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Quantitative Elemental Analysis In Agriculture Drainage Water Using Laser Induced Breakdown Spectroscopy

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ABSTRACT

Application of laser induced breakdown spectroscopy (LIBS) in the qualitative and quantitative analysis of trace elements in agriculture drainage water has been considered. In the present work we are studying the effect of presence of increasing number of different salts on the limit of detection (LOD) in agriculture drainage water.

Key Words: LIBS, toxin elements, drainage water.

INTRODUCTION

An official strategy of the Ministry of public works and Water Resources is to use drainage water as one of the water resources for the supply of water to newly reclaimed lands.
On the other hand the drainage municipal and industrial wastes sometimes pollute waters in certain locations. In addition, the excessive use of fertilizers may lead to water contamination by some components such as phosphate, nitrate, and ammonia.
LIBS is a well-established analytical technique (4,5). It can briefly be described as an elemental analysis based on emission from plasma generated by focusing a laser beam on a sample.
LIBS was applied for experiments on liquids with different types of configurations including plasma formation on the surface (9,10), on the bulk liquid (1,11-14), on droplets (15,16), and on liquid jets (17-22). Experimentally there were many problems to overcome in performing LIBS in liquids generally and in water specially.
In general it was not possible to collect atomic line spectra from breakdown plasma originating in the bulk solution. This was mainly because of in-bulk generation of the plasma carries the penalty that plasma light observation times are extremely short, usually of the order 1 μs or less, Knopp et al. (14).
The major disadvantage of in-bulk analysis is the severely reduced plasma emission intensity in comparison with results obtained at the liquid's surface (9,10).
Different geometrical arrangements have been suggested to avoid splashing and consequent opacity of the light collection optical components in the vicinity of the produced plasma.
Generally, time-resolved LIBS has been used to avoid the intense initial continuum emission and to improve the SNR by gating off the early stages of the plasma emission. A suitable choice of the time delays in detecting the emission spectra allows selective assignment of the resolved line emission to different elements.

EXPERIMENTAL SET UP

The plasma formation was attained with the aid of a Q-switched Nd:YAG laser (Continuum Surelite I - Nd :YAG) operating at 1064 nm (pulse duration of 7 ns). The laser beam with pulse energy of 180 mJ was transported by two reflective dichroic mirrors and focused on the water surface by a 10 cm-focal length quartz lens to generate the plasma. For agriculture drainage water quantitative elemental determination, the laser direction was tilted at an angle of 30^o to the water surface to avoid water splashing. This was done using the dichroic mirror at 45^o with a reflection of about 100 % for the 1064 nm. To improve data reproducibility, a low laser pulse repetition rate of 0.2 Hz was used to get rid off any shockwaves produced ripples on the water surface. A one

meter length fused-silica optical fiber mounted on a micro *xyz*-translation stage is used to collect the emission light from the plasma plume and feed it to an Echelle spectrometer (Mechelle 7500, Multichannel instruments, Sweden). The Echelle grating spectrometers designed for operation in high orders and high angles of incidence and diffraction, can provide high resolution in a more compact size and cover a much wider spectral range than conventional grating spectrometers (24). The Mechelle 7500 provides a constant spectral resolution (CSR) of 7500 corresponding to 4 pixels FWHM over a wavelength range 200 – 1000 nm displayable in a single spectrum. A gateable, intensified CCD camera, (DiCAM-Pro 12 bit Intensified Imaging) coupled to the spectrometer was used for detection of the dispersed light. To avoid electronic interference and jitters, the intensifier high voltage was triggered optically. Echelle spectra display, control, processing and analysis were done using both Mechelle software (Multichannel instruments, Stockholm, Sweden) and GRAMS/32 version 5.1 Spectroscopic Data Analysis Software (Galactic Industries, Salem, NH, USA).

RESULTS AND DISCUSSION

1. Optimizing the Experimental Conditions.

Using a classical LIBS configuration, however, as used for LIBS on solids with the laser beam perpendicular to the surface leads to splashing in the case of liquids. Splashing results in covering the focusing optics with droplets and, therefore, prevents further use of this technique. This can easily be explained by the fact that the plasma expansion at atmospheric pressure is directed perpendicularly to the surface. Thus, a tilted configuration (as described in the experimental setup section) can minimize this phenomenon. Another important effect is the perturbation that takes place at the water surface following each laser pulse. By using a low laser frequency of 0.2 Hz, it was shown that measurements were more reproducible. Moreover, enhancement of the data reproducibility can be achieved by accumulation of consecutively measured spectra. It was previously found that the lowest value of relative standard deviation RSD of the H_α spectral line intensity does not change significantly by increasing the number of shots more than 50; accumulating fifty spectra in each case carried out therefore all subsequent measurements. Mechelle software (Multichannel instruments, Stockholm, Sweden), is used to acquisition, record and to sum separately these 50 spectra; their average value then represents a point on the calibration curve for each element at a specific concentration as will be explained below.

Another important parameter in LIBS is time resolution as already shown for solids and gases analysis (25-29) . For improving the sensitivity in any LIBS experiment. In doing so, a three dimensional display of typical laser induced plasma emission spectra captured at different delay times t_d in figure 1. The figure reveals that a time delay of 1500 ns gives an optimum S/B ratio where the emission coming from the plasma continuum does not interfere with the analytical lines of the elements of interest. A shorter time delay leads to strong background emission and a longer delay does not improve the S/B ratio. This is because, as the plasma cools down, the intensity of the ionic lines decreases, due to recombination, after reaching its maximum, while neutral lines show up for longer time.

2. Trace Determination in Water, Calibration Curves and Limit of Detection

The optimized experimental parameters for laser pulse energy, gate delay time, gate width, number of accumulated single shot spectra, and geometrical arrangements are fixed for all experimental data acquisition procedures. The panoramic Echelle spectra in the spectral range 200 – 1000 nm made it possible, for the first time, to normalize the whole spectral range from the UV-emission of F2- lines at 239.27 to the far visible emission of the Ca-lines at 393.36 and 396.83 nm by the emission of the strong and well resolved H_α line at 665.27 nm applying the so-called internal standardization method. This method is based on the following principle: a number of reference samples are prepared, all having a similar and known elemental composition. Usually, in such a set of samples, a suitable element dominates (internal standard) and defines the sample

properties in view of the 'matrix effects'. It is, therefore, expected that plasma emission be less affected from sample to sample.
In a LIBS experiment, the ratio of the line intensity of a trace element to the emission line of the internal standard is measured and plotted as a function of the known concentration ratios in the reference samples.

A series of measurements between 40 ppm and the detection limits of elements Ca, Fe, Cu were performed for the agriculture drainage water matrix. Figure 1 represents linear calibration curves for Fe (373.71) .

Before performing the calibration curves for the above mentioned eight elements, the agriculture drainage water sample is analyzed for these trace elements using atomic Absorption Spectroscopy AAS technique and their concentration values is shown in table 2. The agriculture drainage water sample is taken from a specific agriculture field located beside a steel factory in the south of Cairo delta. Then the elements concentrations given in table 2 are taken into consideration as a background when preparing the selected elemental sample solution.

Table 2: The agriculture drainage water sample is taken from a specific agriculture field located beside a steel factory in the south of Cairo delta and analysis using AAS.

Element	Fe		Ca		Cu	
Concentration(ppm)	AAS	LIBS	AAS	LIBS	AAS	LIBS
	0.85	1.27	27.89	30.2	0.1	0.1
	0.53	0.34	16.58	17.5	0.09	0.03
	0.54	0.34	14.84	15.28	0.02	0.05
	0.33	0.29	19.52	17	0.15	0.016
	0.20	0.25	31.04	17.39	0.02	0.033
	2.22	2.1	34.75	22.73	0.03	0.032
	0.4	0.7	30.83	29.52	0.12	0.035

The limit of detection was calculated from the formula (30).

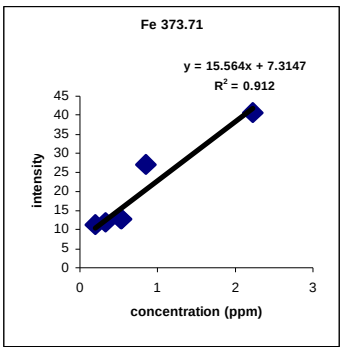
$$LOD = 3\sigma / s$$

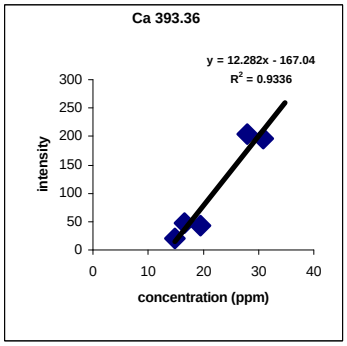
Where σ is the standard deviation of the background and s is the calibration slope.
A comparison of the detection limits found in this work and those reported in the literature are given in table 3.

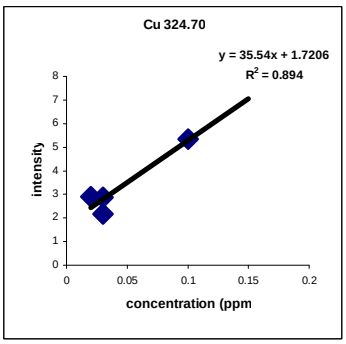
Table 3: The LOD obtained for the measured elements in agriculture drainage water.

Element	Wavelength (nm)	Detection limit (ppm) (this work)	Detection limit (ppm) (literature)
Fe	373.71	0.65	30 (23)
Ca	393.36	0.15	0.3 (23)
Cu	327.70	0.10	7 (23)

Figure 2 represents linear calibration curves for Ca (393.36 nm), Figure 3 represents linear calibration curves for Cu (324.70nm),







(Figure 1) : linear calibration curves for Fe (373.71). (Figure 2) : linear calibration curves for Ca (393.36 nm) (Figure 3) : linear calibration curves for Cu (324.70nm),

CONCLUSION

Exploiting the LIBS technique with an Echelle spectrometer facilitated the simultaneous multi-channel analysis in a wide spectral range which saved time and efforts. Moreover, the use of an Echelle spectrometer coupled to an intensified CCD camera advantageously provided panoramic spectra extending over the wavelength range 200 to 1000 nm. This facilitates the normalization of the analytes line spectra by the strong and well-resolved H_α line that improves the reliability and reproducibility of the LIBS technique compared to the conventional narrow range grating spectrometer LIBS experiments. Simultaneous optimization of the experimental parameters over such wide spectral range and the use of the same delay time, gate width, laser pulse energy, number of accumulated single-shot spectra and geometrical arrangements throughout all the measurements of the selected eight elements. Elemental line intensities were monitored in the LIBS as a function of the analyte concentration to determine both the corresponding calibration curves and the detection limits. The obtained results are compared with previously obtained results by others who performed their experiments using conventional spectrometers and re-optimizing the experimental parameters for each element.

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