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Analysis of liquids using LIBS acoustic levitation in different stages of CW laser drying

P. Veis [1,*], I. Urbina[1], S. Shetty[1], M. Veis[1] and S. Atikukke[2]

[1] Dept. of Experimental Physics, FMPI, Comenius Univ., Mlynská dolina F2, 842 48 Bratislava, Slovakia
[2] Dept. of Astronomy, Physics of the Earth, and Meteorology, FMPI, Comenius Univ., Mlynská dolina F2, 842 48, Bratislava, Slovakia

** e-mail: pavel.veis@fmph.uniba.sk*

Introduction:

Elemental and trace element analysis in liquid samples using Laser-Induced Breakdown Spectroscopy (LIBS) has gained increasing attention due to its capability for rapid and multi-element detection; however, its application to liquids remains challenging because of complex laser–liquid interaction processes and strong quenching of the laser-induced plasma by water molecules and their radicals, which result in reduced signal intensity and poor reproducibility. To overcome these limitations, several modified LIBS techniques have been explored, among which LIBS assisted by acoustic levitation (LIBS-AL) combined with continuous-wave (CW) laser drying has emerged as a promising approach for preconcentrating isolated droplets prior to spectroscopic analysis while minimizing contamination from container walls. Recent studies have demonstrated the successful use of LIBS-AL for quantitative analysis through calibration curve methods, including the determination of difficult elements such as boron in water samples and calibration-free quantification of mineral waters, as well as for elemental analysis of plasma-activated water samples where contamination can occur during production. In this work, the CW laser drying process is investigated as a function of time in order to optimize the LIBS-AL technique by examining the evolution of spectral quality and calibration-free elemental quantification in a water-based matrix at different stages of droplet preconcentration and drying, with the aim of improving the reliability and sensitivity of LIBS analysis of liquid samples.

Experimental Set-up:

A diagram of the experimental setup is shown in Figure 1. Plasma was generated by an Nd:YAG laser operating at 1064 nm, with a pulse width of 7 ns and a maximum energy of 330 mJ. The laser pulse was focused onto the sample using a 50 mm focal length plano-convex lens. The plasma was collected using a set of lenses and directed to the spectrometer via an optical fiber cable (OFC) with a numerical aperture of 0.22 and a core diameter of 600 µm. An echelle-type spectrometer (ME5000, Andor Technology, resolution $\lambda/\Delta\lambda$ = 4000, range 230–850 nm) equipped with an ICCD detector (iStar DH743, Andor Technology, Δt = 5 ns) enabled the study at different delay and gate times.

The acoustic levitator was designed and constructed using two sets of ultrasonic transducers operating at 40 kHz arranged in mirror symmetry to create an acoustic standing wave. Droplets, when carefully suspended within the levitator, settle in low-pressure regions located at the nodes of the acoustic standing wave. A continuous wave IR laser (Thorlabs L1550G1 with a maximum power of 2.5W and λ = 1550 nm) was used to accelerate droplet evaporation, thereby preconcentrating the sample. To ensure consistent preconcentration, the droplet volume is monitored using two Raspberry Pi HQ cameras (12 MP, Sony IMX477 sensor) with adjustable lenses (Arducam 8-50mm C-Mount Zoom Lens) positioned 90° apart. A Python program calculates the droplet volume for each frame in real-time, enabling continuous monitoring throughout evaporation and ensuring uniform preconcentration before laser ablation in every trial

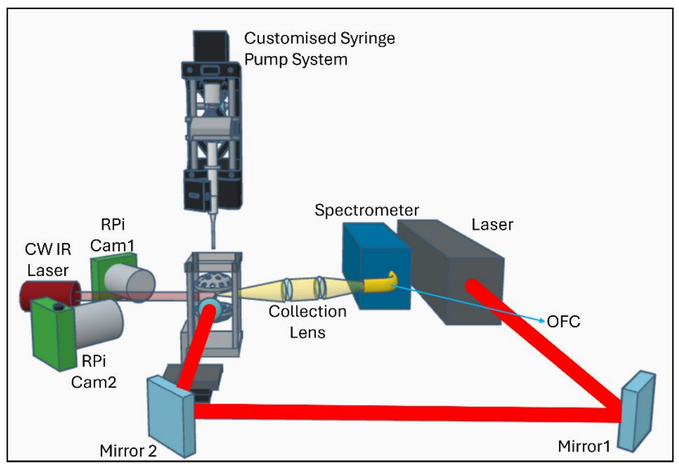

Figure 1. Experimental setup for LIBS-AL.

Parameters and Sample: The experiments was carried out in standard condition of temperature and pressure. In order to obtain high intensity signal to evaluate the thermodynamics parameters, it was prepared an aqueous solutions of Ca and Ni highly concentrated at 2169.46 and 2191.64mg/L to reach a percentual molar fraction of 59.18 and 40.81 respectively. Three preconcentration factor (PF) were considerer: 1(non-PF), 25 and 50. For each PF were recorded the spectrum of three droplets at two different pair of delay and gate time (DT/GT): 300/300ns and 1000/1000ns.

Analysis:

1. **The spectra were captured under two configurations, with gate delay and gate width settings of 300 ns and 1000 ns, respectively.**
2. **The amplifier gain was set to a constant of 180.**
3. **Three trials were averaged to improve the SNR and reduce the poor reproducibility of the LIBS-AL system.**

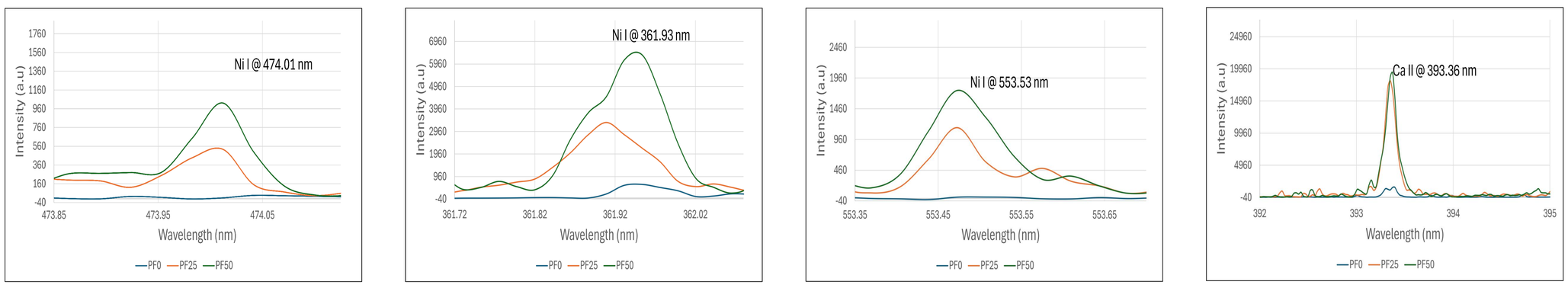

Figure 2. Variation of spectral peak intensity with increasing preconcentration factor (PF) in the LIBS-AL system.

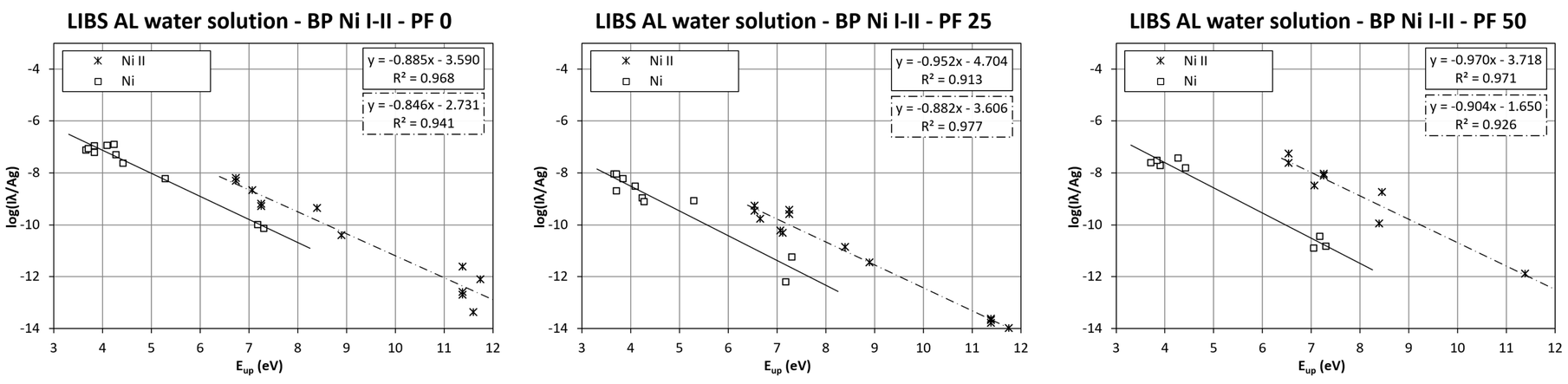

Figure 3. BP of Ni I-II from LIBS-AL water solution spectra for the delay and gate time 300 ns (without preconcentration and for all PF).

Te [eV]	PF 0	PF 25	pf 50
BP Ni I	1.129	1.051	1.031
BP Ni II	1.182	1.133	1.107

Table 1. Electrom temperature T_e evaluated from SB plots for different PF factors.

Results:

Preconcentrating the droplets enhances the lines intensity (see Fig.2) and SNR. Resonant lines like Ca II @ 393 nm (Fig. 2 right) are self-absorbed and were not taken to consideration. The LIBS spectra observed at the delay 1000 ns were very weak, we did the BP analysis only for the delay of 300 ns (without preconcentration and for all PF) – see Fig. 3. The obtained temperature T_e slightly decreases with incearsing PF for booth species Ni I and Ni II (see Tab. 1). The ion abundance of Ni II slightly increases with increasing PF.

References:

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