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Fingerprinting natural graphite deposits

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Graphite is an essential raw material in a range of applications due to its extraordinary chemical and physical properties. As graphite is also one of the fundamental raw materials in energy storage, its responsible sourcing is of prime importance and it is officially classified as a critical and strategic raw material (CRM and SRM). In the European Union, ambitious goals were set (i.e. Critical Raw Materials Act) with respect to the responsible sourcing of critical raw materials. Among others, not more than 65% of each of these materials should stem from a single third country by 2030. However, currently there is no routine methodology available to distinguish natural graphite deposits from each other.

The EU-funded MaDiTrace (Material & Digital Traceability for CRM Certification) project aims at reinforcing the transparency, reliability and sustainability of critical raw material supply chains. The project is focusing on four critical raw materials: Li (BRGM, France), Co (GTK, Finland), natural graphite (University of Leoben, Austria) and rare earth elements (Ghent University, Belgium). The project's goals include the development of analytical fingerprinting protocols (laboratory and on-site), their incorporation into certification schemes and the integration of the above technological solutions into digital product passports. Analytical fingerprinting aims at the differentiation between different natural deposits and the detection of material characteristics, which can be followed along the value chain from the raw material until the product.

Due to its chemical and thermal stability, the analysis of graphite is particularly challenging. In the framework of the project, we test the applicability of Raman-spectroscopy, Carbon stable isotopes, ICP-MS, LA-ICP-MS and Laser-induced breakdown spectroscopy (LIBS) for distinguishing graphite deposits. In this study, we focus on the results acquired by a LIBS coupled to a laser-ablation system (ImageGEO193) on a series of pressed natural graphite pellets, from the most important graphite producers worldwide. By applying LIBS-mapping with 100 µm spot size and 90 µm x-y overlap on a 1*1 mm area on each pellet, we can shed light on the spatial distribution of impurities in graphite. Most detected elements (e.g. Ca, Mg, O, Si) show high anomalies in spatially restricted regions implying mineral impurities as possible sources. On the other hand, during the acquisition of the maps, 100 multielemental LIBS spectra are produced per second, which provide an excellent basis for multivariate classification tasks. We introduce several approaches on raw, normalized and quantitative graphite data by various classifiers. Our tests indicate that individual shots on all graphite concentrates can be classified correctly by at least 80% accuracy. This makes LIBS a strong candidate for becoming a standard tool in graphite traceability tasks, in accordance with its successful forensic application on other materials (e.g. [1]), whereas other methods can be used in combination with LIBS to further improve classification accuracy.

[1] M. G. Nespeca, A. L. Vieira, D. S. Júnior, J. A. Neto, E. C. Ferreira. *Food Chemistry*, 2020, 311,125886.

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