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Quantitative Analysis of Clay Samples, Using Laser Induced Breakdown Spectroscopy (LIBS) in On-Line, Real Time Mode.

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1. Technical task

- Quantitative analysis of clay samples for content of: Al_2O_3, Fe_2O_3, K_2O, CaO, TiO_2, Na_2O and SiO_2.
- Evaluating possibility of on-line, real time LIBS analysis of clay content on a conveyer belt.

2. The samples

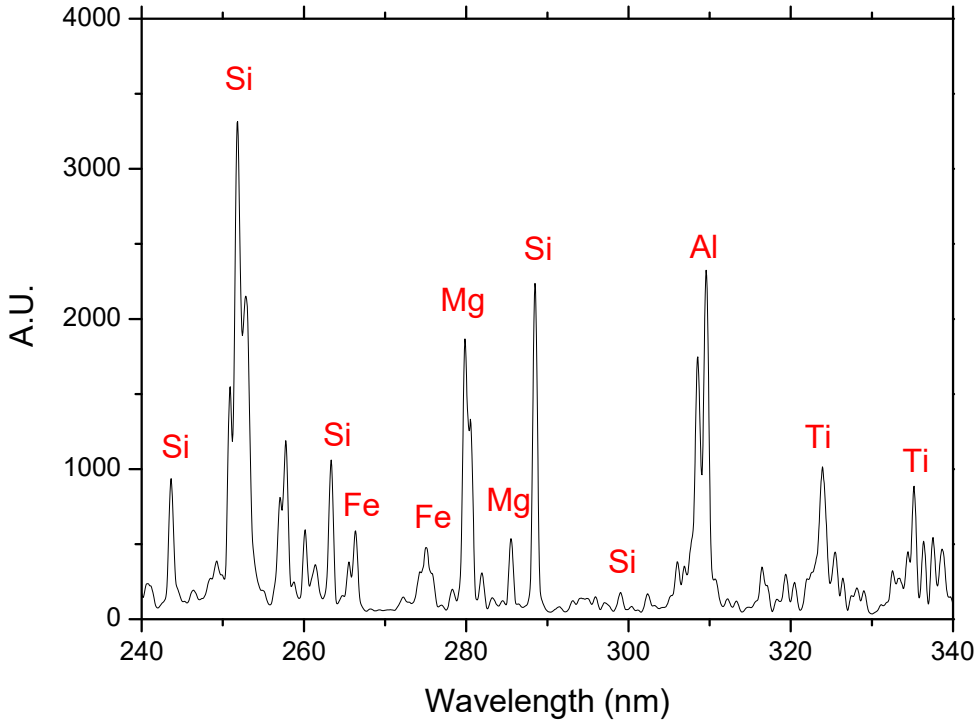
The received delivery contains 10 buckets of 5 litters each. Samples are of different color and texture: Following table describes chemical content of clay samples:

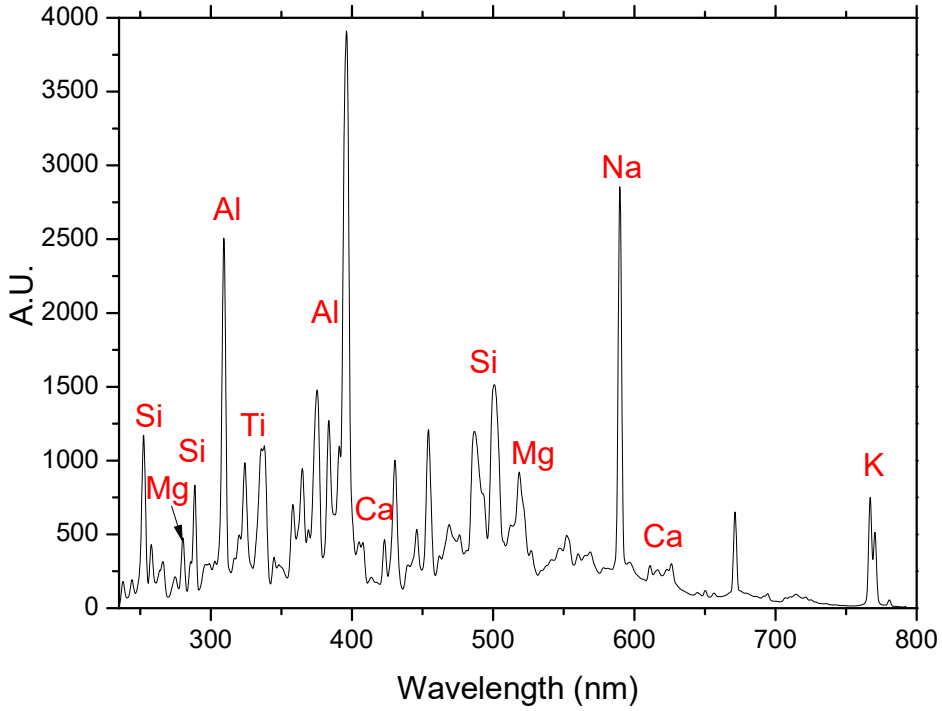
	Chemical XRF							
Sample	Fe2O3	Al2O3	TiO2	K2O	CaO	MgO	Na2O	SiO2
001	1.09	30.91	0.61	1.4	0.21	0.25	0.17	41.6
002	1.00	32.74	1.05	2.5	0.18	0.34	0.39	49.2
003	0.99	32.43	1.01	3.0	0.16	0.39	0.45	52.7
004	0.86	25.26	1.58	2.6	0.14	0.32	0.40	62.1
005	0.74	17.58	1.59	1.8	0.05	0.31	0.31	72.9
006	1.73	16.41	0.13	2.8	0.05	0.33	0.29	74.5
007	1.45	16.65	0.11	5.7	0.05	0.24	0.46	72.6
008	0.87	36.78	0.03	2.3	0.04	0.37	0.11	47.8
009	1.02	27.04	1.33	2.1	0.13	0.34	0.34	57.6
010	0.90	23.53	1.56	2.0	0.10	0.32	0.34	64.3

3. Experimental section.

The experiments were conducted using on-line analysis system equipped with double pulsed laser energy of 80 mJ. Each sample was measured using 2000 laser pulses in each analysis in order to receive sufficient statistics. The samples were rotating under the laser beam, imitating conveyer belt movement so all the surface would be equally analyzed. Spectral data was received by using UV (λ = 250-360 nm) and UV-VIS (λ = 235-800 nm) spectrometers that were chosen as most suitable for current task.

4. Qualitative spectral analysis

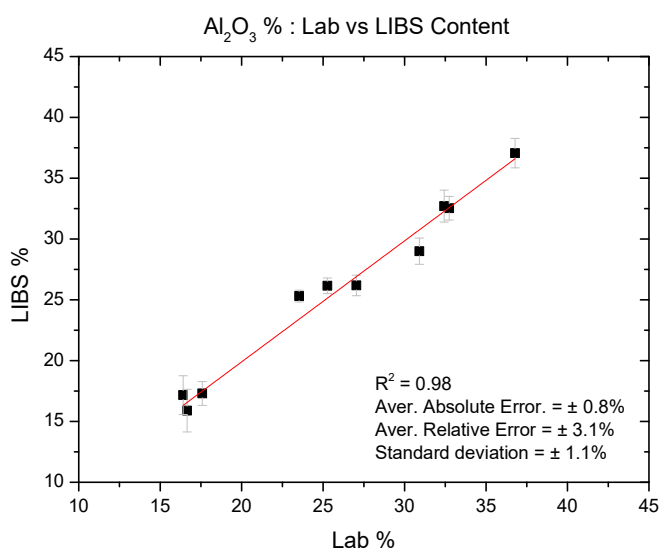


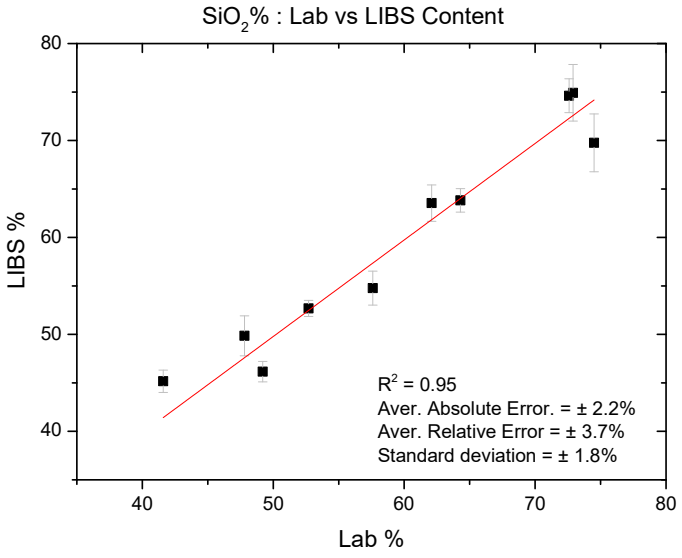


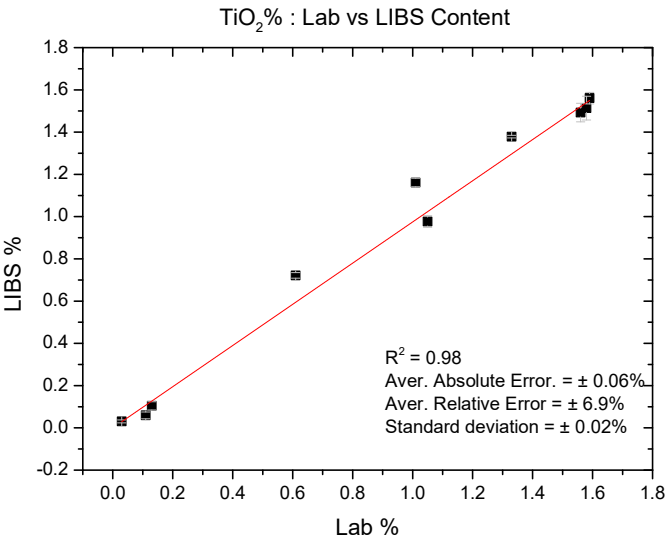
As can be seen from UV and UV-Visible range spectra, well defined lines of: Si, Ti, Al, Ca, Na and K can be clearly detected.

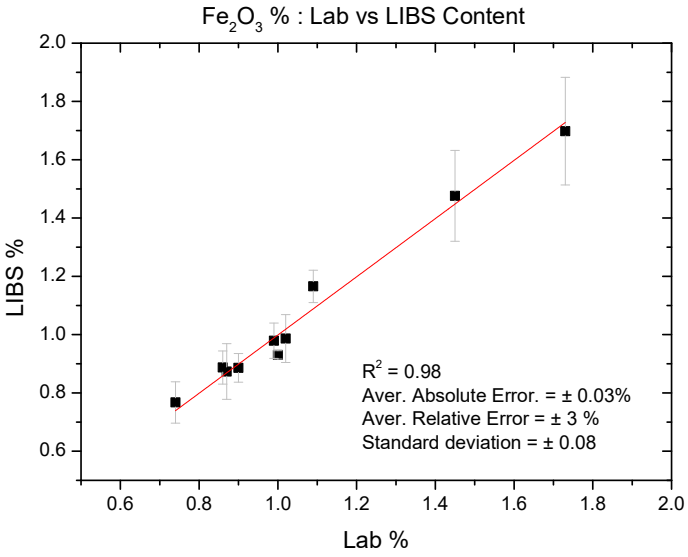
5. Quantitative analysis.

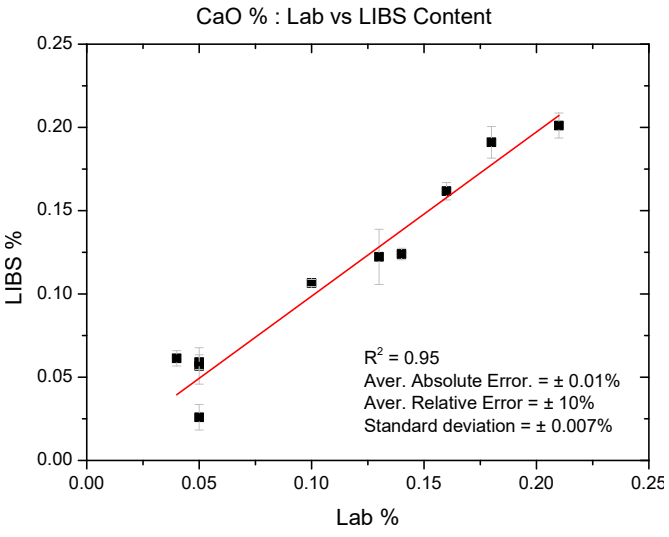
Using XRF data of clay samples calibration curves of LIBS measurement were developed. Vertical lines represent concentration deviation inside each sample.

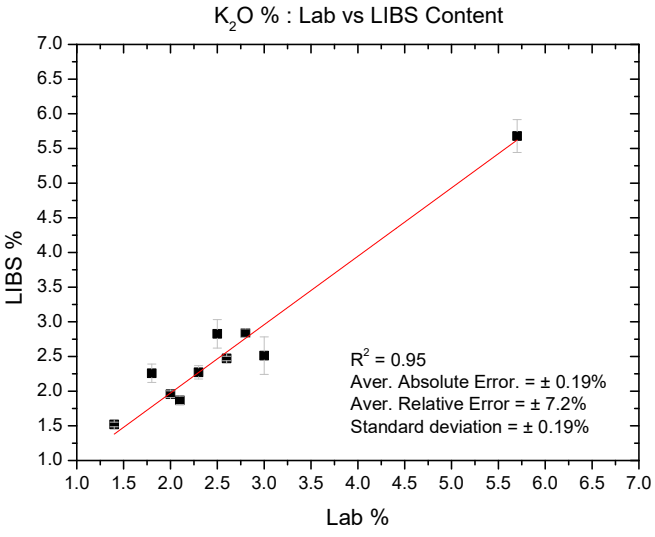


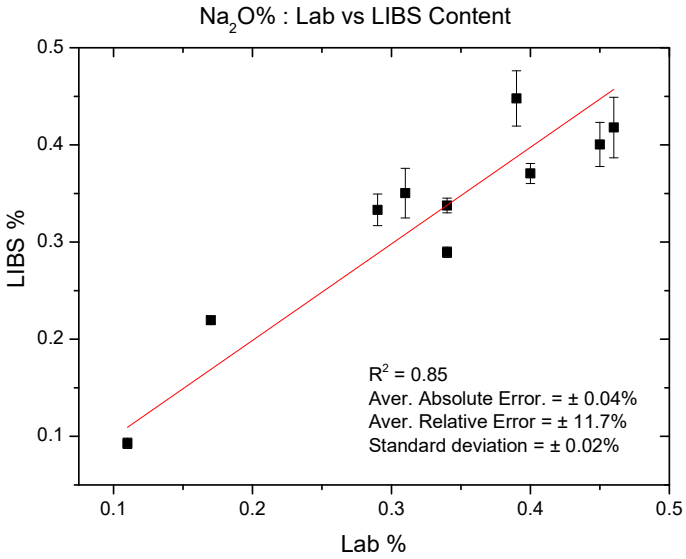












Calibration results show good linearity between XRF and LIBS analysis, thus enabling possibility of on-line, real time LIBS analysis.

Estimated accuracy calculation according to 10 clay samples is described in following table:

Accuracy summary table				
Analyzed Element		Average Error		
	Linearity R^2	Absolute ± %	Relative ± %	Standard Deviation ± %
Al_2O_3	0.98	0.8	3.1	1.1
Fe_2O_3	0.98	0.03	3	0.08
TiO_2	0.98	0.06	6.9	0.02
CaO	0.95	0.01	10	0.007
SiO_2	0.95	2.2	3.7	1.8
Na_2O	0.85	0.04	11.7	0.02
K_2O	0.95	0.19	7.2	0.19

6. Conclusions:

- Good calibration curves between XRF laboratory data and LIBS spectral analysis for all required elements shows high linearity and low error, thus on-line, real time LIBS measurement is applicable.

- Relative errors are around XRF levels and will decrease even further with additional samples.