Development of Liquid Sampling and Correlation Method for Laser-induced Breakdown Spectroscopy

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Quick View

What is LIBS:

-Elemental analysis based on the emission from a laser-induced plasma.

How to LIBS:

-Focus sufficiently high-powered laser beam onto your sample, collect emissions from the laser-induced plasma and analyze the spectrum.

Why use LIBS:

-Low sample consumption
-Easy sample preparation
-Remote, in-situ and multi-element analysis capability
-Simple, rapid, and low-cost analysis
-Wide application variety

Challenges of LIBS and strategy:

*-Serious signal fluctuation=>Normalization=>**this work!!***
*-Intense continuum emission interference.=>Gate-delay detection system or plasma temperature correction=>**this work!!***
-Complicated spectrum=>PCA, PLS and other algebraic methods.
*-Poor LOD and reproducibility=>correlation methods and FI preconcentration =>**this work!!***

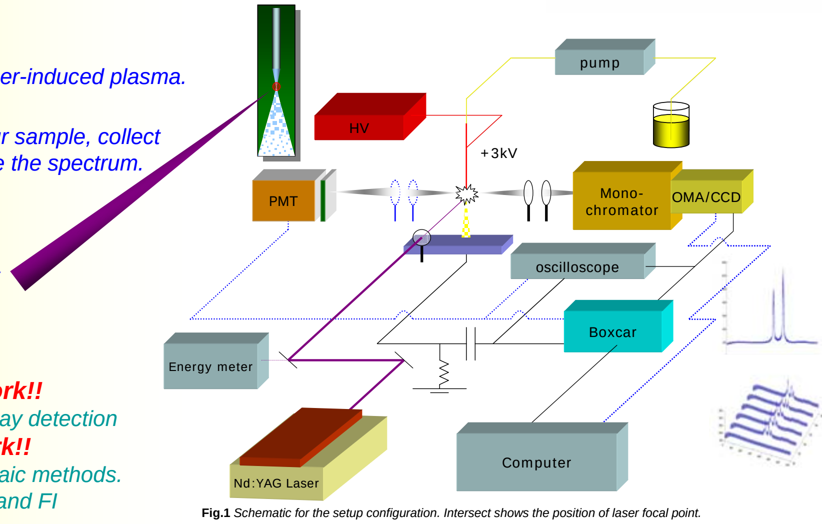

Fig.1 *Schematic for the setup configuration. Intersect shows the position of laser focal point.*

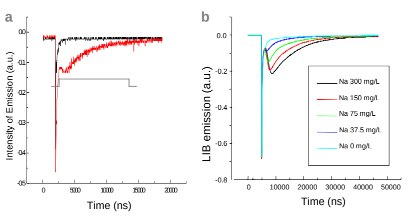

Fig.2 *(a) Single-shot time dependence of LIB spectra for a 100 mg/l Na solution (---) and air (---). The gate is set at 14 μs in width and delayed by 900 ns relative to the trigger of the laser pulse. The spectrum within the gate is integrated to obtain the intensity of LIB emission. (b) Illustration of emission temporal profile changing with increasing [Na].*

Na (mg/l)	K (mg/l)	Al (mg/l)	Method	Group
0.63±0.02 (0.3pg)	1.15 ±0.04 (0.5pg)	43±5 (21pg)	Electro-spray	This work
0.23 (0.4pg)	1.5 (2.6pg)		Liquid Jet	Cheung & Yeung
0.014 (14pg)	1.2 (1200pg)	20 (20,000pg)	Bulk Liquid	Cremers & Radziemski
2.2 (440ng)		5.2 (1000ng)	Isolated Droplet	Crouch & Archontaki

Table.I *Comparison of detection limits for Na, K, and Al in LIBS*

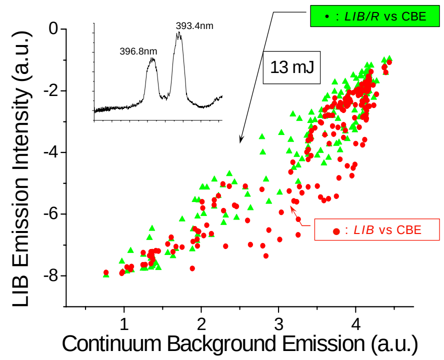

Fig.9 *The correlation plot of LIB emission of Ca versus continuum background emission obtained at the laser energy of 13 mJ. (▲) denotes the LIB emission without plasma temperature correction. (●) denotes the LIB emission normalized by an area ratio of two emission lines. These two emission spectra acquired by an OMA detector are displayed in inset.*

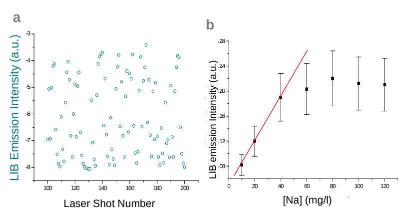

Fig.3 *(a) Intensities of single-shot Na LIB emission for 200 laser shots. Laser pulse is at 355 nm with energy about 13 mJ. (b) Calibration curve of Na at different concentrations without correlation treatment. Each data point of LIB emission intensity is averaged over 200 pulses with an error bar of one standard deviation. Laser pulse is at 355nm with energy about 13 mJ.*

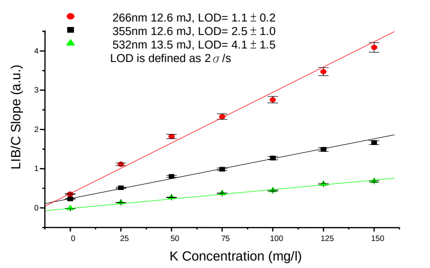

Fig.6 *Calibration curves of concentrations for the K sample at different pulse wavelengths and energies. (●) Laser pulse at 266 nm and 12.6 mJ, LOD=1.1 mg/l; (■) 355 nm and 12.6 mJ, LOD=2.5 mg/l, and (▲) 532 nm and 13.5 mJ, LOD= 4.1 mg/l.*

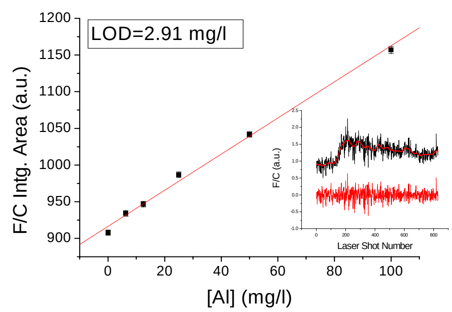

Fig.10 *Calibration curve of Al at different concentrations with FI on-line preconcentration column. LOD is one order reduced compared to previous work (Table I). Intersect shows the E/C ratio variation from shot to shot. The ratio goes up as the eluate comes out and a long tailing is observed due to the longitudinal diffusion and column memory effect.*

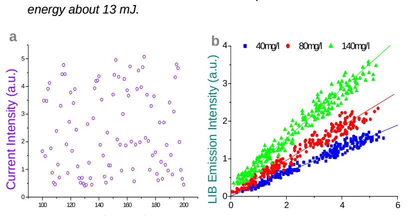

Fig.4 *(a) Single-shot current intensities collected from the laser-induced plasma for 200 data points. Laser pulse is at 355nm with energy about 13 mJ (b) Correlation plot of Na LIB emission vs. corresponding current intensity obtained at the [Na] = 40 (■), 80 (●) and 140 (▲) mg/l, respectively.*

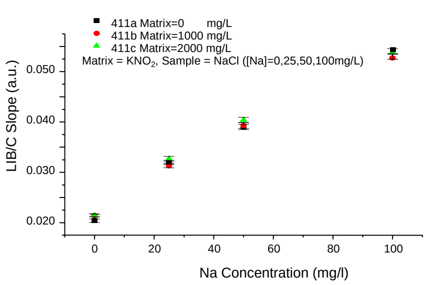

Fig.7 *Calibration curves of NaCl solution with a concentration of 0 (■), 1000 (●) and 2000 (▲) mg/l KCl added as matrix.* ***The method is tolerant of salt matrix!!***

Achievements~

-First combination of LIBS with electrospray for liquid analyses
-First FI-LIBS for on-line preconcentration
-Successful normalization of LIB emission fluctuation with corresponding current signal
-Successful correction of plasma temperature effect with two-line ratio.
-Improvement in LOD and tolerance of salt matrix

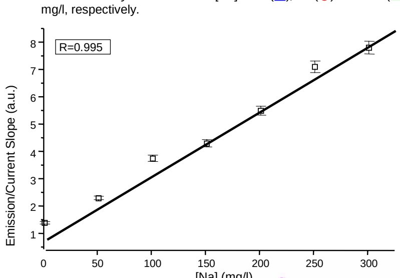

Fig.5 *Calibration curve of Na at different concentrations with correlation treatment. E/C slope is obtained from the correlation plot in Fig 4b.* ***Compared with Fig.3b, LOD and dynamic range is greatly improved!!***

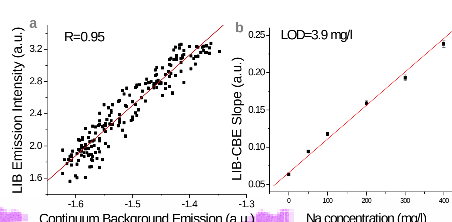

Fig.8 *(a) Correlation plot of Na LIB emission vs. corresponding continuum background intensity; (b) Calibration curve of Na at different concentrations with continuum background emission correlation treatment.*

Publications

Title	Author	Source
Laser-induced Breakdown Spectroscopy Incorporated with Flow-injection System in Analysis of Liquid Droplets	J.S Huang, H. T. Liu and K.C. Lin	**2006**, submitted
Laser-induced Breakdown Spectroscopy of Liquid Droplets: Correlation Analysis with Plasma-induced Current versus Continuum Background	J.S Huang, and K.C. Lin	*J. Anal. and At. Spectrom.* **2005,** *20*, 53-59.
Matrix Effect on Emission/Current Correlated Analysis in Laser-induced Breakdown Spectroscopy of Liquid Droplets	J.S. Huang, C.B. Ke and K.C. Lin	*Spectrochim. Acta* **2004,** *59B,* 321-326.
The correlation between ion production and emission intensity in the laser-induced breakdown spectroscopy of liquid droplets.	J.S. Huang, C.B. Ke, L.S. Huang and K.C. Lin	*Spectrochim. Acta* **2002**, *57B*, 35-48
Flow-injection inductively coupled plasma mass spectrometer incorporated with an ultrasonic nebulizer-membrane dryer: Application to trace lead detection in aqueous solution and seawater.	S.H. Ke, L.S. Huang, J.S. Huang and K.C. Lin	*Applied Spectroscopy* **2001**, *55*, 604-610

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