

Real-time Elemental Analysis for Prompt Process Control in Alumina Production

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Abstract

It is necessary to know the real-time chemical composition of raw materials to provide optimal process control and high and consistent quality final product for the efficient mining, beneficiation and processing of bauxite ores. With real-time data, it is possible to manage variations in ore grade, reject unacceptable material, blend different grades of bauxites, make real-time adjustments to the addition of lime, sodium hydroxide and other process raw materials and reagents, and to provide some measure of product quality control. Lyncis offers laser analysers to detect all elements of interest in real-time, directly on site, without sampling, on conveyor belts and in slurry pipelines, eliminating the need for continuous laboratory analysis, often involving hours of delay, and potential human error. These analysers based on LIBS (Laser Induced Breakdown Spectroscopy) do not produce any ionizing radiation, and have operated 24/7/365 in automatic mode under demanding industrial conditions for many years in metals and fertilizer applications. To ensure stable accurate measurements in real time, chemometrics and optimization approaches are used. Traditional calibration methods give unsatisfactory results for real operations, where many other factors such as weather conditions, grain size variation, moisture, etc., influence results. A range of comprehensive spectra filtration and normalization techniques were studied and implemented in our specialized software. It can be easily used by customers and allows fast building of stable and accurate calibration algorithms for every application.

Keywords: LIBS, online analysis, bauxite, alumina, analyser.

1. Introduction

Variations in mineralogy and the presence of impurities such as silica, iron oxides and titania in bauxite (the main raw material used in the alumina industry), not only influence its processing efficiency, but can also increase production costs and create quality deficits in the final product. With real-time data, it is possible to manage variations in ore grade, reject unacceptable material, blend different grades of bauxites, make real-time adjustments to the addition of lime, sodium hydroxide and other process raw materials and reagents, and to provide some improved measure of product quality control.

Growth in the global alumina supply and the tendency to process lower grades of bauxite in future will only intensify the demand for reliable tools for real-time measurements. These measurements can provide the data required to manage impurities, and to make prompt corrections to process parameters without the potential delays of up to several hours for sampling and laboratory based results.

Raw materials in different industries often come for processing with significant variations in chemical and mineralogical composition. These variations are manifest after their extraction from open cast or underground mining, and after they are delivered to and recovered from production stockpiles and raw material transport and transfer systems. Such variations decrease the stability and efficiency of any subsequent beneficiation or processing steps, potentially deteriorate the quality of final products, and decrease production efficiency in general. It is therefore highly profitable to understand any real-time variation in the quality of the feed to processes where settings must assume receiving raw materials with stable pre-set quality parameters.

It is possible to solve the issues caused by quality variation by sorting raw materials by grade, including the rejection of the material unsuitable for specific applications, and by well-informed adjustment of processing parameters based on the real-time measurement of the chemical composition of the raw material. With this information, adjustments can be applied at all production stages, from minerals survey, mining, beneficiation and up to the preparation of feed grades, with more accurate pre-set composition and prompt automatic adjustment of the processing parameters.

In most cases, the information on chemical composition of raw materials on conveyers required for the process control after mining, crushing and blending, for averaging of stockpiles and batching of mixture components, only becomes available to process operators some hours after sampling. For large-tonnage production, such information delays significantly influence the efficiency of the process control. Besides, the precision of the information received is not always high enough, due to the complex procedures required to ensure the representativeness of the samples and their preparation for laboratory analysis.

2. Application of Online Measurement Tools in Alumina Production

Many alumina refineries experience efficiency losses due to the lack of uniform quality bauxite. Bauxite consists of different minerals with silica, iron oxide and titania as the major impurities. The proportion of the minerals varies depending on the bauxite source, and variations in production grades from the same source.

Impurities contained in bauxite are one of the major causes of the inefficiency in the Bayer process. The caustic soda component of Bayer liquor is a critical raw material, being a significant part of total operating costs in an alumina refinery. Its consumption largely depends on the composition of the bauxite processed, and the chemistry of the desilication product produced in the desilication and digestion steps. The concentration of silica in the final alumina product is also strictly controlled due to its negative influence on the aluminium metal product.

With online measurement tools (Figure 1) to provide the chemical composition of materials, production controllers receive the most efficient information for accurate process control in real-time. Such tools make possible timely changes to the Bayer process chemistry, manage impurities in the Bayer circuit (in bauxite ore or in liquor), control caustic soda consumption in desilication, scale formation, and quality of the final product. All this improves cost and energy efficiency, increases refinery output and reduces the variations in alumina quality.

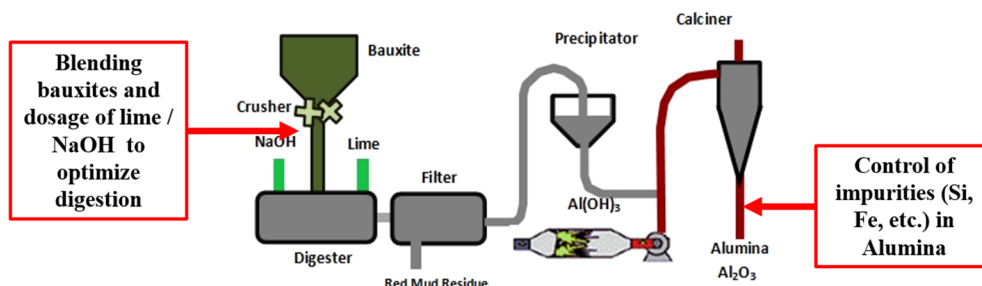


Figure 1. Possible points for online measurement.

Tools that can provide real-time information on chemical composition of the materials directly on conveyor belts or in slurry pipelines without sampling improve the efficiency of the operation and increase the correspondence of analytical information to real composition of the material flow [1 - 4]. However, most analysers on the market are based on different sources of ionizing radiation (Roentgen, neutron or gamma), presenting risks to operating personnel and the environment. Such analysers usually require special certification from regulatory bodies dealing with radiation monitoring, and are often complicated pieces of technology.

3. Industrial LIBS Analyser

To provide this capability, Lyncis offers laser analysers to detect all elements of interest in real-time, without sampling, directly on site, on conveyor belts and in slurry pipelines, eliminating the need for continuous laboratory analysis which often involves hours of delay from the sampling time, and subject to potential human errors. These analysers are based on LIBS (Laser Induced Breakdown Spectroscopy) and do not produce any ionizing radiation. The wide experience accumulated in the manufacture and servicing of the MAYA online LIBS analysers developed by Laser Distance Spectrometry, is successfully used by the Lyncis team to bring the MAYA analyser to a qualitatively new level. This provides customers with a new level of technical service, and offers advanced analytical tools for greater customer independence in every day operation.

The operating principle of the MAYA analyser is laser optic-emission spectrometry (LIBS), where the solid-state pulsed Nd:YAG laser serves as an excitation source for the material studied. With a measuring rate from 1 to 20 times per second, the laser beam is focused on the surface of the material being analysed, where temperatures of more than 30,000°C is achieved instantly, and a small sample of the substance is excited into a plasma state. The plasma is practically electrically neutral, but heavily ionized gas containing the molecules, atoms and ions of the chemical elements of the material being measured, in addition to free electrons. The processes of recombination of positively and negatively charged particles begin in the plasma immediately and are accompanied by emission of photons, producing the plasma “glow”. The atoms of every chemical element emit photons with strictly defined wavelengths, and the emission intensity is proportional to the concentration of this element in the material analysed. The spectra measured is characteristic of the material analysed, figuratively speaking their “fingerprints” [5-7].

The optical radiation of the plasma within wide optical range from 190 to 1000 nm is measured with special spectrometers before transferring the digital information of the spectral composition to the computer in the analyser (Figure 2).

As a result of the statistical processing of a set of spectra for the period of time required to resolve the production measurement (usually from 20s to several minutes), the concentrations of the elemental components of the material analysed are calculated [8].

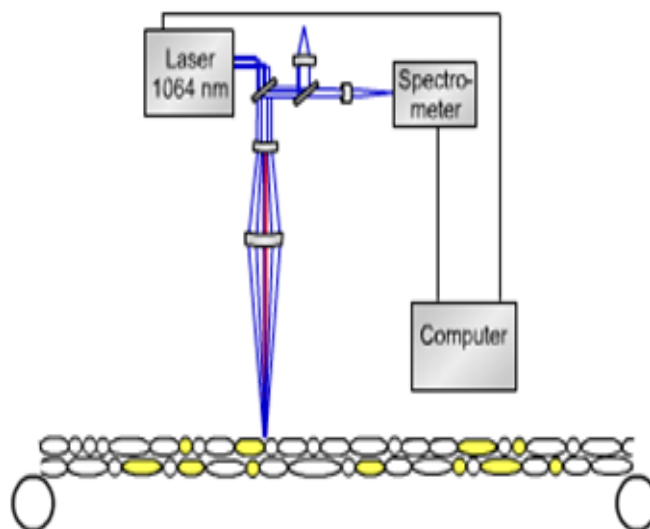


Figure 2. Principal scheme of a LIBS system.

The main advantages of the LIBS systems compared to the other analytical methods are the following:

- high accuracy, low detection limits and high sensitivity due to clear spectral lines of most elements within a wide optical range, with no interferences;
- the ability to analyse simultaneously all elements of interest, including light elements (Si, Mg, Al, C,);
- no ionizing radiation (neutron, gamma and Roentgen) risk for production personnel, no requirement for special permits or regular inspections by the regulatory bodies dealing with radiation monitoring;
- good accuracy regardless of bulk size, surface quality, thickness of the material layer on the belt;
- stable long-term calibration;
- simple and safe operation and maintenance;
- low cost of ownership.

MAYA is currently used in the metallurgical, refractory, potash and phosphate industries [9-11]. It has also been successfully tested in coal, non-ferrous and cement industries, and the production of various industrial materials (lime, limestone, quartz, etc.). MAYA has been designed to operate in automatic mode all-year-round, 24/7, under very harsh industrial conditions (high humidity and temperature variation, vibration, dustiness, etc.). MAYA can be easily integrated with any kind of SCADA and sorting/crushing/dosage equipment for process control. According to our customers' feedback, a typical payback period for our system is only several months. Figures 3 and 4 present breakdown spectra of bauxite and alumina. Both major, Al, and minor elements, impurities, such as Si, Fe, Mg, Ca, can be confidently identified and measured.

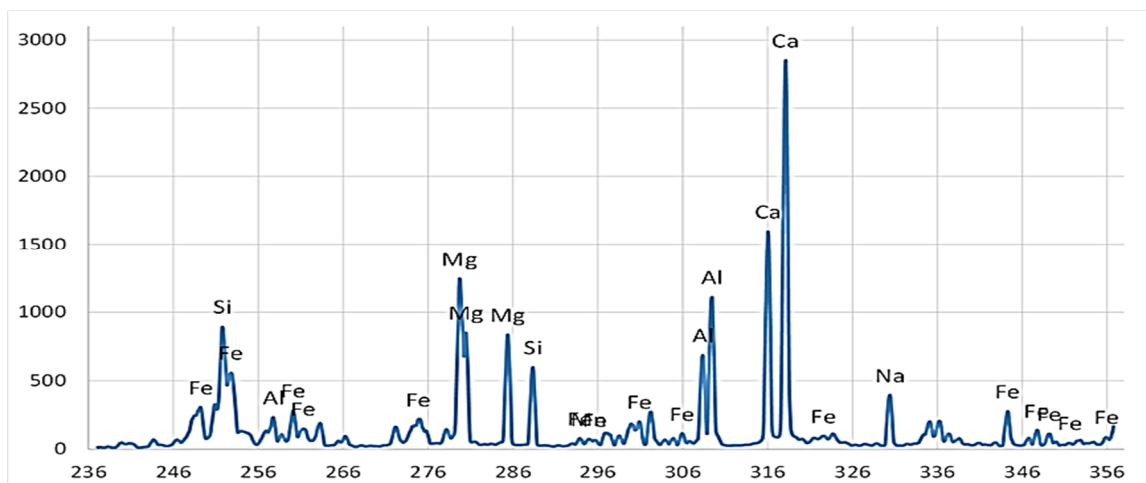


Figure 3. Typical bauxite UV spectrum.

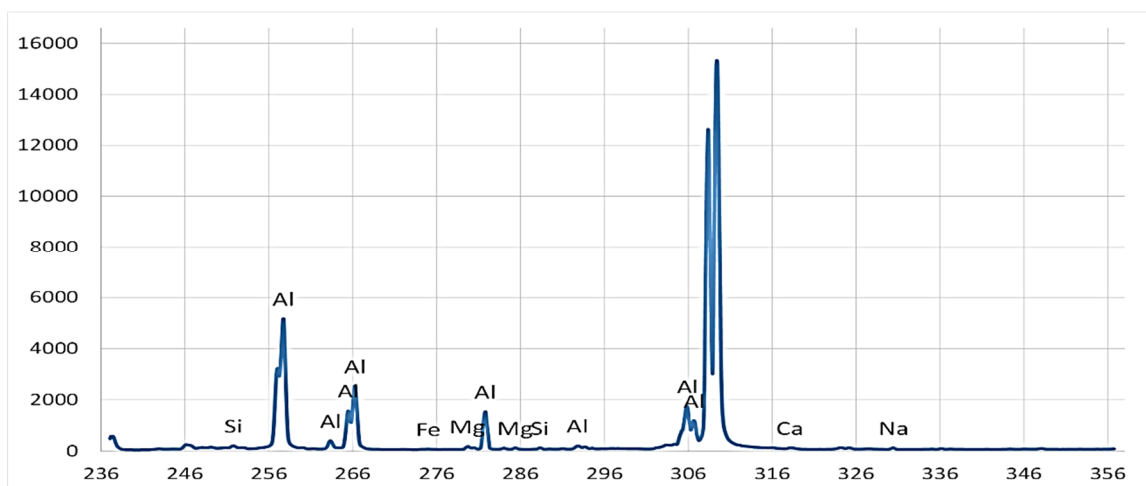


Figure 4. Typical alumina UV spectrum.

Such clear analytical lines enable very good correlation between online LIBS and traditional laboratory results (Figure 5 - 6). According to our accumulated experience, such data indicates successful industrial implementation.

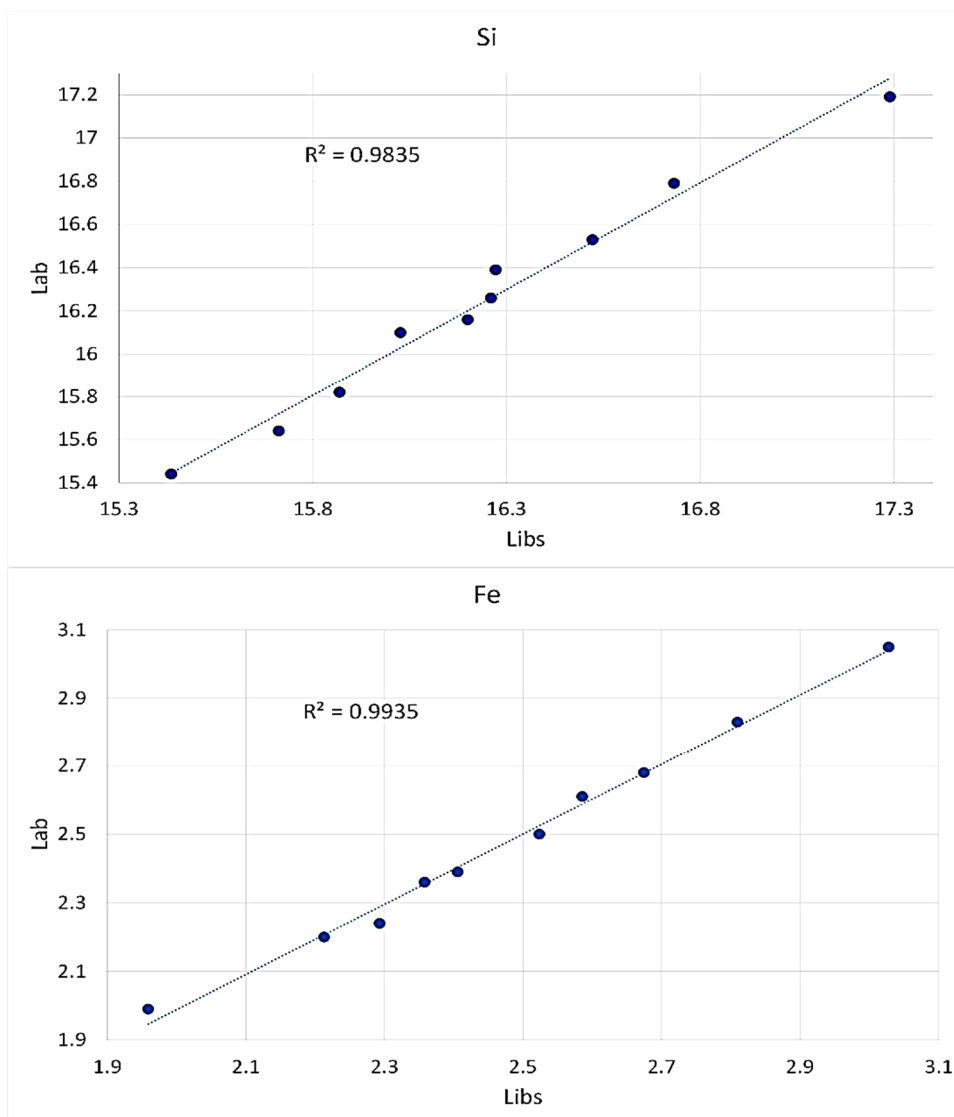


Figure 5. Comparison between online LIBS and laboratory data for bauxite samples.

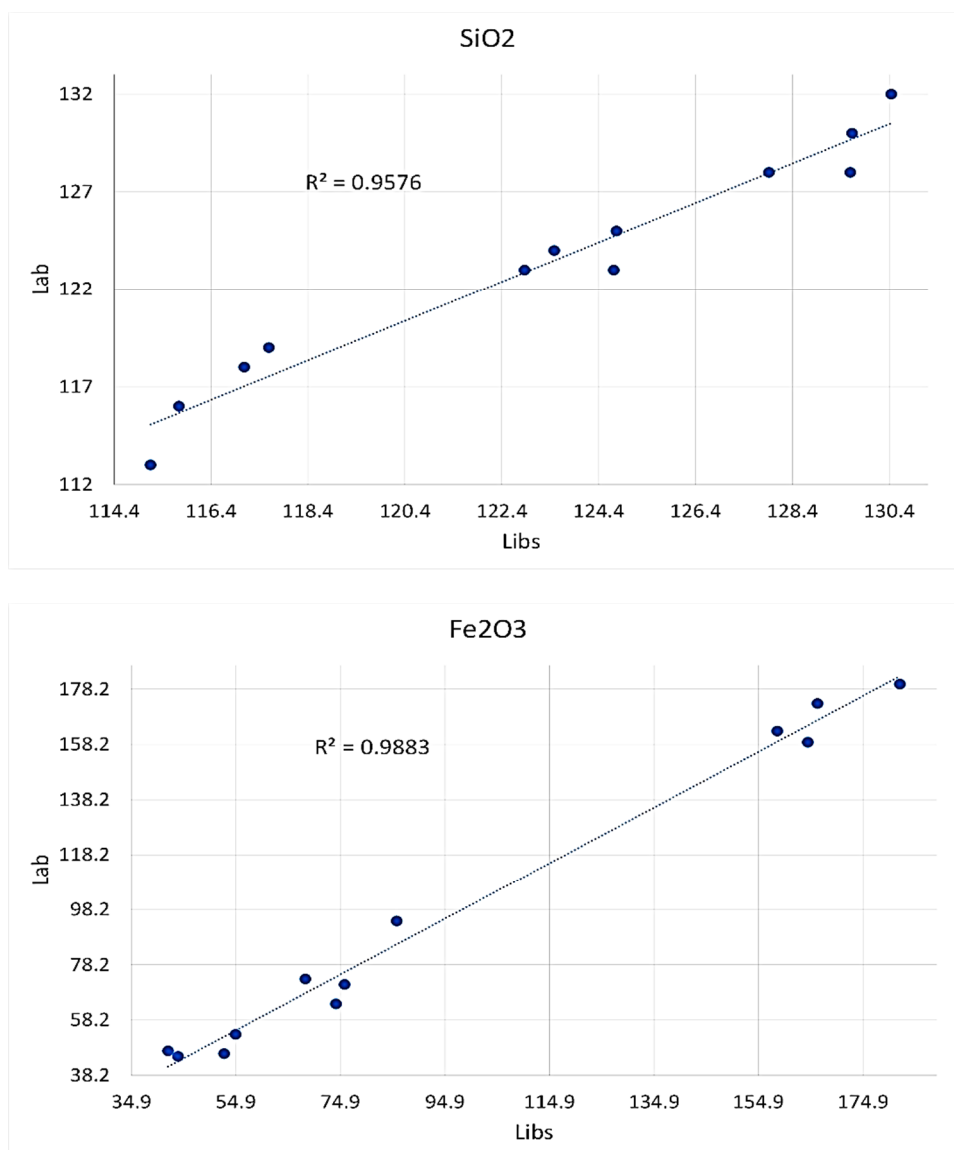


Figure 6. Comparison between online LIBS and laboratory data for alumina samples.

4. Advanced Analytical Approaches

To ensure stable and accurate measurements in real time, Lyncis uses chemometrics and optimization approaches. Traditional calibration methods often give poor results for real plant operation, where many other factors influence results (e.g. weather conditions, grain size variation, moisture level, etc.).

For a long time, specialists in the field of laser ablation and optical emission spectrometry (OES) have been solving the problem of calibration of LIBS systems. Despite the fact that Plasma Physics is a very complicated field, most mathematical models have long been based on the basic principles of analytical chemistry, where it is generally believed that the growth of the peak of the element under ideal laboratory conditions is proportional to the increase in the concentration of this element.

However, the need to analyse materials under industrial conditions, where, in contrast to the laboratory environment, a large number of factors vary in addition to the actual concentrations

of the elements studied, including particle size distribution, material density, layer thickness on the conveyor belt, flow turbulence or laminarity in the case of liquid analysis, dustiness, humidity, variations in ambient temperature at the measurement place, etc. All these factors lead to a number of challenges, which cannot be solved by the classical approach described above.

These challenges can be partly overcome and their impact mitigated using various types of Calibration-free approach. However, the accuracy of the tools calibrated by this approach only allow qualitative analysis of materials, and are not always suitably accurate or precise for process control, often expected to be within narrow concentration limits. That is why Chemometrics, in which most of the methods are directly borrowed from those widely used in Machine Learning, has become recently very popular in LIBS, providing a new approach to task solving from a Machine Learning perspective.

The main feature of these approaches is the number of samples required for calibration. If 5 - 6 samples are enough to build a linear model based on peaks of 2 - 3 elements or a calibration-free model, the classic number of samples in data science is not even hundreds, but tens of thousands of samples. This is absolutely impossible under the operating conditions of industrial enterprises. The direct use of methods borrowed from Machine Learning leads to the illusion of building a good calibration on dozens of samples, which subsequently produces unreliable results in a real environment. For example, this can be observed as a result of the direct use of Artificial Neural Networks in LIBS, and attempts to use Deep Learning only aggravate the problem. Therefore, the key challenge in building a reliable calibration with a limited number of samples is the proper integration of Machine Learning methods into the general ideas about the physics of the process.

Some methods that have proved themselves in practice are described below. Many works in the field of LIBS use the PCR (Principal Component Regression) method for calibration at the laboratory scale. At the industrial scale, PCA (Principal Component Analysis) can be used to pre-process data. The key advantage of this method is that it does not require tags (lab data) at all. Therefore, the training set can be formed not by the averaged spectra of the samples (there are few of them), but by the single spectra (it does not take long to get 10,000 spectra of the material, and the data volume already fully corresponds to the methods of Machine Learning).

At the same time, it is possible to use a PCA decomposition not only for regression purposes (this usually does not allow achieving the required accuracy), but also for the purpose of data normalization. The issue is that with the PCA decomposition, the first one or two components describe not the required concentration fluctuations, but those undesirable effects mentioned above. Therefore, the following method is quite meaningful: obtaining a PCA decomposition for all available spectra, zeroing out the first 1-2 components in the diagonal matrix, and the subsequent inverse transformation. The spectra obtained at the output are fairly qualitatively differentiated in intensity and in the influence of moisture and non-focus. In view of the fact that PCA is a technique of linear transformations, and many effects of plasma physics are fundamentally nonlinear, it also looks promising to replace PCA with Autoencoder, which, with a very similar ideology, uses more complex nonlinear models.

The second most important element in building models is the use of models of gradient descent with learning rate not equal to 1. Selection of an effective learning rate largely depends on the volume of the training sample. The classic Machine Learning often involve very small values of the learning rate of the order of 10^{-6} and even smaller. However, this requires training sample sets of the classic volume for Machine learning. Therefore, to calibrate LIBS systems on several dozen samples, we can recommend the use of the learning rate at the level of 0.1 - 0.25, if several hundreds of samples are available, it is possible to consider the values about 0.03 - 0.1. The experience shows that a meaningful choice of the learning rate makes it possible to obtain

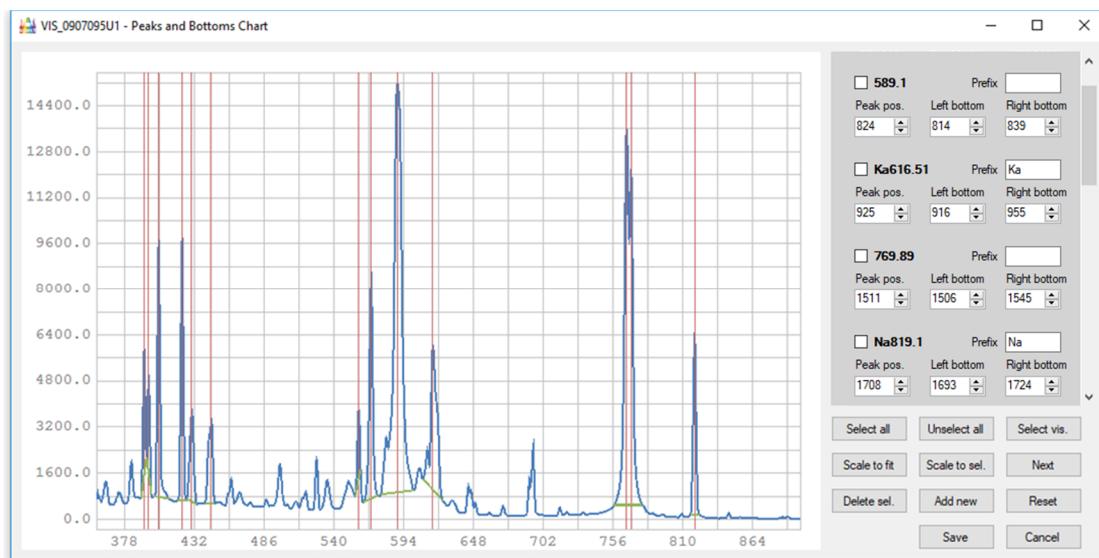


Figure 8. Spectra mark-up.

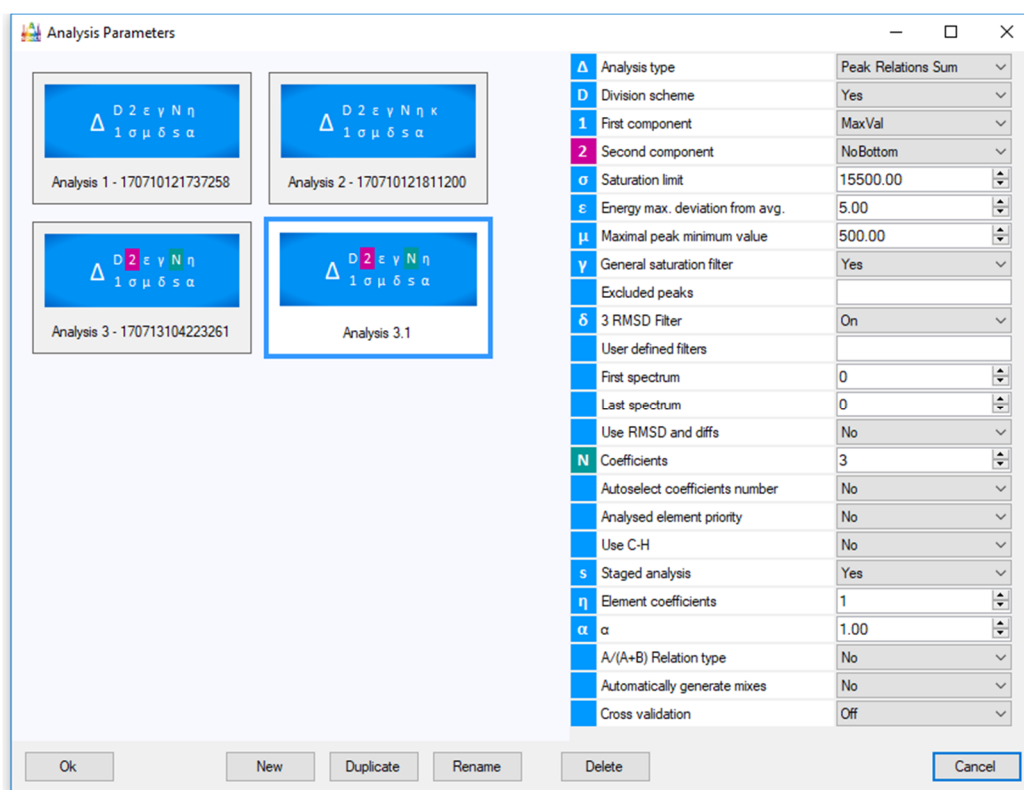


Figure 9. Analysis parameters.

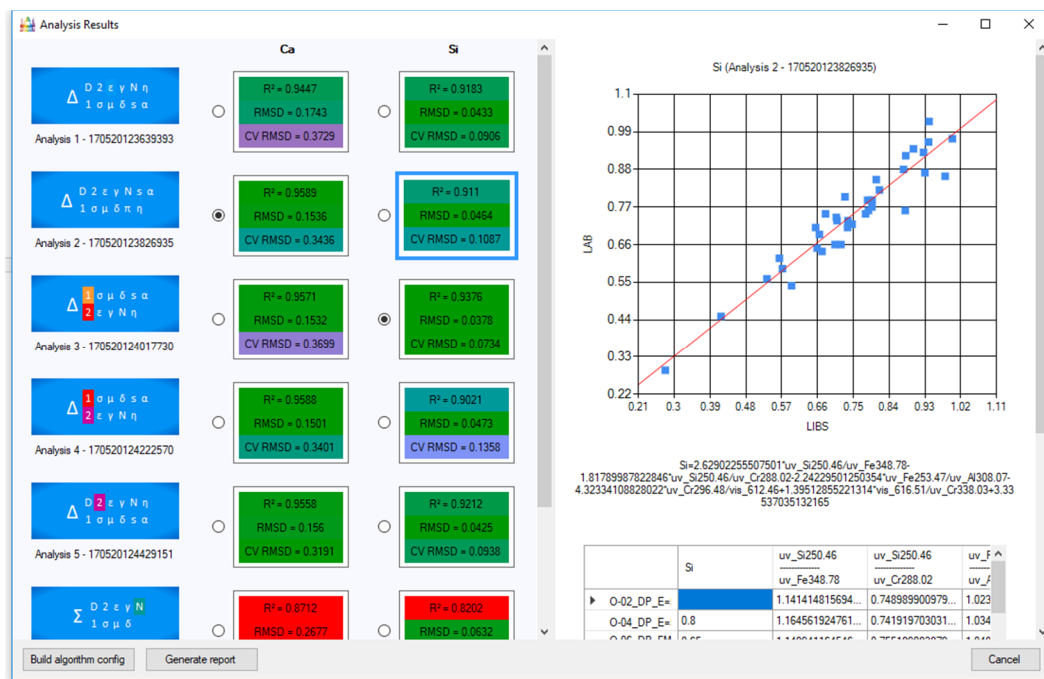


Figure 10. Analysis results.

The algorithms and their optimal default parameters, developed at the stage of studying the spectral properties of the material and performing of the on-site calibration of the analyser, allow even the beginner-level users (technicians and laboratory staff) to achieve good results not only at the initial on-site calibration, but also in its further adjustment. This is required as more data on the properties of the material on the conveyor belt are accumulated, which cannot be studied during the initial commissioning period and vary depending on the material supply with different chemical and mineralogical compositions, variations in density, particle size distribution, moisture and temperature.

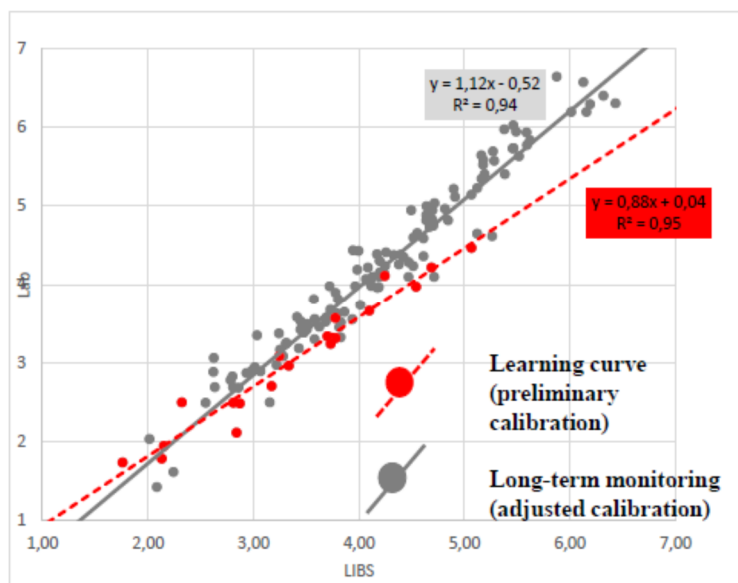


Figure 11. Long-term calibration.

Our experience shows that to get a stable algorithm that can work for a long period, the system user needs to carefully monitor the analyser readings for several months after commissioning and, if necessary, add new calibration samples to the calibration algorithm to take into account the factors described (Figure 11).

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