LIBS-SCReeN Screening Critical Raw materials from exploration to (post)beneficiation using LIBS techniques

Contract - B2/191/ P1/LIBS-SCReeN

Context

Critical Raw Materials (CRM) are economically and strategically important for key industry sectors and future applications but have a high-risk associated with their supply. Meeting the growing demand for CRM is one of the greatest global challenges in the next decades, and (re)exploitation of domestic resources, including recycling, will become increasingly significant. In Belgium, zinc-lead deposits have been known since Prehistoric times and were mainly exploited in the 19th century, but the economic potential of what remains is largely undetermined. It is known these deposits host CRMs such as germanium (Ge) and gallium (Ga). Of equal importance in the case of new mining and beneficiation activities is the presence of elements that can turn into contaminants for the environment, e.g., arsenic (As) and cadmium (Cd).

Flexible, rapid and reliable techniques are needed to enhance our capacity of CRM exploration, exploitation, recycling and environmental impact monitoring. Traditional geochemical characterisation techniques are typically time-consuming, expensive, and require sample preparation, which limits the size and representativity of datasets. In this context, Laser-Induced Breakdown Spectroscopy (LIBS) emerges as an advantageous alternative for characterisation of geological material. The technique is based on emission spectroscopy using high-energy laser pulses as the excitation source. Besides allowing rapid data acquisition with no to little sample preparation, one significant advantage of LIBS is the possibility of simultaneous analyses of major, minor, and trace constituents, including lighter elements (e.g., C, B, Be, and Li) which are notably difficult to detect with many other techniques.

One of the challenges to upscale the application of LIBS techniques for large screening efforts is the generation of large amounts of complex spectral data. This requires adapted technologies of data processing and analyses and can greatly benefit from the evolving landscape of Machine Learning (ML) and Artificial Intelligence (AI) tools. Especially in potential future automated LIBS analyses, data extraction and processing pipelines that are also automated are strongly needed. Protocols for screening, element identification and mineral classification using LIBS represent important steps towards standardisation, which can provide a valuable contribution to the international LIBS community.

Objectives

The project LIBS-SCReeN aimed at improving already existing LIBS technology in Belgian laboratories and developing and implementing further LIBS analytical capacity to test the application of the technique to the whole life cycle of CRM, from exploration of resources to post-beneficiation scenarios of recycling and/or environmental issues. The specific objectives of the project were to 1) develop and implement scientifically sound workflows to exploit LIBS for elemental screening at multiple scales, 2) to optimise element identification workflows from data acquisition to treatment to ensure the applicability of LIBS to different deposits and environmental needs, and 3) to disseminate and cluster local activities with the global scientific LIBS community and EU projects to establish a Belgian LIBS expertise hub.

Methodology

The project utilized two primary LIBS setups: one at UMONS and another at RBINS-GSB, with a third setup being developed at ULiège. Methodological approaches included :

- Reference Sample Analysis: Comparing LIBS elemental analyses with SEM-EDS, μ XRF, and LA-ICP-MS on 13 reference rock samples.
- **Sample and Core Inventory**: Cataloguing historical Zn-Pb ore samples from geological collections at project partner institutions and collaborating with UGent for drill core samples.
- Screening Approaches: Implementing targeted and randomized screening methodologies on hand samples and drill cores to evaluate user-bias and automation suitability.
- Soil Contamination Screening: Conducting semi-portable LIBS analyses on soil samples from Belgian and French sites to assess heavy metal contamination.

Key Achievements

- Development of LIBS Workflows: The project successfully developed and tested screening protocols from data acquisition to LIBS spectral processing. Hundreds of rock and soil samples were analyzed, demonstrating LIBS's capabilities in detecting CRM and potential environmental contaminants. Two methodological approaches were compared: targeted analyses with fewer, more controlled measurements, and randomized, automated analyses with less user bias.
- Optimization of Element Identification: LIBS techniques were optimized for various scales, from hand-sized samples to entire core samples. The project enhanced setups at UMONS, RBINS-GSB, and ULiège. Advanced machine learning models were developed to manage large spectral data volumes, enabling differentiation between mineral phases and rapid detection of critical elements such as germanium (Ge) and gallium (Ga), as well as pollutants like cadmium (Cd) and arsenic (As).
- 3. Screening Pilots: A total of 408 samples were screened, encompassing a wide variety of mineralogical compositions associated with Zn-Pb occurrences in Belgium. The screening pilots highlighted LIBS's ability to provide detailed multiscale distribution maps of chemical elements, showcasing its potential for both CRM exploration and environmental monitoring. Soil analyses from contaminated sites, such as the Sclaigneaux brownfield, validated LIBS's effectiveness in environmental applications.
- 4. **Dissemination and Valorization:** The project engaged in extensive communication and dissemination activities, including workshops, conference presentations, and collaborations with international research institutions. The establishment of the Belgian LIBS Research Cluster

(<u>belibs.naturalsciences.be</u>) was a significant outcome, aiming to continue LIBS research and offer expertise and services through the infrastructure developed during the project.

5. **Digital Presence and Publications:** The project maintained a robust digital presence with a dedicated LinkedIn page and Twitter profile, attracting followers and generating significant engagement. A series of educational videos on YouTube explained the main tasks and innovations of the LIBS-SCReeN project. Additionally, several peer-reviewed publications and conference abstracts were produced, further disseminating the project's findings.

Conclusions

The LIBS-SCReeN project demonstrated that LIBS is a powerful tool for CRM exploration and environmental monitoring. The development of optimized workflows and advanced data processing techniques, combined with the establishment of BELIBS, positions Belgium as a leader in LIBS research and application. Future research could focus on further refining these techniques and exploring new applications in various industrial and environmental contexts. The project's findings enhance the capacity to use LIBS in for pollutant-free CRM exploration and exploitation in diverse environments, including the recycling industry.