ON-LINE ANALYSIS FOR PROCESS MONITORING AND CONTROL AND AT IRON ORE CONCENTRATORS[1]

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Abstract

This paper presents the art of on-stream particle size and elemental analysis at iron ore concentrators, followed by the results and experiences from some sites. On-line particle size analysis is rather common at medium to large sized concentrators. On-line XRF elemental analysis, however, a routine practice in base metal concentrators, is only used in some iron ore concentrators. This analysis is applicable for process monitoring and control down to the 4-5% silica level by measuring Fe assays accurately. With lower silica content, direct measurement of silica and other light elements is required. Some light element analyzers based on prompt gamma neutron activation (PGNAA) method have been developed, but they have not performed up to expectations. Other methods have been tried but none has been used beyond prototype installations. Operating on-line light element slurry analyzer installations are very rare in iron ore applications. There is an economic incentive to mine lower and variable grade iron ore resources, which require concentration and removal of several impurities to produce high quality concentrates for pellet production. Monitoring such operations by on-stream analysis has significant economic benefits and there is a need to develop a practical slurry on-line analyzer for Si, Al, Mg and other light elements. The latest developments in slurry on-stream light element analysis are described.

Keywords: On-line slurry analysis; Particle size measurement; XRF analysis; Light element.

1 INTRODUCTION

Carefully collected and prepared samples analyzed in laboratories are the reference for process monitoring, control, optimization and material balancing. The relatively high cost per assay and manpower requirements limit the frequency of such assaying to bare necessity. Shift composite sample assays describe the past behavior of the plant with some time delay, but are not good for reacting to changes in the feed, alarming for equipment failures and process optimization.

An on-line analyser offers the following advantages:

1. Required manpower and cost per assay is radically cheaper than in a laboratory
2. Low cost of assays allows frequent assaying and reveals upsets and variability in the process operation
3. Frequent assays give fast feedback and plenty of data for process analysis, development and optimisation

    Automatic sampling (and sample preparation in some cases) is repeatable and less subject to human individual practices, errors and variances

4. Delay from sample extraction to the moment when the assay is available for operators and control systems is only some minutes, whereas laboratory results take many hours.
5. Closed loop and expert control based on assays is possible.

Laboratory automation and robot laboratories have reduced both the cost of assaying and assaying time delays, however, they are still inferior to on-line analyzers. The main role for on-line analysis is to produce frequent assays for process control. Laboratory analysers are capabile of measuring a wide range of elements and low concentrations for quality control and mass balancing. Due to limitations in taking and measuring coarse samples and lower analytical sensitivity, these tasks are not usually carried out by on-line analyzers.

2 ON-LINE PARTICLE SIZE ANALYSIS

A typical hematite mineral processing plant for high grade iron ore pellet material has a primary/secondary grinding circuit followed by a reverse flotation or other process to remove gangue minerals and impurities. In many cases the concentrate is re-ground and possibly upgraded again before thickening and filtering for pellet plant feed (Figure 1).

2.1 Primary/Secondary Grinding

Particle size analysis after primary/secondary grinding is used to monitor and control particle size at the secondary ball mill cyclone overflow. Variations in ore hardness change the particle size distribution and influence the liberation of minerals and the production of fines. The flotation reagent feed requirements are changed by varying particle size as the total surface area of particles in the slurry can change radically. Usually the average t/h feed to the grinding circuit can be increased by on-line particle size measurement and control by 5 – 10%. This has a positive effect on the cash flow from the operation. The most important particle size measure at this stage is D_{80}. In some cases fines measurement is necessary for slimes control. Slimes removing cyclones can be monitored by D_{80} size in the overflow.
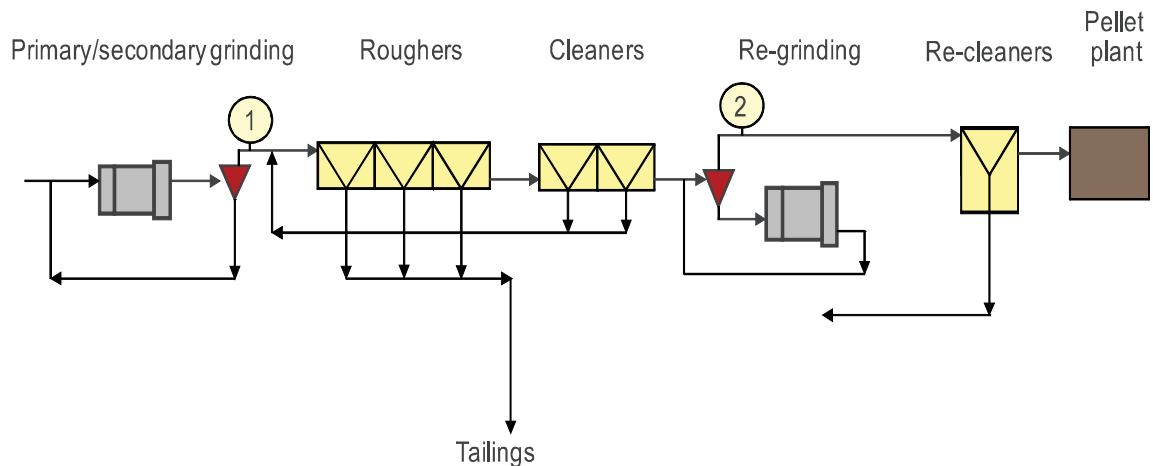
Figure 1. Particle size sampling points.

2.2 Regrinding

The particle size distribution of the regrinding product has a significant influence on the quality of the final product and requires full size distribution analysis. On-line measurement and control of the specific surface area also makes it easier for filters to produce cake with the stable moisture and size distribution required to have good yield from the green pellet balling circuit (Figure 2). An on-line particle size analyzer based on laser scatter technology is required for this measurement (Figure 3).
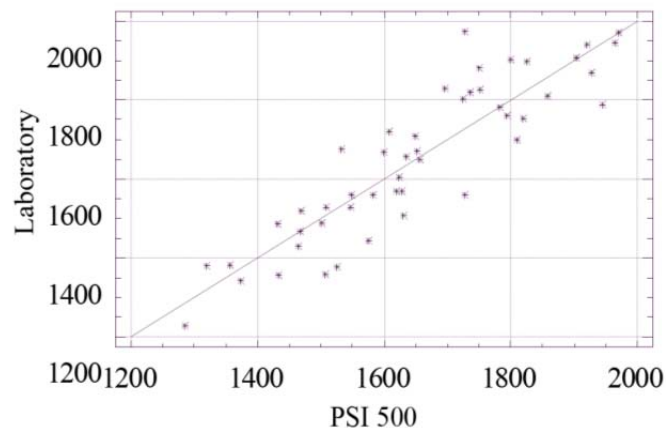
Figure 2. Comparison of on-line specific surface area measurement with manual laboratory measurement (units of cm^2/g). Laboratory measurement repeatability is about +/-100 cm^2/g

Magnetite minerals particles form agglomerates, and these bias the on-line measurement results to the coarse side. With a typical concentrate for Fe pellet production, the effect on the − 44 µm fraction is 5-10% relative as compared with carefully made laboratory measurements. This effect is eliminated by de-magnetizing the sample and subjecting it to ultrasound dispersion before measurement.

Figure 3 Laser scatter on-line particle size analyzer and XRF on-line slurry analyzer.

3 ON-LINE FE ANALYSIS

Iron content in slurry can be measured with good accuracy directly from the slurry by an on-line XRF analyzer from all process streams. An absolute error of 0.5% has been achieved in some installations (Figure 4). The on-line measured Fe concentration often has a good correlation (R^2 well over 0.9) with SiO_2 (insoluble) content above 3-5% SiO_2 concentrations for process control purposes. Mineralogy variations have an effect on the achievable accuracy.
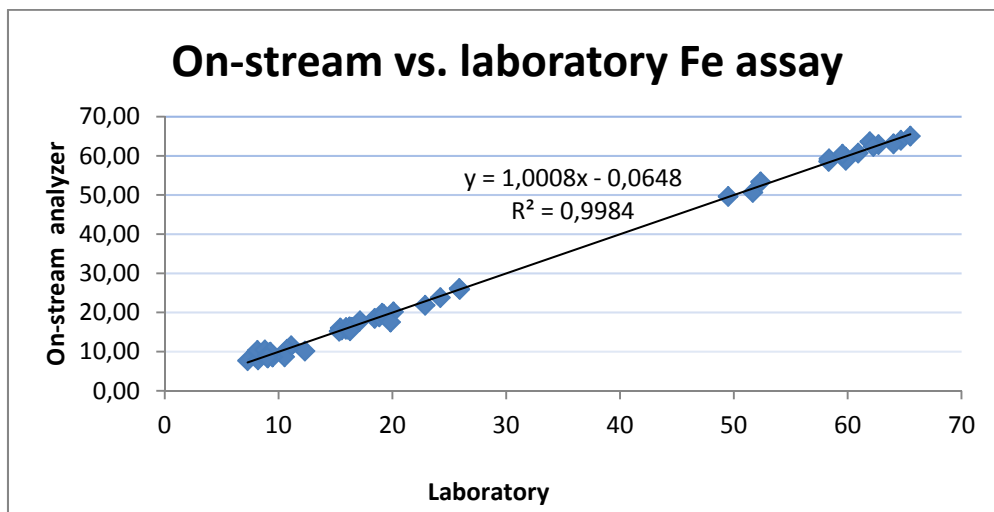
Figure 4 Correlation between on-stream XRF analyzer and laboratory iron measurements for hematite concentrator tailings and concentrate samples. Out of 50 samples, 3 outliers were rejected.

Typical sampling points are indicated in Figure 5.
1. **Feed**
   Fe/silica content in the feed can be used for feed-forward control of silica removal by flotation. The disturbance caused by ore variability has to be reduced at later processing steps.
2. **Rougher concentrate**
   Rougher concentrate silica (insoluble) content can be measured indirectly by Fe measurement. Rougher area control has the most significant role of reducing the assay variability resulting from ore changes.
3. **Primary cleaner concentrate**

Cleaner concentrate silica content can be measured indirectly by Fe measurement. The primary cleaner tailings can also be measured to assess the circulating load in the cleaner circuit.

4. **Tailings**
   Fe assay is used to maximize recovery without sacrificing product quality. With today's Fe concentrate prices and lower ore grades, recovery has a significant effect on cash flows.

5. **Final concentrate**
   For 1 – 2% silica content, accurate indirect measurement of gangue minerals is not possible based on Fe measurement. From a process control point of view, the primary cleaner concentrate grade has a significant effect on the final concentrate grade.

Very similar sampling points can be used for magnetic separation circuits (Figure 6).
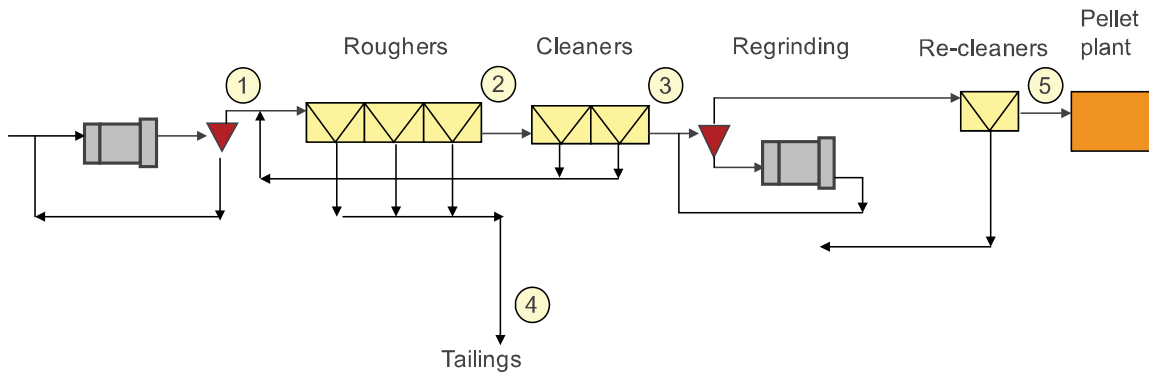
Figure 5 Typical hematite reverse flotation assay sampling points.
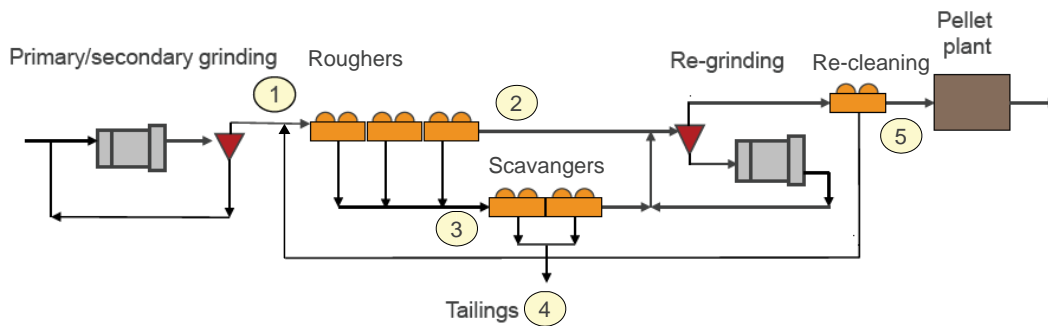
Figure 6 - Typical sampling points in a magnetic separation circuit.

4 DIRECT ON-LINE SLURRY LIGHT ELEMENT ANALYSIS

On-line XRF can in specific cases provide information of the light element content of the intermediate products by indirect analysis based on measurement of the heavier elements. However, where the correlation of the element of interest with the directly measured element is poor, such inference does not produce the required accuracy, in particular the robustness of calibration. In some cases the composition of gangue minerals may be necessary information for process operators. Technologies for direct on-line slurry analysis of light elements exist, however, the application has been limited due to the challenging slurry properties.

4.1 Robot Laboratory

Process sample can be re-sampled, ground to a finer particle size, dried, formed into a briquette and fed to a laboratory XRF analyzer by a robotic system. Sample size reduction and drying enables the measurement of many light elements, for example silica. The limitations of robot laboratories are low feasible sampling frequency and relatively high maintenance requirements.

4.2 Prompt Gamma Neutron Activation Analysis (PGNAA)

PGNAA has been known as an on-line analytical technique for decades, and it has had a number of successes mostly in cement and bulk minerals analysis.[1] The limiting factor of the measurement precision is the weak interaction of neutrons with the sample and consequently the low frequency of the PGNAA events. The resulting large statistical variance of the measured gamma-ray intensity can be smoothed out only by very long measurement times. In the literature, minimum detection limits of 0.3-0.5% SiO_2 have been quoted for bulk solids with 10 min analysis time. In slurries, an additional dilution factor increasing the MDL (Minimum Detection Limit) needs to be taken into account. In iron ore slurry applications, another challenge arises from the fact that the analysis requires a large amount of heavy solids to be maintained in representative suspension in the sensitive analysis volume.

4.3 Laser Induced Breakdown Spectroscopy (LIBS)

This technology is discussed in detail below.

5 LIBS MEASUREMENT TECHNOLOGY

Laser Induced Breakdown Spectroscopy is an optical emission analysis technique originally developed in the 1960s. After the fast development of pulsed lasers and optical detection technology over the past decades, it has found real applications as an analysis method. As compared to other optical emission techniques, it has important advantages, especially in on-line analysis.

The LIBS measurement principle is presented in Figure 7. The arrangement includes a pulsed laser source, optics that focuses the laser beam into a small spot on the sample surface, and collection optics and a spectrometer to record the emitted light from the sample. The laser generates short (~10 ns) pulses that, when focused on the sample surface, create a hot plasma spark around the focal point. The laser pulse energy, of the order of 10-200 mJ, is sufficient to atomize and ionize a small amount of sample material. After the pulse, the atoms return to their ground state and emit optical light within a few microseconds. The atomic state transitions of the sample atoms are shown as emission lines at characteristic wavelengths in the emission spectrum. Figure 8 shows an example spectrum from an iron ore slurry sample. The intensity of the characteristic lines depends on the atomic concentration in the sample material. The analysis is usually calculated using a linear calibration model of intensities and/or intensity ratios of various element lines. The measurement is typically averaged over several pulses to compensate for pulse energy variations and limited representativeness.
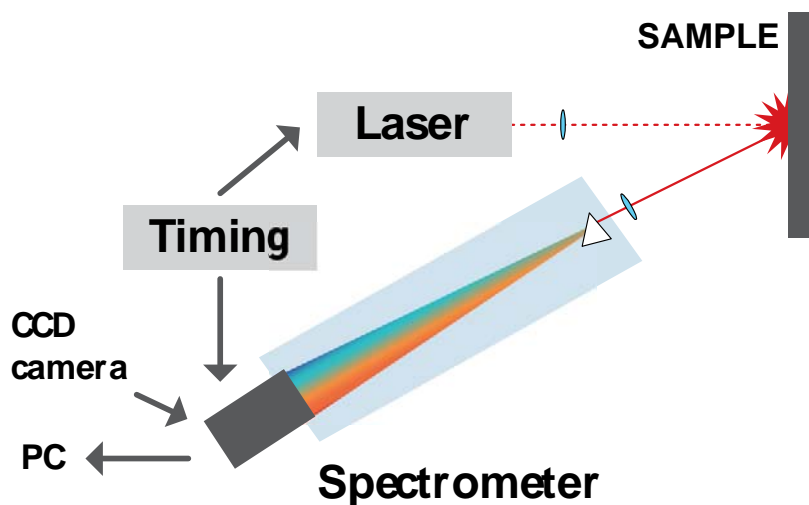
Figure 7 LIBS analysis principle.

The analytical performance is dependent on the element of interest, the sample matrix and the measurement setup. In principle, the whole periodic table of elements can be measured using LIBS, however, some of the elements require special arrangements. Bibliographic data on LIBS analysis reports for various applications are given in the references by Cremers and Radziemski.[2]The obvious advantage of LIBS analysis over other optical emission techniques (eg, ICP and spark-OES) is the simplicity of sample preparation. Gaseous, liquid and solid sample analyses are all possible. Although a single laser pulse shot looks at a small area of the sample surface, the representativeness is improved by repeated pulses. A typical repeat frequency of the laser pulses is 1-100 Hz, which makes it possible to do several hundred measurements within minutes. Low concentrations, down to 1 ppm in homogenous samples, can be detected. If needed, the optical components can be located far from the sample and the signal can be transmitted through air or optical cables. The most important limitation in applying LIBS analysis is that, even though it is a direct elemental method, the measured intensities are to some extent dependent on the sample matrix, including its chemical composition, mineralogy, physical form and so on. Since the measured spectrum contains a lot of information, chemometric methods have been used with success to compensate for these effects.

Because of the simplicity of the measurement setup, LIBS is quite amenable to on-line elemental analysis. A number of industrial on-line applications have been reported by Noll et al.,[3] Gaft et al.[4] and Sabsabi.[5]
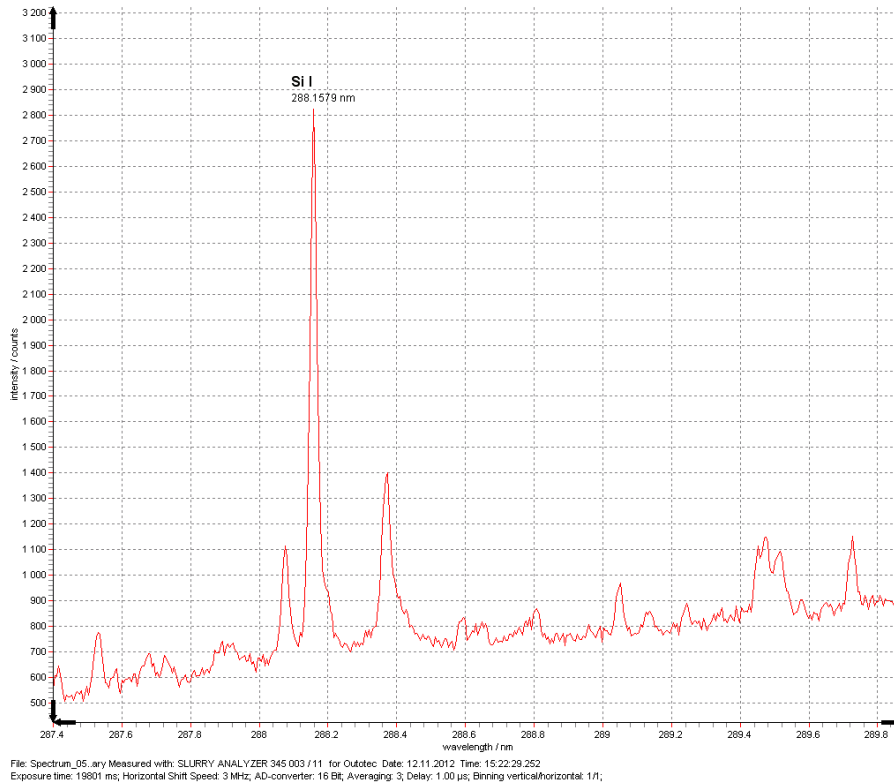
Figure 8 Typical Si peak in a high resolution LIBS spectrum for an iron ore sample.

6 ON-LINE LIBS ANALYSIS FOR MINERAL SLURRIES

When the LIBS analysis technique is applied to mineral slurry samples, a number of challenges are faced due to the heterogeneous nature of the sample. As water tends to absorb the laser energy, more energetic pulses are needed as compared to solid samples. A large number of repeat pulses are required to integrate over a large number of particles involved in the plasma. As the analysis can be done only on a free surface of the slurry, stabilization of the position of the surface is important. Special care must also be taken to ensure that the particles that are present on the surface represent the concentrations and particle sizes in the bulk flow. Compensation is required for changes in the solids content of the process stream.

Several aspects of LIBS analysis of slurries are covered by earlier publications associated with the research work at COREM, Quebéc by Barrette et al.,[6] Michaud et al.[7] and Michaud, Leclerc ad Proulx.[8] The research includes a lot of feasibility studies over various applications, and development of presentation concepts. Finally, an industrial prototype was installed at the Quebéc Cartier Mine iron ore concentrator plant as reported by Michaud and Chevalier.[9] The system is set up for the measurement of C, Mg, Si, and Ca contents in a pelletizing feed stream with very encouraging results. Even though it is considered as an alpha test prototype, the analyzer has been shown to produce good sensitivity and accuracy over long periods of time. To stabilize the measurement, the solids content of the primary sample stream is kept constant using a fixed volume scale and controlled dilution water addition.
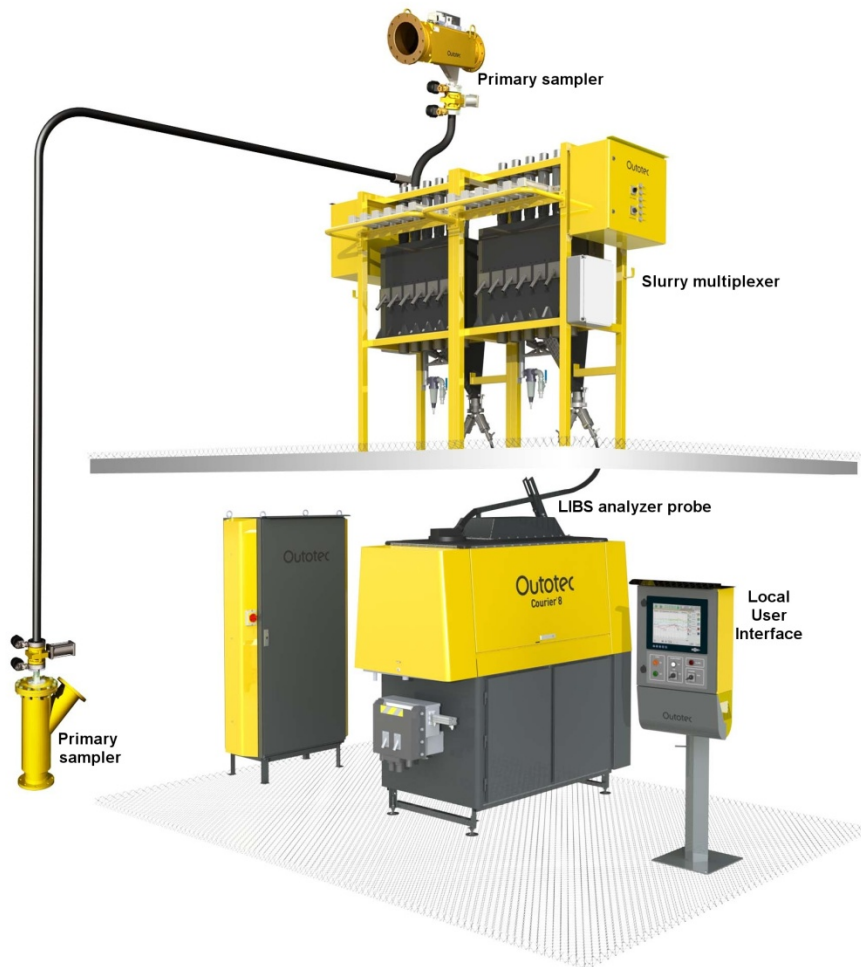
Figure 9 On-line LIBS analyzer system.

Samples from up to 12 primary samplers are sent to a multiplexer (Figure 9). It selects one sample at a time to pass through the analyzer probe flow cell. The sample is screened to remove trash and the sample flow is stabilized by a level controlled tank before measurement. A local user interface displays the assays as well as the sampling system and analyzer status. Assays and analyzer status information is sent to the plant control room and are available through the analyzer network to plant metallurgists, laboratory and maintenance teams.

The system has been tested in various applications to determine the optimal design parameters for both the measurement technology and sample presentation. Figure 10 shows an example spectrum from an iron ore concentrate with ~1% SiO_2 concentration.
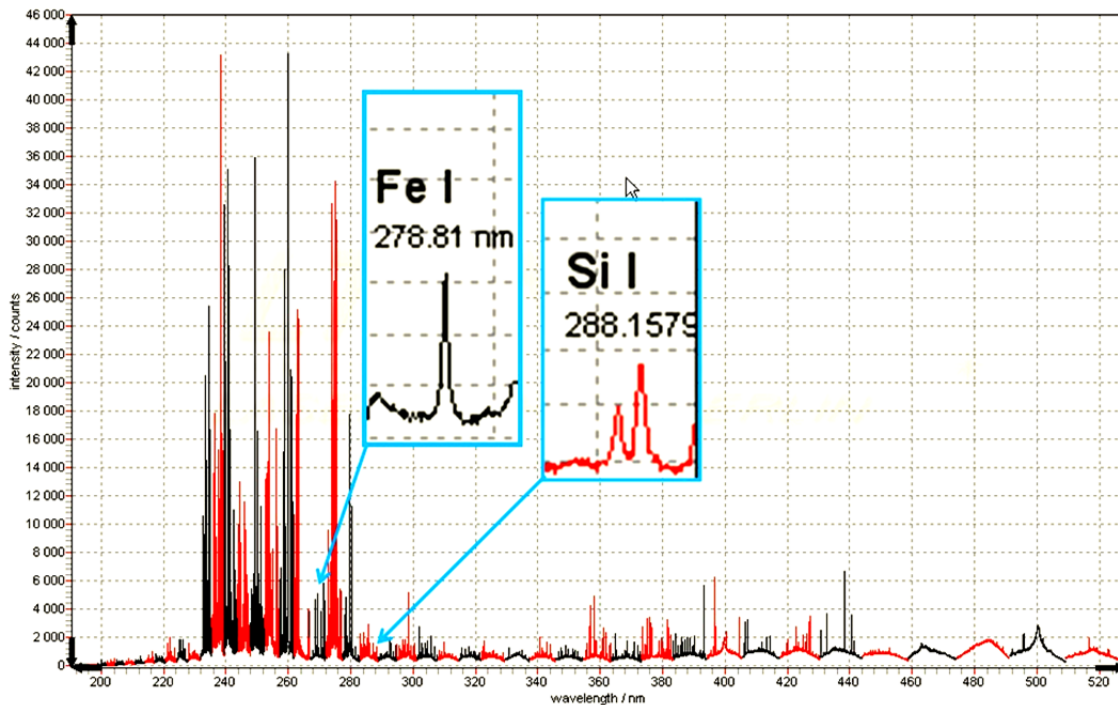
Figure 10 On-line LIBS spectrum from an iron ore concentrate slurry.

7 FURTHER IRON ORE PROCESSING APPLICATIONS

Typical elements of interest in iron ore concentrates are Si, Al and in some cases S and P. Tests of samples from several concentrators have been performed. Typical accuracies of measurements are listed in Table 1.

Some additives have to be added to iron ore concentrates before green pellets are made. Typical additives are dolomite, olivine, bentonite, coke and lime. In order to reach target pellet quality, the composition of the iron ore concentrate as well as the iron ore concentrate with additives is valuable information. Using on-line LIBS, it is possible to measure on-line the Si, Ca, Al, S and C contents of the solids at their typical concentrations. As additives can be added before and after filtering, filter cake would have to be sampled and mixed with some water before measurement by the on-line slurry analyzer. Tests with a laboratory closed loop sample circulation analyzer indicate that the accuracies in Table 2 can be achieved. Figures 11 and 12 show example calibrations for SiO_2 and CaO.

Table 1 Observed performance for iron ore concentrate measurement

	Range	Measurement error (absolute)	Precision (repeatability) absolute
SiO_2	0.8-3.2%	0.1%	0.01%
Al_2O_3	0.3-0.5%	0.02%	0.01%

Table 2 Some iron ore pellet plant feed sample analysis results

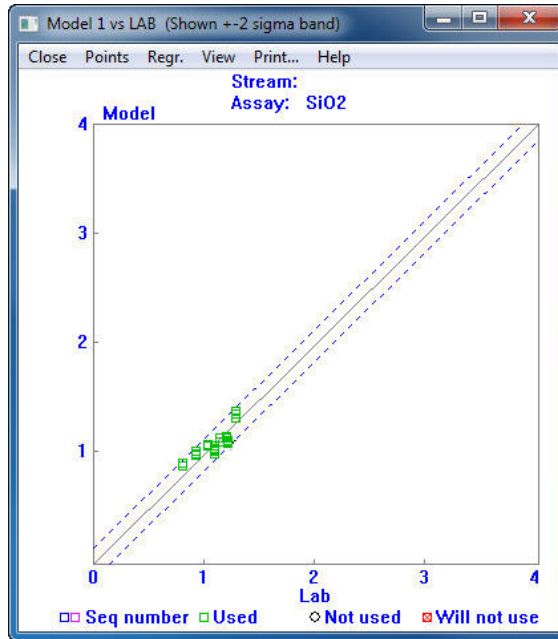| Element	Measurement error (absolute)
Si	0.1%
Ca	0.1%
Mg	0.1%
Al	< 0.1%

Figure 11 - Example of SiO$_2$ calibration for iron ore concentrate samples.
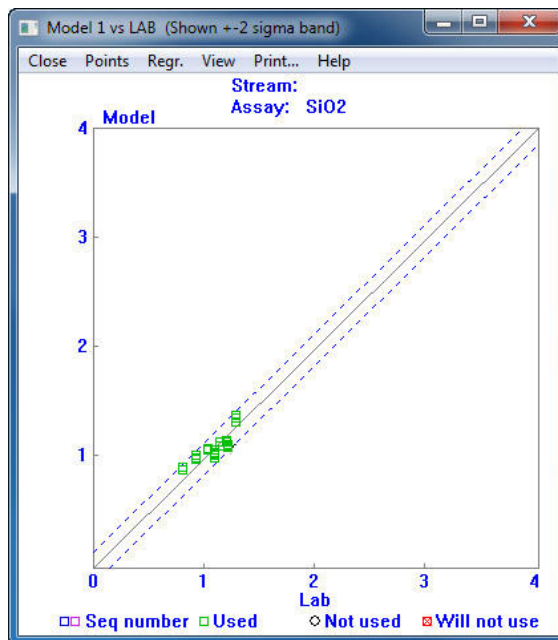

Figure 12 - Example of CaO calibration for iron ore concentrate.

7 CONCLUSION

Dependable measurement information is the key to successful control applications. On-line analyzers offer new possibilities for process monitoring and control in the mineral processing industry.

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