



Studying the Effect of Delay Time on Intensity of Laser Induced Breakdown Spectroscopy Technique Using Mas Cement Sample

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Abstract: Cement is a soft bond material that hardens and acquires its mechanical properties in the presence of water; it is an industrial product that has many uses, including its use in construction, roads, bridges, and others. It is important in our life to know its components. In this study, a laser-induced breakdown spectrometer was used to detect the elements present in a sample of Sudanese mas cement. In this technique, a high-energy laser is applied to the surface of the sample, ionizing the atoms of the material and then evaporating it, and then producing hot plasma on the surface of the sample, and this plasma is analyzed with a spectrometer, and thus the components of the sample can be known. The laser source used in this study is (Nd: YAG). Delay times are one of the factors affecting the intensity of the libs signal, To determine the relationship between laser delay times and LIBS signal intensity, a mas cement sample was irradiated with 32mj laser pulse energy and (200, 300, 400, 500, 600, 700, 800, 1000, 2000) nanoseconds delay times. We observed a decrease in the LIBS signal with increasing laser delay times. For quantitative analysis, the plasma temperature and electron density at (300, 500 and 700) laser delay times at 32mj laser pulse energy were calculated using standard equations and well-resolved spectral lines for Ca in the region of (422.67, 430.25 and 430.774) nm. It has been observed that when the delay time increases, the plasma temperature decreases and the electron density decrease. It was found that the density of electrons is directly proportional to the temperature of the plasma.

Keywords: Mas Cement, Laser Induced Breakdown Spectroscopy, Laser Delay Time, Plasma Temperature, Electron Density

1. Introduction

Laser-induced breakdown spectroscopy is an atomic emission spectroscopic technique in which high-energy laser light is used [1, 2]. When the focused laser pulses are directed at the sample, it leads to the formation of plasma of an ionized matter [2-4]. The plasma emitted from the sample provides spectral signatures of the sample's compounds for all states of matter: solid, liquid and gaseous [5, 6]. The libs system consists of a laser source, a sample, a spectrometer and a detector, and it may need a control and capture spectrum system [5, 7]. The emission spectrum of the sample contains the spectra of the various elements that make up it. The intensity of the peaks will be a

function of the ionization energy of each element and the amount of ionized element [8]. The libs system has been used to identify detect and analyze materials in many applications [5, 9]. Such as Determine the constituent elements of cement powder because it is has an important role in industry and construction [10, 11]. There are many types of cement in Sudan, and in this study, mas cement was used.

Libs technology enables quantitative and qualitative analysis of a large number of elements [12].

Knowing the plasma temperature is important to know the processes of dissociation, dissolution and excitation, and the Boltzmann diagram method is the most widely used method for calculating the plasma temperature [13].

The LIBS technique has many advantages that distinguish it from other techniques in spectroscopy, as it is characterized by its quick preparation, high sensitivity, real-time analysis and multi-element analysis [14, 15].

2. Materials and Methods

Mas cement Sample pellets of 20mm diameter were produced by pressing 3.0g of each cement using hydraulic pressure. For pressing of pellets uniform load at pressure of 12 tons was applied for 2.0 minutes duration. Later for sample analysis, the pellet samples were placed in the sample holder. LIBS setup are shown in Figure 1. The main apparatus in LIBS is the Laser, which is used to induce the plasma on the surface of the sample. In this system a pulsed Nd: YAG laser (266 nm) is used. A Q-switch is used to generate a pulsed laser. The laser beam falls on the sample (which is on an X-Y translation stage) [16].

A focusing lens is used to obtain an increase in radiation to generate the plasma, an optical fiber is used to collect the optical emission from the plasma, a spectrometer is used to obtain the spectral signature, and it detects the light emitted by an ICCD camera and finally processes the spectrum using a computer [2].

The Mas cement sample was irradiated by 32mj laser pulse energy and (200, 300, 400, 500, 600, 700, 800, 1000, and 2000) ns delay times, and the Spectra of these samples were recorded as shown in figures 2 to 10.

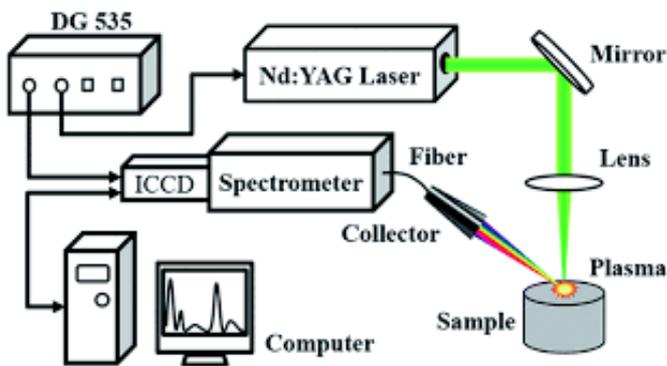


Figure 1. Schematic diagram of the LIBS setup.

3. Results and Discussions

3.1. Dependence of LIBS on Delay Time

Delay time, gate time, laser type and laser energy are the main parameters in the LIBS experiment [17].

To find out the relationship between the delay time and the LIBS signal intensity, the plasma emission signals were recorded at different delay times (200, 300, 400, 500, 600, 700, 800, 1000 and 2000) ns. Regarding the laser pulse, a laser pulse of 32 mj was used and the spectra were recorded in the figures 2 to 10. The recorded spectra were analyzed and the spectral lines were determined that showed different elements such as (Ca, Fe, Ti, Zr and k). And then the relationship between the delay times and the intensity of the LIBS signal was clarified in figures 11.

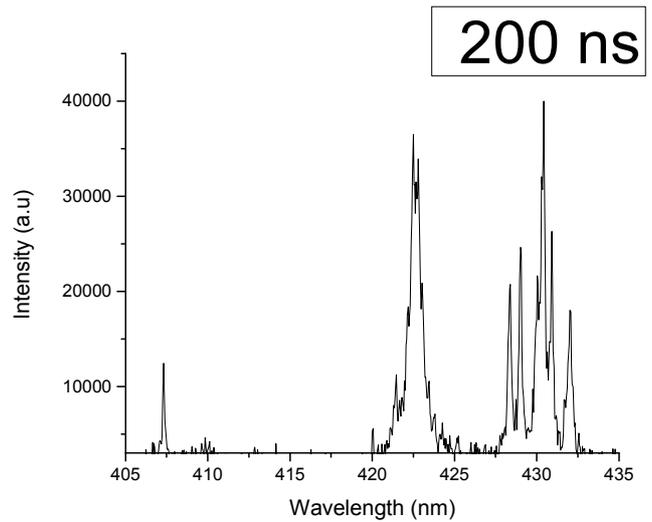


Figure 2. Libs spectra of Mas cement at 200 ns delay time, 32mj laser energy.

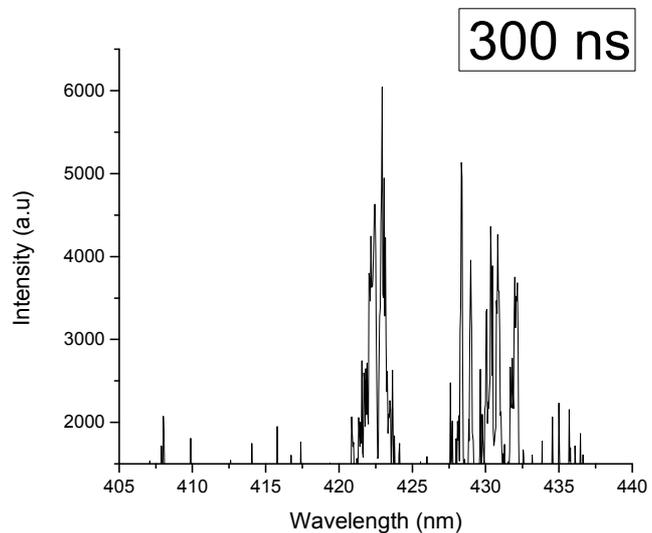


Figure 3. Libs spectra of Mas cement at 300 ns delay time, 32mj laser energy.

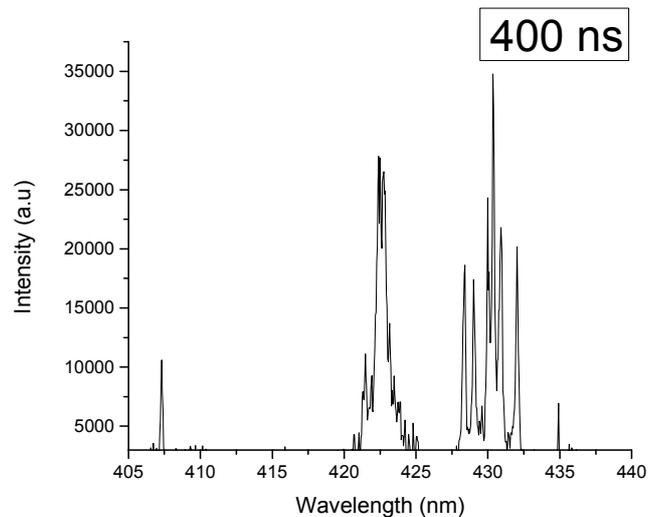


Figure 4. Libs spectra of Mas cement at 400 ns delay time, 32mj laser energy.

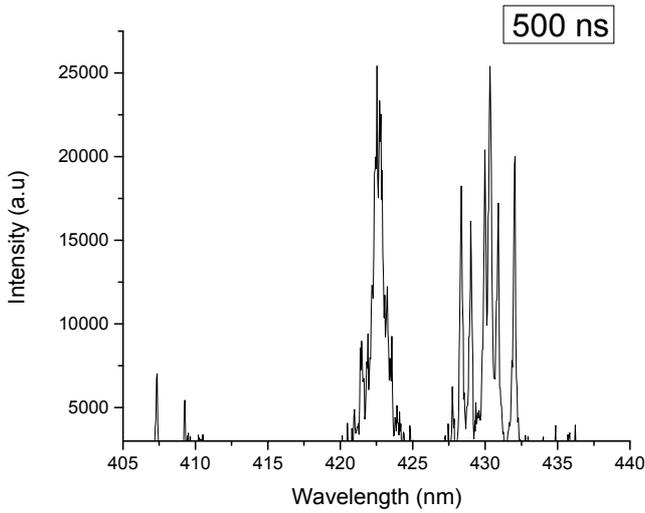


Figure 5. Libs spectra of Mas cement at 500 ns delay time, 32mj laser energy.

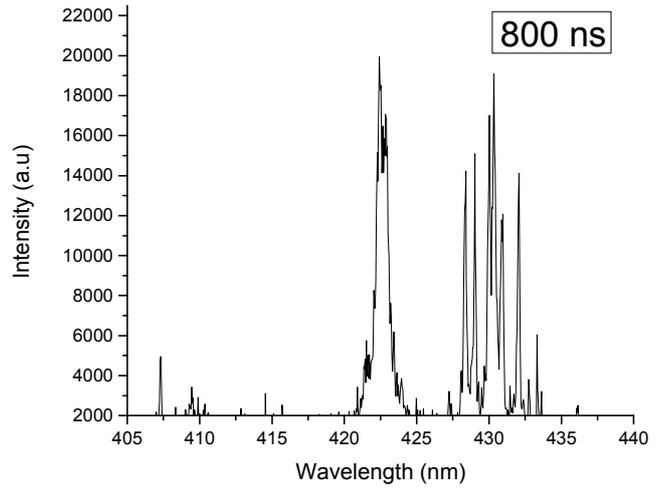


Figure 8. Libs spectra of Mas cement at 800 ns delay time, 32mj laser energy.

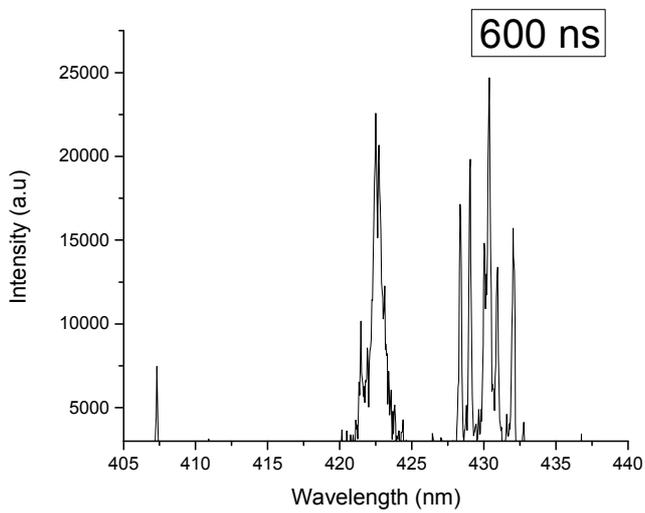


Figure 6. Libs spectra of Mas cement at 600 ns delay time, 32mj laser energy.

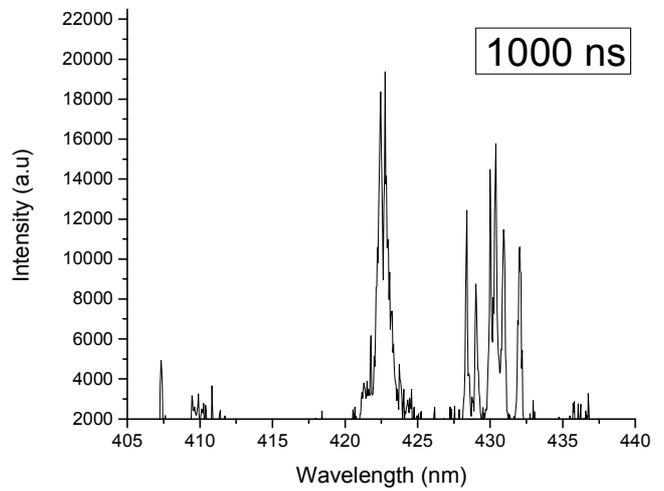


Figure 9. Libs spectra of Mas cement at 1000 ns delay time, 32mj laser energy.

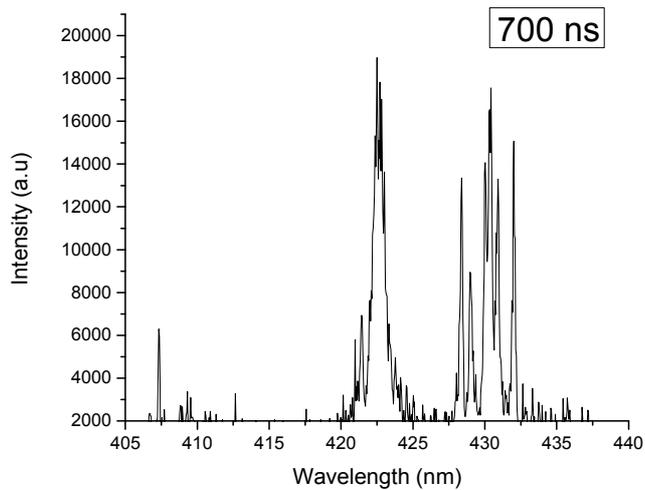


Figure 7. Libs spectra of Mas cement at 700 ns delay time, 32mj laser energy.

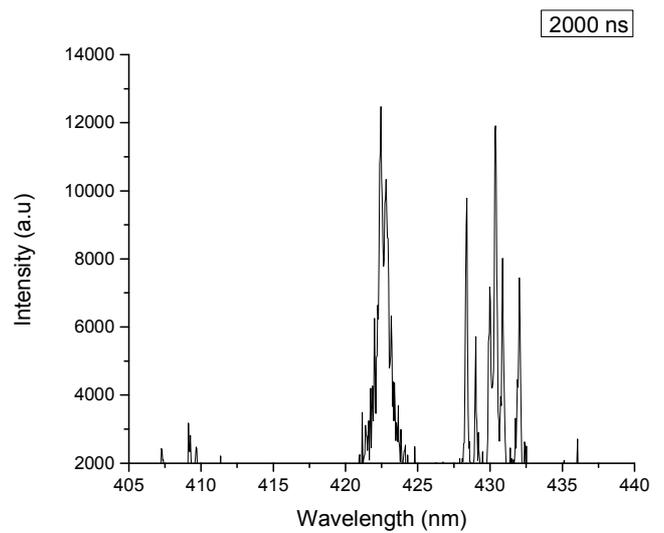


Figure 10. Libs spectra of Mas cement at 2000 ns delay time, 32mj laser energy.

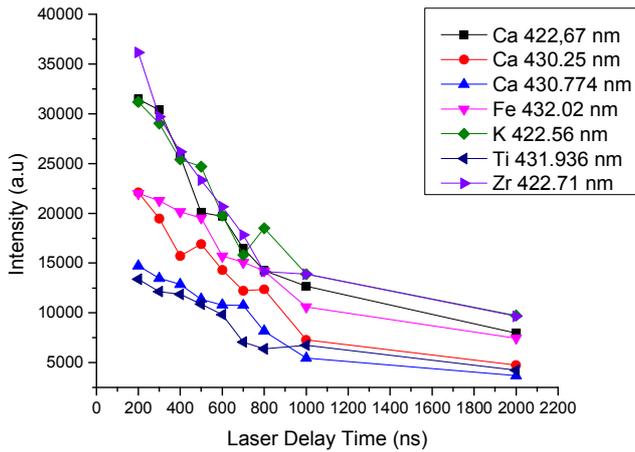


Figure 11. Relationship of delay times and libs signal intensity.

3.2. Quantitative Results: Dependence on Delay Time

3.2.1. Plasma Temperature

To calculate the plasma temperature, the Boltzmann method was used and the equations were solved using the spectral constants of the Ca lines in the (422.65, 430.25, 430.774) nm spectral region obtained from the NIST database. The spectra were obtained at a laser pulse energy of 32 mj. The plasma temperature was calculated at (300, 500 and 700) ns delay time.

Table 1. Ca lines Spectroscopic constants are used in temperature determination.

Wavelength (nm)	$A_{ki} \cdot 10^8$	g_i	g_k	E_i (ev)	E_k (ev)	ΔE
422.67	2.8	1	3	00	2.93	2.93
430.25	1.36	5	5	1.89	4.78	2.88
430.774	1.99	3	1	1.88	4.76	2.877

Table 3. Plasma temperatures and electron densities at different delay times of Ca lines in Mas cement sample.

Delay time (ns)	Plasma temperature (K)	Electron density (cm ⁻³) (Ca at 422.67 nm)	Electron density (cm ⁻³) Ca at 430.25 nm	Electron density (cm ⁻³) Ca at 430.774
300	100250	1.27*10 ¹⁶	1.21*10 ¹⁶	1.2*10 ¹⁶
500	45608	0.86*10 ¹⁶	0.816*10 ¹⁶	0.813*10 ¹⁶
700	45337	0.85*10 ¹⁶	0.813*10 ¹⁶	0.811*10 ¹⁶

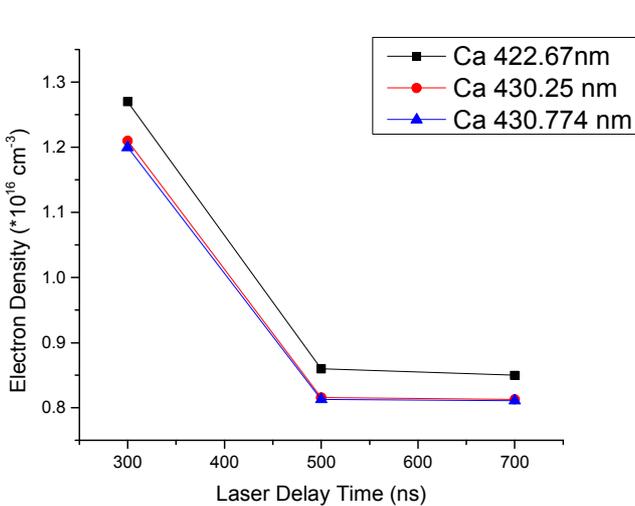


Figure 13. Electron density of Ca emission lines dependent on delay time.

Table 2. Plasma temperature at different delay time.

Laser energy	300ns	500ns	700ns
Plasma temperature	100250K	45608K	45337K

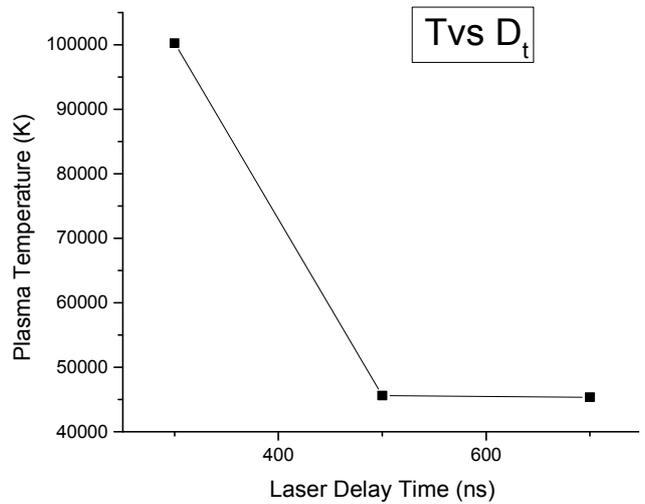


Figure 12. Dependence of plasma temperature on delay times.

3.2.2. Electron Density

In this study, the electron density at the Ca spectral lines was calculated using the equation below

$$N_e = 1.6 * 10^{12} \Delta E^3 T_e^{-\frac{1}{2}}$$

Where, ΔE = the largest observed transition energy T_e = the excitation temperature (K). The table below shows the electron density and the plasma temperature, which was calculated at (300, 500 and 700) ns delay times at 32mj laser pulse energy.

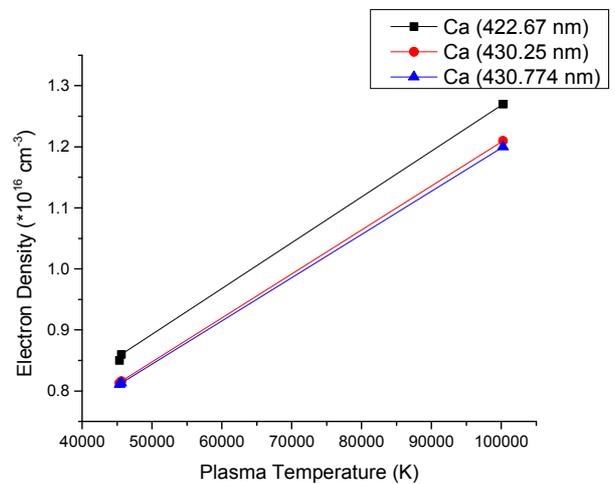


Figure 14. Electron density of Ca emission lines dependent on plasma temperature.

4. Conclusion

From the obtained results one can conclude that:

- 1) A single pulsed Laser Induced Breakdown Spectroscopy (LIBS) technique was used to detect elements in Mas cement sample.
- 2) Elements that were detected are Ca, Zr, K, Fe and Ti.
- 3) An optimum value for time delay was obtained for the LIBS signal which was found to be 200 ns.
- 4) LIBS signal decreases with increase in plasma delay time.
- 5) Increased in delay time that will lead to decreased in plasma temperature.
- 6) The electron density decreased by increased in delay time.
- 7) Electron density increase with increase in plasma temperature.

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