Critical Minerals Measurement by LIBS

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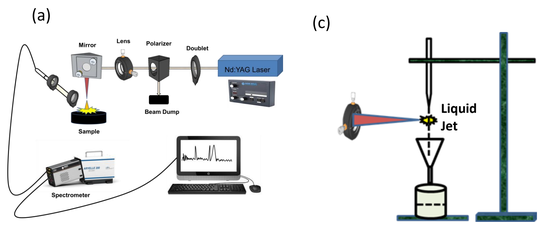
Objective

To develop a laser-induced breakdown spectroscopy (LIBS) system for critical minerals measurements.

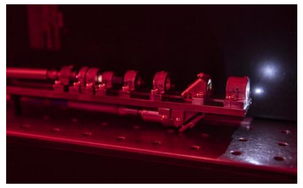
Laser Induced Breakdown Spectroscopy

- LIBS is an atomic emission spectroscopy-based analytical technique to obtain qualitative and quantitative elemental information of the materials.
- High-energy laser pulse creates a micro plasma plume on the sample by ablating a very small amount of material.
- The ablated material dissociates into excited ionic and atomic species.
- The excited atoms/ions present in the plasma emit light at their characteristic wavelengths.
- Spectral analysis of the emission spectrum from the plasma is used to infer the elemental composition of the sample.

LIBS Measurement Systems



(b)



(a) LIBS Bench top set up
(b) LIBS for Liquids
(c) LIBS Probe

Rare Earth Elements Measurement

- Test samples were collected directly from natural sites.
- For calibration two different mineral matrices, coal and rock were prepared. Rare earth elements (REE); La and Nd were doped into simulated coal and rock samples with their varying concentration.

Coal Simulant		Rock Simulant	
Coal Mineral Phase (wt.%)	6	Rock Mineral Phase (wt.%)	93
Graphite (wt.%)	94	Graphite (wt.%)	7

Composition of Coal & Mineral Phase Simulant

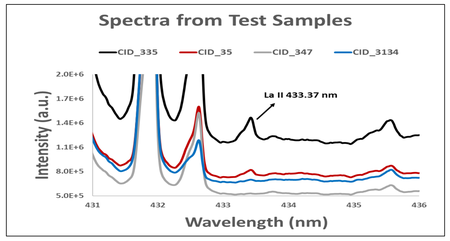
Compound	Coal Mineral Phase (wt.%)	Rock Mineral Phase (wt.%)
Al_2O_3	14.03	18.90
SiO_2	17.40	68.48
Fe_2O_3	7.08	3.15
KCl	0.47	3.81
$CaCl_2 \cdot 2H_2O$	41.43	1.62
$MgCO_3$	13.50	2.78
TiO_2	0.50	0.77
NaCl	5.58	0.49

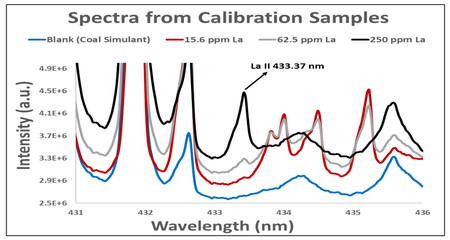
* Mineral simulant composition represents the average (ash) composition of coal samples (samples with 0–10 wt.% ash) and rock samples (samples with 90–100 wt.% ash) from the given sample set. Metallic elements composing greater than 0.1 wt.% of the ash (average of all samples) were selected to produce the simulants.

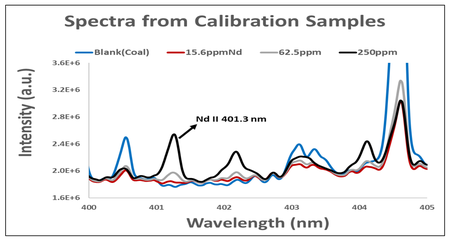
Emission Signal Detection

La II 433.4 nm, Nd II 401.2 nm

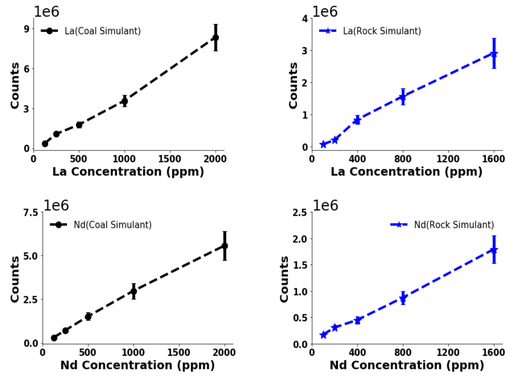
Experimental Parameters		
	Bench Top Setup	LIBS Probe
Laser energy	6 mJ	4 mJ
Gate delay	0.5 µs	0.5 µs
Gate width	5 µs	5 µs





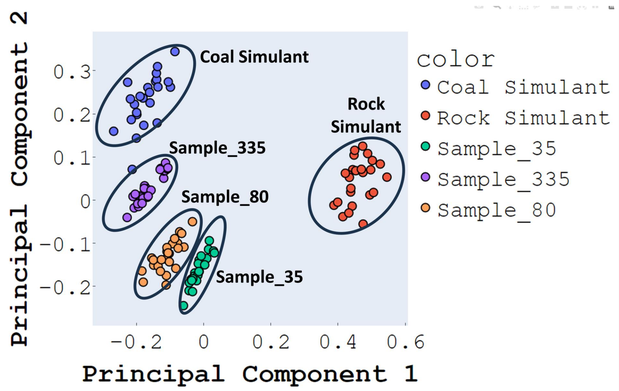


Calibration Curves & Limit of Detection (LOD)



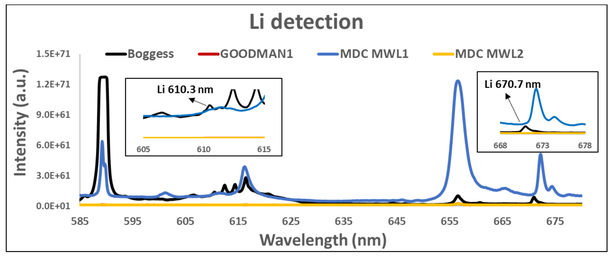
Matrix	La(Coal)	La(Rock)	Nd(Coal)	Nd(Rock)
LOD (PPM)	11.55	12.92	21.07	54.8

Principal Component Analysis (PCA)



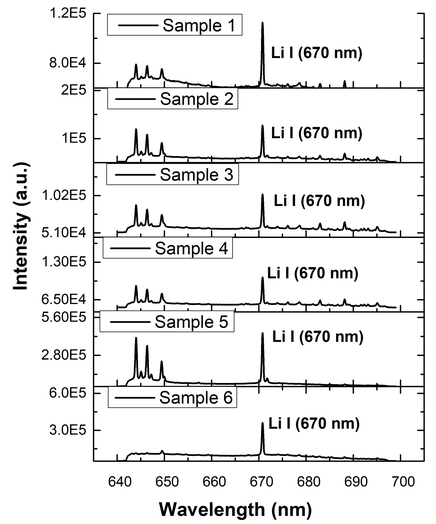
Li and Mn Measurement

Li Detection in Liquids

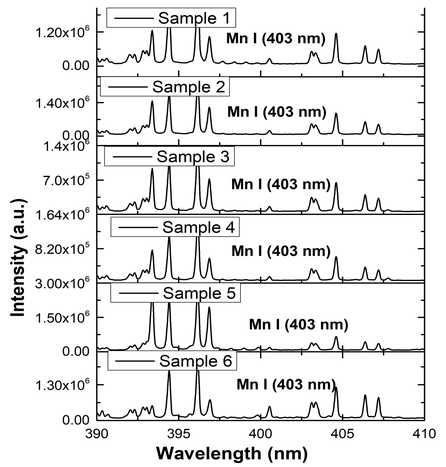


Samples	pH
Boggess	4.86
MDC MWL1	7.56
MDC MWL2	7.58
Goodman 1	3.22

Li Detection in Solids



Mn Detection in Solids



Conclusions

- LIBS technology was evaluated for some critical minerals measurements in solids and liquids.
- Results demonstrated that LIBS is promising technique for critical elements including REE measurements.

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