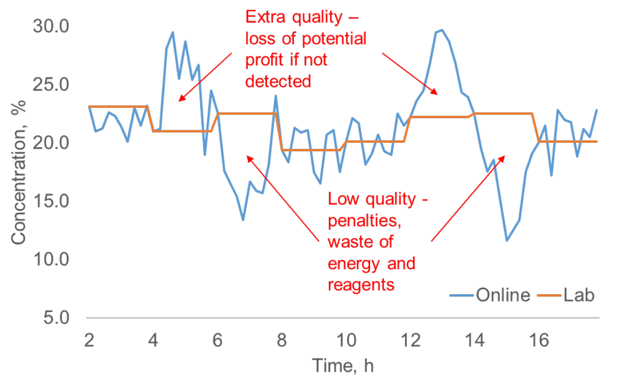
MOVING ONLINE

Alexander Baryshnikov and Mindaugas Dailide, Lyncis, Lithuania, outline safe and cost-effective techniques for online elemental analysis in fertilizer production.

The chemical composition of fertilizer ores differ from mine to mine and even from different parts of the same mine. Concentration variations of valuable elements and impurities like Mg, Si, Fe, Na, Ca, and heavy metals increase processing expenses and degrade the quality of the final product. Using laboratory analysis for continuous process control is not possible as it takes several hours or days from sample collection until the results are received. When the results arrive it is already too late to adjust process parameters. If extra quality goes undetected, the company

loses the additional profit it could get for selling higher-grade ore or products. If low grade material goes undetected, the company not only faces penalties from the customers but also wastes energy and reagents on processing off-grade material. Only online analysers operating in continuous mode and providing chemical composition results in real time can allow timely process adjustments (Figure 1). Only online analysers provide a full picture of concentration variation change over time and allow timely process adjustment.



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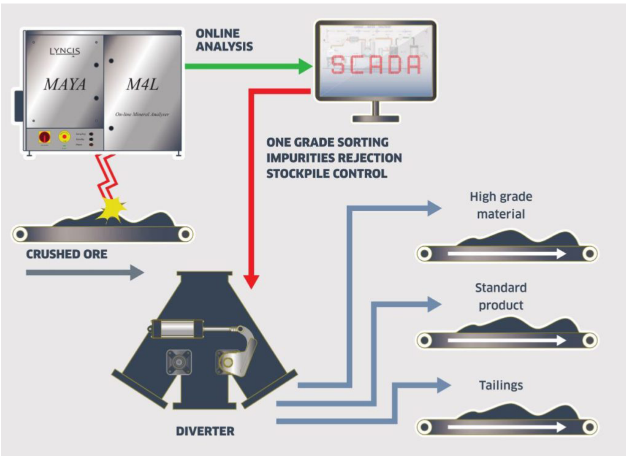

Figure 2. Automated grade sorting/impurities rejection.

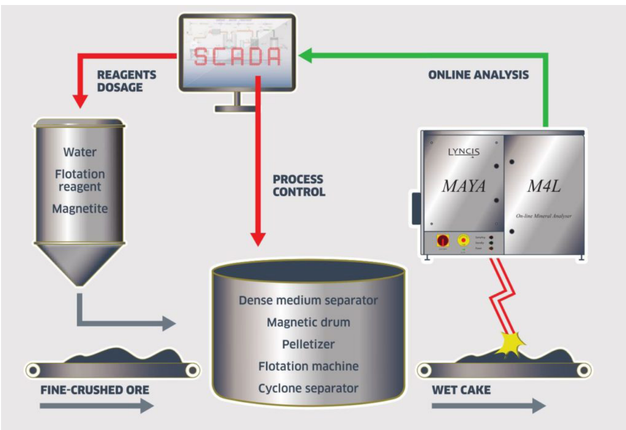

Figure 3. Automated process control – reagent dosage, adjustment of process parameters like heat, etc.

Why LIBS?

Historically, prompt gamma neutron activation analysis (PGNAA), pulsed fast thermal neutron activation (PFTNA), and X-Ray fluorescence (XRF) analysers were used for online analysis. However, these techniques suffer from harmful radiation, high maintenance expenses, and some limitations in analytical capability.

Laser-induced breakdown spectroscopy (LIBS) is emerging as a safe and radiation-free technique. In addition, the analytical performance is excellent and maintenance expenses are very low.

Industrial LIBS

Modern LIBS analysers use a pulsed laser which is focused on the material directly on the conveyor or onto a slurry stream. Laser evaporates a small portion of the material and creates plasma. Plasma is a mix of neutral and charged atomic and molecular particles that lose excess of acquired energy in the form of light. Plasma emission light is collected and analysed with spectrometers and software to yield LIBS spectra. Each element has characteristic fingerprint lines, the intensity of which is proportional to the concentration of elements in the sample. Laser pulse frequency is adjustable in the range of 3 – 100 pulses per second allowing one to collect and average hundreds of spectra in a matter of seconds or minutes. Chemical composition is sent to plant control systems for automated process adjustment. Modern industrial LIBS analysers are built to withstand harsh industrial environments with the presence of dust, corrosive materials, vibrations, cold and heat, and can operate continuously 24/7 for decades. Very low-cost maintenance requires mostly periodical changing of air-cleaning filter inserts and cleaning of the optical window. LIBS is suitable for analysis of any element in any material including solids, slurries and liquid solutions with a high level of confidence and without sample preparation.

Online LIBS applications

Since the first installation of a LIBS online analyser for phosphate ore analysis in Florida in 2008, Lyncis has gathered a lot of experience in the analysis of phosphate, potash, NPK, and polyhalite fertilizers. All online analyser applications can be grouped into sorting and automated process control (Figures 2 and 3). The analyser has been used for online analysis of raw materials, intermediates, and final fertilizer products.

Phosphate ore impurities rejection

A LIBS online analyser was installed on a conveyor belt for real-time analysis of apatite ore which contained quartzite and clay impurities (Figure 4). After an initial study of P_2O_5, MgO, Fe, Al, bone phosphate lime (BPL), insoluble phase and metal impurity ratio (MER) in real-time, 2% of MgO was selected as a decision point for an off-grade ore. Online analyser data was used to reject ore exceeding 2% of MgO which resulted in payback of the instrument in around two months due to avoided penalties for off-grade product shipments and higher prices of high-grade products.

Two-laser double-pulse system for low concentrations

A special high-sensitivity two-laser double-pulse system was used for the analysis of low-concentration phosphate ores.

Figure 4. LIBS online analyser installed on a conveyor belt.

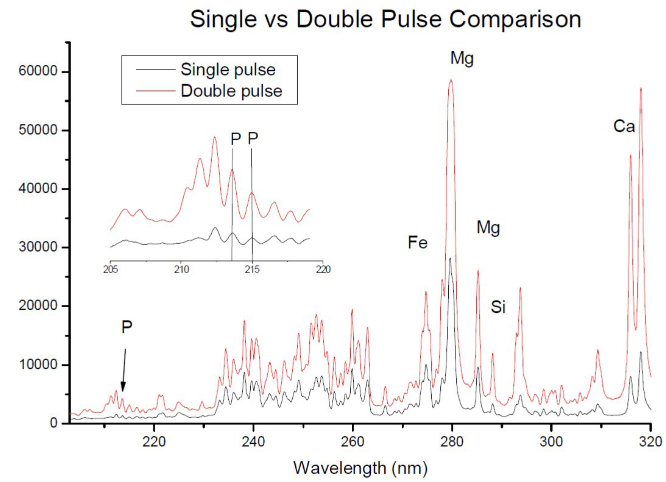

Figure 5. The double-pulse system allows an increase in the intensity of signals by up to eight times.

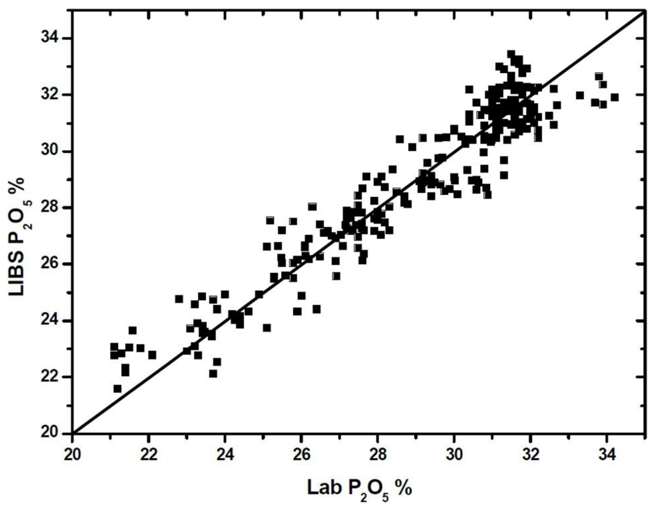

Figure 6. Longterm laboratory and online LIBS analyser result correlation.

Apatite ore contaminated with magnetite, baddeleyite, and other minerals contained only 3 – 7% of P_2O_5. A special LIBS double-pulse technique using two synchronised lasers allowed for an increase in the intensity of element emission signals up to eight times (Figure 5). The double-pulse system can be useful for analysis of other elements requiring high sensitivity as well.

Slurry analysis and flotation reagents dosage

For flotation reagents dosage in phosphate slurry beneficiation, a double-pulse system was used to increase the sensitivity. Analysis of 256 samples collected over half a year showed good accuracy and correlation with laboratory analysis: R^2 = 0.91, average absolute error +/-0.75 % (Figure 6).

Automated water dosage in potash production

In potash fertilizers, NaCl comes as the main impurity. It can be washed away with water, but water dosage should be controlled carefully as valuable material KCl is also soluble in water. To address this critical task, a LIBS online analyser was installed on a conveyor belt of concentrate cake and connected to an automated water dosage unit. This allowed the concentration of NaCl to be maintained at a precisely desired level specific to each product grade (95%, 98%, 99% of KCl) automatically and continuously providing economic benefits by stopping the loss of valuable KCl and eliminating quality claims.

Polyhalite ore analysis

Polyhalite is a unique multi-nutrient mineral providing K_2O, MgO, CaO and SO_3 nutrients to ensure an increased crop growth. LIBS technology proved to be very useful for stockpile homogenisation, mining grade control and impurity rejection. All elements of interest and impurities yield intensive sets of peaks in LIBS spectra, allowing for reliable and accurate measurement. Control of the concentration of nutrients and impurities at the early stages of fertilizer production prevents wastage of reagents, water and energy for processing of the low-grade ore.

Potash fertilizer quality control at seaport

Quality control of fertilizers' final product is required at various stages starting from the warehouse of the production plant for grade sorting to the final quality check before loading to transportation vessels. Lyncis is currently in the supply process of an online analyser to be installed on a conveyor in Vancouver seaport for the final quality check of potash fertilizers before loading to ships. Lyncis online analysers are designed to resist corrosive, dusty, humid environments and are suitable for installation in seaports. As demonstrated in other cases, the analyser is expected to eliminate quality claims.

Conclusion

LIBS online analyser technology has matured and has proven itself for wide spectra of fertilizer analysis. It has been used at all production stages from ores, to intermediate materials and final products. Safety, low maintenance expenses, and excellent analytical capabilities make it advantageous against traditional techniques. WF