

Phosphate ore beneficiation via determination of phosphorus-to-silica ratios by Laser Induced Breakdown Spectroscopy

George Asimellis^{a,*}, Aggelos Giannoudakos^{b,c}, Michael Kompitsas^c

^a Faculty of Physics, Aristotle University of Thessaloniki, Thessaloniki 54124, Greece

^b School of Chemical Engineering, National Technical University of Athens, 9, Iroon, Polytechniou Str., Zografou, 15780 Athens, Greece

^c National Hellenic Research Foundation, Theoretical and Physical Chemistry Institute, 48, Vasileos Konstantinou Ave., 11635 Athens, Greece

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Abstract

We report development and application of an in-situ applicable method to determine phosphate ore rock quality based on Laser-Induced Breakdown Spectroscopy (LIBS). This is an economically viable method for real-time evaluation of ore phosphate rocks in order to separate high-silica pebbles prior to deep beneficiation. This is achieved by monitoring relative emission line intensities from key probe elements via single laser ablation shots: the ratio of the phosphorous to silica line intensities (P/Si ratio) provides a simple and reliable indicator of ore rock quality. This is a unique LIBS application where no other current analytical spectroscopic method (ICP or XRF) can be applied. Method development is discussed, and results with actual ore samples are presented.

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1. Introduction

Rapid classification of phosphate ore samples during extraction on site and in real time is very important in the phosphate industry. Gradually, the high-grade, easy-to-process deposits are being depleted. Eventually, then, lower-grade, more contaminated ore bodies must be considered. These bodies are often rich in high silica-content ore pebbles, which are undesirable because, due to their highly abrasive nature, they induce heavy wear in subsequent ore processing (grounding) hardware.

To detect these rich-silica content pebbles, current practices must stockpile ore until quality control data from laboratory-based analysis from expensive, massive, centrally located instruments, such as neutron activation analyzers become available. This requires samples to be collected, transported to the laboratory, analyzed, recorded, and data transmission back

to the excavation area. Only then the shipping or discarding decision is made. This process is slow, subject to numerous errors and does not support real time management of the extraction process.

Alternatively, the old-fashioned visual observation of ore during production can be employed to classify the ore. Obviously, this practice can result in shipping of undesirable ore to the processing plant or in discarding of acceptable ore.

Other ore beneficiation techniques include flotation, however there is significant loss of good grade phosphate ore when the ground high-silicate pebbles go through the desliming process, which separates the rocks by size. Furthermore, it is important to significantly reduce high-silica content ores before the grounding step. Thus a reliable on-line, in-situ analytical method has a significant potential.

This paper investigates a beneficiation concept to analyze fast-moving ore rocks on a conveyor belt by estimation of key elemental properties to enable separation of high silica content (poor ore) rocks, thus improving the overall ore quality. Identification of different types of ore rocks can be achieved by laser-induced breakdown spectroscopy (LIBS) via determination of Phosphate-to-Silica content with rapid, single-shot

* Corresponding author.

E-mail address: gasim@auth.gr (G. Asimellis).

multi-element analysis of ore rocks requiring no previous processing or direct contact with the sample, just optical access to the samples.

The experimental realization of LIBS ([1], and references therein) is simple: a laser pulse is focused on the sample surface. With irradiances in the range of 1–10 MW/cm², breakdown occurs, causing localized material ablation, because the electric field at the focus exceeds the dielectric strength of the surrounding environment. Via further photon (and multi-photon) absorption, atoms and ions are created in a hot (many 1000 K) plasma. After ion–electron recombination, subsequent radiative decay leads to emission of specific atomic lines. This optical signal is spectroscopically analyzed and thus the sample elemental composition can be identified. Often, time-resolved detection is employed to differentiate between specific emission lines, necessitating a gated detector.

LIBS has been widely used for direct real-time and in-situ elemental analysis of a variety of materials [2–9]. Due to its specific advantages, LIBS is particularly suitable in quality control applications where fast, non-contact analysis is required [10–13] and specifically, in minerals [14–18]. For this specific application, no other current spectroscopic method (such as Inductively Coupled Plasma Atomic Emission Spectrometry, ICP-AES [19,20] or X-ray Fluorescence, XRF) can be applied: ICP due to significant sample pre-processing required and XRF because in general low-atomic number elements (e.g. P) are very difficult to detect.

While there is a large number of publications reporting LIBS application on metallic elements [21], other elements with strong non-metallic character, such as sulfur, carbon and phosphorus, as well as halogens, have been less investigated. Qualitative and quantitative determination of these high-excitation energy, non-metallic elements is necessary in a wide range of applications, such as: analysis of pharmaceutical products (via chlorine [22] and fluorine detection), on-line compositional analysis of liquid metals and alloys (carbon, silicon, phosphorus etc. in molten steel), analysis of polluting

hazardous gases and aqueous solutions (via chlorine and fluorine detection), environmental applications (via sulfur [23] detection), mineral classification and ore beneficiation (via phosphorus detection), recycling of electronics (via bromine [24] detection) or chemical weapons traces detection.

The number of LIBS reports on phosphorus detection is rather small. The first-known report on phosphorus LIBS detection dates back to 1983, when Radziemski et al. used a nebulizer/heat chamber system to produce an aerosol from a liquid solution and subsequently analyze it [25], with a detection limit of 1.2 ppm. J. Sneddon [26] reported P detection at 15 ppm in atmosphere. M. Casini et al. [27] reported P detection at 442.1 nm with a limit of detection in the vicinity of 200 ppm. St. Onge et. al. [28] reported P detection by LIBS in pharmaceutical products. Rosenwasser et.al. [14] employed phosphorus detection for ore beneficiation.

For method development a simple laboratory LIBS setup was used (Fig. 1) using a fundamental Nd:YAG pulsed laser (YG 981, Quantel, France) with pulse energy 30 mJ and pulse duration of 7 ns, conventional optics for laser delivery to the target and plasma optical emission collection, and a Czerny–Turner (HRP, Jobin-Yvon, Edison, NJ, USA) spectrometer fitted with 2400 lines/mm holographic grating. A gated intensified CCD camera (GEN II, Andor Technologies, Belfast, UK) with 15% quantum efficiency (average) was used for optical signal detection. A servo-controlled, sample positioning stage was used to position the sample on the desired location relative to the incident laser beam, allowing the required number of shots to be fired at each spot, before it could be moved to the next spot or next specimen.

2. Method development

The application seeks to classify individual ore rocks based on the ratio of phosphorus to silicon wt.% content (actual P₂O₅/SiO₂ ratio) in real-time. Method development and testing was based on actual phosphate rock samples from

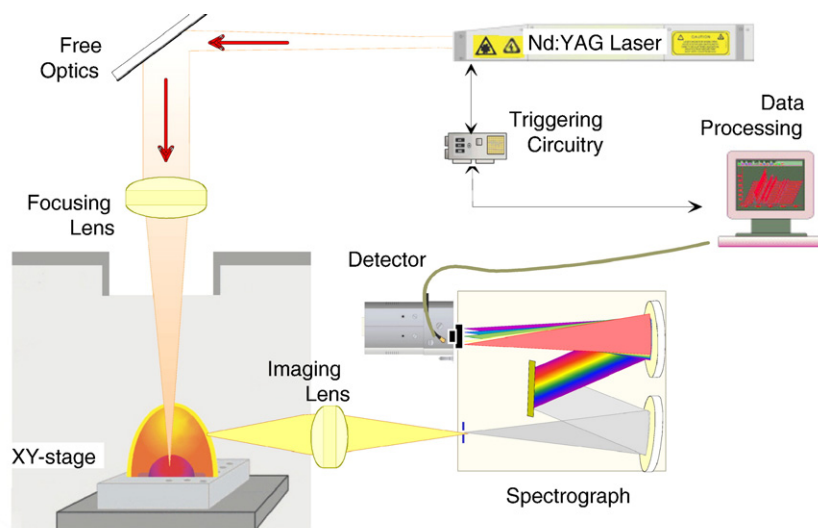


Fig. 1. Schematic of LIBS experimental setup.

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