on where the sample to be measured is located in the bigger image. The pixels in this rectangle will be counted. After this initial set-up, the image acquired at the maximum temperature is opened together with the image immediately preceding. The program then automatically determines the difference between the two images, thresholds the image, converts it to a binary image, and removes any outliers. These steps are illustrated in Figures 4 and 5.

It can be seen that in the temperature increment from 1202°C to 1204°C, only the Northern Cape ore tablet showed a measurable change in area (Figure 5). After this step the program makes use of a matrix to measure the number of pixels that make up the difference in surface area measured (red pixels in Figure 5). This gives an indication of the total reactivity of the ore type during that temperature increment. The amount of pixels counted can now be divided by the initial width measured by the program from the rectangular

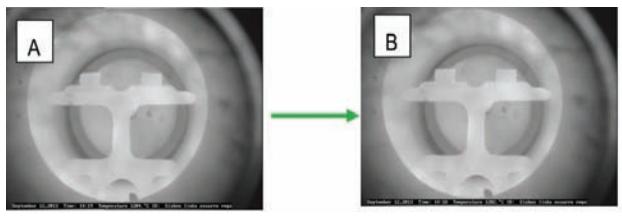

Figure 4—Typical Northern Cape ore (top left on stand) and West-African ore (top right on stand) iron ore at 1202°C (A) and 1204°C (B) in the ash fusion furnace

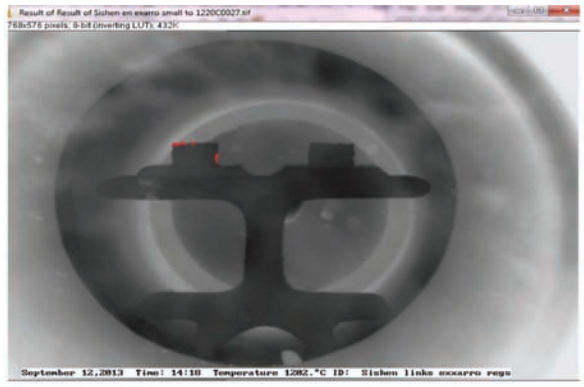

Figure 5—Difference between images A and B in Figure 4 (indicated by red) as measured by the macro written in Image J

selection to determine the height change during the temperature step. The data is displayed in a text file and can be imported directly into Excel® to construct a graph and to quantitatively determine the results. The greater the change in height for the sample, the more reactive the ore type.

Since the reactivity analysis is done by making use of the images as well as the written program, the samples removed from the furnace can be subjected either to a strength test or a porosity measurement, as no further dimensional measurements need to be taken.

Results and discussion

Eight samples of each of the three different ore types were tested by the two test methods. The average penetration depth measured for the penetration tests and average change in height for the length reducibility test were used to quantitatively compare and analyse the two test methods. The results are given in Table I

Standard test method and new test results

Table I shows that the trends of the results obtained for both test methods were similar. Both methods indicated that the West-African ore had the lowest reactivity, followed by the Northern Cape ore with medium reactivity, and then Marra Mamba with the highest reactivity. These results show that the new length reducibility test can therefore be used to obtain the same type of information as the standard penetration test used in industry.

Reproducibility of test methods

To determine the reproducibility of both methods, the coefficient of variation was plotted for each ore type. The coefficient of variation, Cv, is calculated by:

$$C_v = \frac{\sigma}{\mu} \qquad [1]$$

where σ is the standard deviation and μ is the mean. A lower coefficient of variation indicates a higher reproducibility and more precise results.

Figure 6 shows that the new length reducibility test has a consistently lower coefficient of variation than the standard penetration test, indicating that the new method has a higher reproducibility than the standard test, potentially leading to more precise results.

Additional results

Figure 7 indicates that the Marra Mamba and Northern Cape ores start reacting at about 1185°C, while the West-African ore starts reacting only above 1200°C. This information cannot be obtained from the standard test.

Advantages and disadvantages

In order to determine which test method would produce results faster and easier, the disadvantages and advantages of both methods are listed.

Standard test

Advantages:

- Since this test is carried out in the infrared furnace, a high heating rate can be used, thus simulating the temperature profile of the sintering process more closely
- The infrared furnace cools down, and the sample can be removed, within an hour

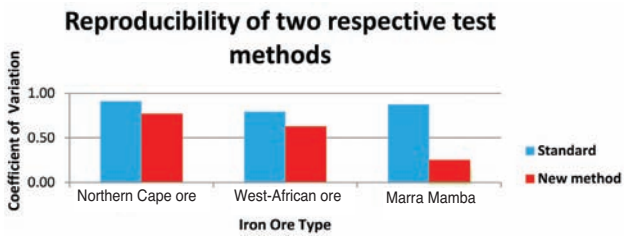

Figure 6—Coefficient of variation for each ore type tested using both methods

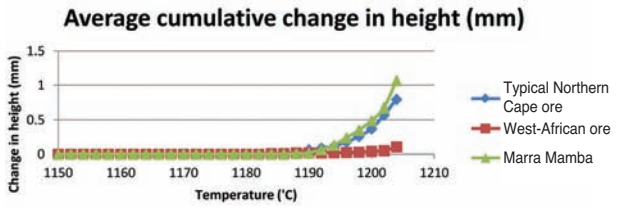

Figure 7—Average change in height as a function of temperature for the three ore types tested as determined by the length

Table I

Comparison of results

	Total change in height (New Method) (mm)			Total Penetration (Old Method) (mm)		
	Typical Northern Cape ore	West-African ore	Marra Mamba	Typical Northern Cape ore	West-African ore	Marra Mamba
1	1.44	0.04	1.55	0.36	0.22	0.10
2	0.23	0.22	0.74	0.64	0.66	0.59
3	1.41	0.16	1.21	0.62	0.22	1.82
4	0.56	0.05	1.27	1.38	0.29	0.49
5	1.82	0.02	0.83	0.48	0.48	1.24
6	1.52	0.15	1.02	0.48	0.69	3.64
7	0.79	0.19	0.86	1.21	1.74	1.87
8	0.20	0.02	1.07	2.69	0.27	4.49
Average	1.00	0.11	1.07	0.98	0.57	1.78
Standard deviation	0.63	0.08	0.27	0.78	0.51	1.56
σ/μ	0.63	0.77	0.25	0.80	0.89	0.88

- Since the sample has to be mounted and sectioned in order to determine the reactivity, a porosity measurement can easily be carried out.

Disadvantages:

- Only one sample can be tested at a time, making this a time-consuming method.
- All samples are mounted and vertically sectioned, and thus are not available for strength tests
- Working with the mould to produce the melt tablet requires great care since the 5 mm mould is extremely delicate and can resist hardly any pressure
- When placing the tablets in the tube of the infrared furnace, balancing the small melt tablet on top of the larger ore tablet is very difficult
- The depth of penetration is hard to measure. For some ore types a definite penetration can be seen as a uniform and darker phase penetrating into the granular phase of the ore, but this is not necessarily the case for all ore types
- The depth of penetration is measured by hand and the final value is often subjective.

New length reducibility test

Advantages:

- The ash fusion furnace allows testing of up to four samples per test run. The length reducibility test is therefore a more productive test
- The analysis of the reactivity is done automatically by a computer program and this removes operator subjectivity
- No vertical cross-section of the sample after the test is necessary, since the reactivity is a function of the change in height of the sample. The samples are thus available for either a strength test or a porosity measurement
- The sample consists of a single tablet that is easy to produce to the required weight and dimension specifications
- This method does not require a 5 mm diameter tablet made in a fragile mould.

Disadvantages:

- The maximum temperature for these three ores could not be set higher than 1204°C, since at 1206°C the Marra Mamba ore melted completely. The melt could leak into the furnace tube, causing it to crack
- The ash fusion furnace has a very slow heating rate and cannot replicate the actual heating rate in the sinter process.

Comparison of methods

Apart from its proven greater reproducibility and precision, the length reducibility test also provides the following advantages:

1. The time required for sample preparation is reduced, since only one tablet is required instead of two for each ore type and test run. Four samples can be analysed simultaneously during a single 4-hour run, including cooling time, compared with two hours for a single sample sample by the standard method. The new test method also requires only one cylindrical tablet which is simple to reproduce. There is no need to produce two tablets, each with a different mixture of raw materials
2. Results are obtained automatically by making use of the images taken with the digital camera and the image analysis program. The results are therefore obtained easily and the error associated with human judgement is avoided.

Future work and recommendations

In order to obtain international accreditation for this new method, the following aspects are of particular importance according to ISO 17025:

- Reproducibility/repeatability
- Discrimination of samples
- Sensitivity
- Detection threshold
- Comparison against existing methods
- Inter-laboratory tests.

Conclusion

In a comparison of the standard test with the newly proven length reducibility test it was found that:

1. The new length reducibility test yields more reproducible results
2. The results obtained by the new test method are more precise
3. The new length reducibility test is less time-consuming and easy to implement
4. The new test can therefore be used with confidence to evaluate and quantify the reactivity of iron ore in terms of lime assimilation in the sintering process.

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