



TECHNICAL NOTE

Determination of uranium content in phosphate ores using different measurement techniques



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Abstract The most important unconventional source of uranium is found in phosphate deposits; unfortunately, nowadays its exploitation is limited by economic constraints. The uranium concentrations in phosphate ores in the world vary regionally and most countries with large phosphate deposits have either plant in operation to extract uranium or are at the stage of pilot extraction plants. The aim of this investigation is to evaluate uranium content in the Saudi phosphate ores for, at least, two reasons: firstly, upgrading the phosphate quality by removing the uranium content in order to reduce the radioactivity in the fertilizer products. Secondly, getting benefit from the extracted uranium for its domestic use as a fuel in nuclear power and desalination plants. The results of this study show that the uranium concentration in Saudi phosphate rocks is relatively low (less than 100 ppm), which is not economically encouraging for its direct extraction. However, its extraction as a byproduct from the phosphoric acid, which will have higher concentration could be quite promising and worth exploiting.

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1. Introduction

Long term sustainability of nuclear power is a thriving thought for the ambitious countries which are looking forward to start a nuclear program for power generation and desalination. Without depending upon the suppliers, the nuclear fuel must

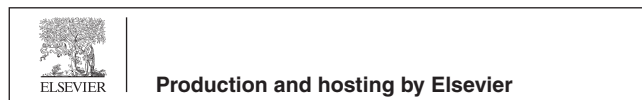
produce power in the long-term. Requirement of high technological maturity, complex fuel cycle and huge investments calls for the nuclear fuel import at the beginning. So, initially it is not feasible when the country does not have a proper nuclear reactor park. However, having some uranium reserve in the country will be helpful to obtain the required nuclear fuel either from outside or inside. Therefore, countries that plan to start nuclear power generation should start in parallel a conventional and unconventional uranium acquisition program.

The largest phosphate rock deposits worldwide are located in one belt covering all North African countries and continue through Jordan and north-west of Saudi Arabia (Ragheb and Khasawneh, 2010). The Saudi Arabian phosphate deposits are estimated well above 7000 million tons located in various locations in the north of the county (Saudi Geological Survey,

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2010). In 2012, a consortium of local companies comprising Ma'aden and SABIC started extraction and processing of the phosphate rocks. The project based in Ras-Alkher will produce 3 million tons per year of di-ammonium phosphate fertilizer. Moreover, it will also produce 400,000 tons of ammonia and 200,000 tons of phosphoric acid per year, which will be sold domestically.

The uranium concentrations in phosphate deposits vary worldwide with a range of 50–200 ppm (parts per million). In general, uranium extraction requires the conversion of phosphate to phosphoric acid, followed by solvent extraction. The estimated world annual capacity of uranium phosphates production is about 3700 tons. There are 400 wet process phosphoric acid plants in operation, worldwide. Some plants were in operation in the United States since 1976 and many others were built in different countries such as Canada, Belgium, and Spain; while in the Arab world Morocco, Tunisia, Egypt and Jordan that have big phosphate reserves are at the stage of pilot extraction plants (Chatra, 2009).

The increasing cost of uranium in the international market in the last decade encourages its recovery from the phosphate ore. Therefore, it is necessary to launch research studies for uranium recovery locally from phosphate ore in order to use this byproduct as a fuel in nuclear power and desalination plants in the future. In order to address this issue, different measurement techniques were used in this study to determine the concentration of uranium in various phosphate rocks. The X-ray fluorescence spectrometry (XRF) is a well-established measurement technique used in many fields due to its advantages: simultaneous multi-element capability, rapid, wider dynamic range, simple sample preparation procedure, good reproducibility and low operating costs (Beckhoff et al., 2006; Adam et al., 2009, 166). The laser induced breakdown spectroscopy (LIBS) is a relatively new method for elemental analysis; but it has recently become very attractive for the same advantages as that of XRF and in addition it is very sensitive to light elements (David et al., 2006; Chinni et al., 2009, 1240; Popov et al., 2009, 166). These two techniques were used in this work for elemental analysis of several phosphate samples in order to compensate the limitations of each other. The low background counting (LBC) system was employed to confirm the uranium concentration in phosphate samples (Michael, 2010).

2. Experimental setup

2.1. Sample collection and preparation

The samples were collected from the mine as a rock raw material from three different locations (L1–L3) and taken to the laboratory for sample preparation and measurements with different techniques. The phosphate rocks from each location were crushed by Jaw Crusher to a size of about 5 mm thick granules, then further processed in Ball mill PM-200 for 20 min fine grinding. The last operation was repeated until all the powder passed through 75 μ m mesh sieve.

The last step of sample preparation was the pellet pressing. The powder obtained from each location was used to prepare fifteen pellets of about 10 g each. The powder was placed in an aluminum cup of 32 mm diameter and pressed using hydraulic press of 30 ton pressure.

2.2. XRF measurements

The energy dispersive X-ray fluorescence spectrometer (EDXRF) unit, JSX-3202-M used in this project was manufactured by JEOL Company in Japan. This unit is able to analyze a wide range of specimen samples in the form of solid, powder and liquid. The elements that can be measured by this instrument range from sodium (Na) to uranium (U). The spectrometer incorporates the X-ray tube, a Si(Li) detector with 133 eV resolution at 5.9 keV (Mn- k_{α}) and the vacuum analyzing chamber. The accompanied computer contains the software to drive the unit and spectrum analysis software. This software allows simultaneous multi-element spectral measurement, and qualitative and quantitative elemental analysis using fundamental parameter (FP) and reference methods (Jeol, 2005). The detection limit of the system varies from 10 to 100 ppm depending on the atomic weight of the element (Kadachi and Al-Eshaikh, 2012, 350).

After the equipment setup and calibration of the system using phosphate ore reference standards, each of the prepared samples was measured for 3 min, which is enough to collect sufficient counts to minimize the counting statistical error even for minor peaks present in the spectra. The spectrum of each sample was analyzed qualitatively and quantitatively in order to identify all elements present in the sample and to determine their respective concentrations (Al-Eshaikh and Kadachi, 2011, 75). Fig. 1 shows XRF spectrum of one sample from Jalamed phosphate ore.

2.3. LIBS measurements

The LIBS unit used in this project is called Spectrolaser-7000 that is a compact, self-contained device that utilizes laser-induced breakdown spectroscopy to determine the elemental composition of different sample types. The Spectrolaser is an integrated analysis system comprising an excitation laser, optical spectrographs and gated CCD detectors. The laser used in the system is a high power (5–300 mJoule) Nd: YAG laser which yields a 7 ns pulse, at a repetition rate of up to 15 Hz and parallel processing design comprising seven spectrographs. Each spectrograph is preset to wavelength ranges encompassing the regions of richest elemental fluorescence. Therefore, the device using multiple spectrographs achieves wide spectral coverage while maintaining the resolution required in observing the elemental fluorescence with minimal interference. The Spectrolaser system is managed by an interactive software system that allows full instrument control from the computer. This includes configuration of the instrument, calibration of the individual spectrometer channels and parameter selection of the system such as delay time, laser power, number of shots, that were fixed in order to obtain the optimum optical spectrum (Spectrolaser, Elemental analysis system, 2010; Cremers and Chinni, 2009, 660).

After the equipment setup, the acquisitions of 30 shots spectra per reference standard sample of known composition were performed. These shots were made at 10 different positions with three shots per position. These spectra were processed to construct the calibration curves of element concentrations versus line area intensity. In order to avoid plasma fluctuation and shot location heterogeneities, an average spectrum of all shots by sample was calculated. In

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