Determination of the Fertility of Southern Iraqi Soil Using Laser - Induced Breakdown Spectroscopy System

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Abstract: In this work, seven soil samples were brought brought to study and analyses the element concentrations from different southern regions of Iraq using laser-induced breakdown spectroscopy (LIBS) technique. It has been documented as an atomic emission spectroscopy (AES) technique. Laser-induced plasma utilized to analyze elements in materials (gases, liquids, and solids). In order to analyze elements in materials (gases, liquids, and solid). The Nd: YAG laser excitation source at 1064 nm with pulse width 9 ns is used to generate power density of $5.5 \times 10^{12} \text{ MW/mm}^2$, with optical spectrum in the range 320-740 nm. From this investigation, the soil sample analysis of the southern cities of Iraqi, it is concluded that the rich soil element of P, Si, Ca elements make the Faw and Nesseriah fit for cultivation.

Keywords: Laser-induced breakdown spectroscopy, LIBS, atomic emission spectroscopy, AES, and soil fertility.

Introduction

Laser-induced breakdown spectroscopy (LIBS) has been developed since the invention of the laser in 1960. The arrival of the laser in May, 1960 opened up new possibilities for microanalysis which were quickly explored [1]. The first instrument that based on laser-induced excitation were launched by “Zeiss, Jarrell-Ash and Jeol ltd” between 1964 to 1967, in which Generalov and other workers published early papers based on laser plasma and spectrochemical analyses during the period from 1970 to 1974 [2]. LIBS were first used to evaluate sodium and potassium in the conversion of charcoal into gas in 1981[3]. In 1986, Cremers and coauthors used LIBS to evaluate the effective factors on metal analyses [4]. Micro-plasma technique has been used to elementally analyze solid, liquid and gas. This procedure used focused laser pulses, which has more advantages than other conventional methods [5]. In 2003, evidence showed a high development in the LIBS technology, which demonstrated a high sensitivity measurement based on visible photon and Ultra Violet (UV) wavelength bands [6].

Laser Induced Breakdown Spectroscopy (LIBS) or Laser Induce Plasma Spectroscopy (LIPS) is method used to determine the composition of investigated samples [7]. The initiation, formation, and decay of the plasma are complex physical and chemical processes. In the LIBS process, a laser beam is focused on a small area of the sample. When the laser energy exceeds the breakdown threshold, the
plasma with high temperature and high density is produced in the portion. The core of plasma is firstly produced by the absorption of the incident laser energy, such as multi photon ionization. The creation of the plasma core induces the rapid growth of plasma through the absorption of the laser light by electrons and the electron impact ionization process in it. After the termination of the laser pulse, the plasma continues expanding because of its high temperature and pressure gradients compared with the ambient conditions. At the same time, the recombination of electrons and ions proceeds due to the collision process and the temperature decreases gradually compared with that in the plasma generation process. Emission signals arise in the plasma cooling period. In the plasma, the ions, atoms, and molecules distributed in the different levels transit from the high energy level to the low energy level, emitting the strong emission spectra. The emission intensity from the atomized species provides the elemental compositions of the materials. The light corresponding to a unique wavelength of each element is emitted from excited atoms in plasma [8].

The LIBS technique can give information about composition of elements. Max Plank is the first person who inspired the concept of LIBS method. He suggested that quantum theory could explain the radiation emitting properties by heated bodies. It is very well stated that the quantum theory has two important hypotheses [9]. The first hypothesis documented that existing of atoms, ions and molecules can be merely in a particular discrete state, which can be characterized by definite and precise amount of energy. It is very well known that all the bonds of molecules are broken during plasma formation, and that will cause free electrons emission. Then it will create a continuous emission, which quickly disappears. The reason of disappearance of continuous emission belongs to capturing of free electrons by atoms and ions. Therefore, it will be absorption and emission energy, which is exactly the same difference in energy amount between the states of atoms, ions and molecules.

Laser-induced breakdown spectroscopy (LIBS) is an analytical technique that allows for the determination of a sample’s elemental composition based on laser ablation followed by atomic, ionic, and molecular emission processes coming from elements transferred into the plasma as a result of laser-induced breakdown. This process illustrated in figure 1 [10, 11, 12].

Figure 1: Life cycle diagram showing of the main events in the LIBS process [13].

One of the major advantages of LIBS is its speed and ability to make a multi-elemental analysis. By using suitable spectrograph equipped with a multi-channel detector, analysis of dozens of elements in a single laser shot may take just a second of time [14]. LIBS are also applicable to the analysis of extremely hard materials that are difficult to digest or dissolve, such as ceramics and semi or super-conductors [15].

There are many applications of LIBS for qualitative and quantitative elemental measurements in a wide range of samples such as various environmental samples, non-metallic solids, liquid samples and gases samples. Among the application of LIBS in biological and medical samples includes calcified tissue materials (e.g. teeth, bones, sea shells), soft tissue materials (e.g. human skin, plant parts like leaves and wood) [16, 17].

LIBS have been used for the materials detection and analysis in various applications, such as steel, wood pre-treatment, bacteria, molds, pollens, proteins, space exploration, and books [18-25].

The X-ray fluorescence XRF technique is a very powerful, portable and easy tool compared to the LIBS technique. The ionizing electromagnetic radiations, which are X and gamma-rays are also...
used to move the electron away from its orbital (energy level) causing ionization. In other words, both X and gamma ray photons are able to eject an electron from its orbit in an atom (“are ionizing radiation”), instigating ionization, which is the process of removing one or more electrons from atoms by the incident radiation leaving behind electrically charged particles (“an electron and a positively charged ion”). X and gamma rays can expel an electron, force it to leave its interior orbital; however, the orbital will be held instigating unstable atom [26].

The aim of this work to employ the high power laser-induced plasma spectroscopic analysis techniques to identify soil elements by determining emission spectra formed as a result of laser interaction with an Iraqi soil sample.

Experimental Setup

The experimental setup as illustrated in figure 2. LIBS experimental arrangements used in this paper are single-pulse configuration, and it was performed under fixed environmental conditions, and consisted of following parts:

1. Pulsed laser source which is used to generate the plasma material.
2. Optic lens or fiber is used to collect light.
3. Spectral analysis system.
4. Computer to control both laser and the detector as well as save the resultant spectra.

The soil samples were used in experimental work to determine the fertility of north Iraqi soil. Nd: YAG laser excitation source (λ=1064 nm) of pulse width and pulse duration of about ~ 9 nm is used. The laser beam is focused by using fused silica quartz lens having a focal length of 10 cm onto the sample placed on a stage with an adjustable height. The optical emission from the plasma is collected and guided to the spectrometer via the optics, to yield information on the material composition. The fiber bundle is positioned at a distance of nearly 1.0-1.5 cm from the plasma, making an angle 45° to the laser beam in order to generate the power density of 5.5 x 1012 MW/mm², with optical spectrum in the range of 320-740nm as shown in figure 3. The soil of Northern region is characterized by its diversified structure including valleys, low land and mountains. The highland soil has a less chance for agriculture and shows signs of water and ice. The soil of valleys and mountain’s low land are suitable for agriculture. Developed chestnut, gray- brown and black soils are available.

A total of seven various depth levels down to 30 cm soil samples were brought from different south regions of Iraq, as shown in figure 4. Samawah, Faw, Najaf, Nesseria, Karbala, Basrah and Babylon. Initially, the soil samples were pressed in the mold of 2 cm diameter followed by heating them in an oven for up to 3 hours. After that, these samples were pressed again and kept in containers and left to dry in places away from surrounding lab effects.
Results and discussion
The results shows there are many emission peaks present in these spectra. These peaks and regions will be shown in more details. In Samawah region, the emission peaks that present show as in fig.4 are Fe I at wavelength 383.422 nm, intensity 0.03 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.7 a.u and 396.846 nm, intensity 0.07 a.u, two peaks of Ca I 422.672 nm, intensity 0.1 a.u and 445.478 nm, intensity 0.12 a.u, N II 499.436 nm, intensity 0.003 a.u, Si II 568.881 nm, intensity 0.06 a.u Na II 588.995 nm, intensity 0.21 a.u, K II 612.027 nm, intensity 0.03 a.u, P II 616.559 nm, intensity 0.18 a.u.

In Faw region, the emission peaks that present show as in fig.5 are Fe I at wavelength 383.422 nm, intensity 0.02 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.27 a.u and 396.846 nm, intensity 0.22 a.u, two peaks of Ca I 422.672 nm, intensity 0.19 a.u and 445.478 nm, intensity 0.07 a.u N II 499.436 nm, intensity 0.06 a.u, Si II 568.881 nm, intensity 0.37 a.u Na II 588.995 nm, intensity 1 a.u, K II 612.027 nm, intensity 0.01 a.u, P II 616.559 nm, intensity 0.04 a.u.

Figure 3: The samples used in the experimental work, Southern region.

Figure 4: LIBS spectra recorded from Samawah soil.
In Najaf region, the emission peaks that present as in fig. 6 are Fe I at wavelength 383.422 nm, intensity 0.1 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.97 a.u and 396.846 nm, intensity 0.82 a.u, two peaks of Ca I 422.672 nm, intensity 0.14 a.u and 445.478 nm, intensity 0.16 a.u, N II 499.436 nm, intensity 0.05 a.u, Si II 568.881 nm, intensity 0.03 a.u, Na II 588.995 nm, intensity 0.27 a.u, K II 612.027 nm, intensity 0.04 a.u, P II 616.559 nm, intensity 0.2 a.u.

Figure 5: LIBS spectra recorded from Faw soil.

Figure 6: LIBS spectra recorded from Najaf soil.
In Nasseria region, the emission peaks that present show as in fig. 7 are Fe I at wavelength 383.422 nm, intensity 0.01 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.22 a.u and 396.846 nm, intensity 0.17 a.u, two peaks of Ca I 422.672 nm, intensity 0.1 a.u and 445.478 nm, intensity 0.14 a.u, N II 499.436 nm, intensity 0.01 a.u, Si II 568.881 nm, intensity 0.17 a.u, Na II 588.995 nm, intensity 1 a.u, K II 612.027 nm, intensity 0.02 a.u, P II 616.559 nm, intensity 0.3 a.u.

In Karbala region, the emission peaks that present show as in fig. 8 are Fe I at wavelength 383.422 nm, intensity 0.008 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.21 a.u and 396.846 nm, intensity 0.2 a.u, two peaks of Ca I 422.672 nm, intensity 0.04 a.u and 445.478 nm, intensity 0.04 a.u, N II 499.436 nm, intensity 0.01 a.u, Si II 568.881 nm, intensity 0.005 a.u, Na II 588.995 nm, intensity 0.13 a.u, K II 612.027 nm, intensity 0.02 a.u, P II 616.559 nm, intensity 0.1 a.u.

Figure 7: LIBS spectra recorded from Nasseria soil.
In Basrah region, the emission peaks that present show as in fig.9 are Fe I at wavelength 383.422 nm, intensity 0.02 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.31 a.u and 396.846 nm, intensity 0.28 a.u, two peaks of Ca I 422.672 nm, intensity 0.3 a.u and 445.478 nm, intensity 0.12 a.u, P I 499.436 nm, intensity 0.01 a.u, Si II 568.881 nm, intensity 0.02 a.u, Na II 588.995 nm, intensity 0.62 a.u, K II 612.027 nm, intensity 0.02 a.u, P II 616.559 nm, intensity 0.22 a.u.

**Figure 8:** LIBS spectra recorded from Karbala soil.

**Figure 9:** LIBS spectra recorded from Basrah soil.
In Babylon region, the emission peaks that present show as in fig. 10 are Fe I at wavelength 383.422 nm, intensity 0.001 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.09 a.u and 396.846 nm, intensity 0.08 a.u, two peaks of Ca I 422.672 nm, intensity 0.01 a.u and 445.478 nm, intensity 0.03 a.u N II 499.436 nm, intensity 0.002 a.u, Si II 568.881 nm, intensity 0.005 a.u Na II 588.995 nm, intensity 0.04 a.u, K II 612.027 nm, intensity 0.005 a.u, P II 616.559 nm, intensity 0.06 a.u.

The emission spectrum analysis for the soil of Samawah city indicates the abundancy of elements Na II, Ca II, Ca I and Si II in compare to the other low abundancy elements of Fe I, K II, N I, and P II. The emission spectrum analysis for the soil of Faw city indicates the abundancy of elements Na II, Ca II, and Si II in compare to the other low abundancy elements of Fe I, K II, Ca I, N I, and P II. The emission spectrum analysis for the soil of Najaf city indicates the abundancy of the elements Na II, Ca II, Ca I, Fe I, and P II in compare to the other low abundancy elements of Ca II, K II, Si II and N I. The emission spectrum analysis for the soil of Nasseria city indicates the abundancy of the elements Na II, Ca II, Si II, Ca I, and P II in compare to the other low abundancy elements of Ca II, K II, N I and Fe I. The emission spectrum analysis for the soil of Karbala city indicates the abundancy of the elements, Ca II, Ca I, and P II in compare to the other low abundancy elements of Ca II, K II, Na II, Si II, N I and Fe I. The emission spectrum analysis for the soil of Basrah city indicates the abundancy of the elements, Ca II, Ca I, and P II in compare to the other low abundancy elements of Ca II, K II, Na II, Si II, N I and Fe I. The emission spectrum analysis for the soil of Babylon city indicates low abundancy of the elements Na I, Ca I, Ca II, Fe I, K II, N I, Si II and P II show as in fig. 12. Fig. 12 illustrated the emission spectra of the different southern soil of Iraqi cities using an atomic emission spectroscopy (AES) technique.
Table 1 explain the soil content ratio of the elements Ca II, in Faw, Nasseria and Karabala cities is the same, the soil content ratio of the elements K II, N II in Nasseria, Karabala and Basrah cities is the same. The elements Ca II, N II, Si II, Na II, K II and P II are all ionic elements, while Fe I is atomic.

Figure 11: Illustrated the emission spectra of the different southern soil.
element. The soil content ratios of Ca II and Na II in the southern regions are the highest in compare to other soil elements.

Table 1: Elements ratio of Southern regions.

| Cities | Elements of soil | Intensity (a.u) | Wavelength (nm) | Wavelength (nm) Standard |
|--------|------------------|----------------|----------------|-------------------------|
|        |                  |                |                |                         |
| Samawah | Fe I             | 0.03           | 383.422        | 383.422                 |
|         | Ca II            | 0.7            | 393.366        | 393.366                 |
|         | Ca II            | 0.07           | 396.846        | 396.847                 |
|         | Ca I             | 0.1            | 422.672        | 422.673                 |
|         | N II             | 0.003          | 499.436        | 499.437                 |
|         | Si II            | 0.06           | 568.881        | 568.882                 |
|         | Na II            | 0.21           | 588.995        | 588.995                 |
|         | K II             | 0.03           | 612.027        | 612.028                 |
|         | P II             | 0.18           | 616.559        | 616.560                 |
|         | Fe I             | 0.02           | 383.422        | 383.422                 |
|         | Ca II            | 0.27           | 393.366        | 393.366                 |
|         | Ca II            | 0.22           | 396.846        | 396.847                 |
|         | Ca II            | 0.19           | 422.672        | 422.673                 |
|         | Ca I             | 0.07           | 445.478        | 445.477                 |
| Faw    | N II             | 0.06           | 499.436        | 499.437                 |
|         | Si II            | 0.37           | 568.881        | 568.882                 |
|         | Na II            | 1              | 588.995        | 588.995                 |
|         | K II             | 0.01           | 612.027        | 612.028                 |
|         | P II             | 0.04           | 616.559        | 616.560                 |
|         | Fe I             | 0.1            | 383.422        | 383.422                 |
|         | Ca II            | 0.97           | 393.366        | 393.366                 |
|         | Ca II            | 0.82           | 396.846        | 396.847                 |
|         | Ca I             | 0.14           | 422.672        | 422.673                 |
| Najaf  | Ca I             | 0.16           | 445.478        | 445.477                 |
|         | N II             | 0.05           | 499.436        | 499.437                 |
|         | Si II            | 0.03           | 568.881        | 568.882                 |
|         | Na II            | 0.27           | 588.995        | 588.995                 |
|         | K II             | 0.04           | 612.027        | 612.028                 |
|         | P II             | 0.2            | 616.559        | 616.560                 |
|         | Fe I             | 0.01           | 383.422        | 383.422                 |
|         | Ca II            | 0.22           | 393.366        | 393.366                 |
|         | Ca II            | 0.17           | 396.846        | 396.847                 |
|         | Ca I             | 0.10           | 422.672        | 422.673                 |
| Nasseria | Ca I            | 0.14           | 445.478        | 445.477                 |
|         | N II             | 0.01           | 499.436        | 499.437                 |
|         | Si II            | 0.17           | 568.881        | 568.882                 |
|         | Na II            | 1              | 588.995        | 588.995                 |
|         | K II             | 0.02           | 612.027        | 612.028                 |
|         | P II             | 0.3            | 616.559        | 616.560                 |
|         | Fe I             | 0.008          | 383.422        | 383.422                 |
|         | Ca II            | 0.21           | 393.366        | 393.366                 |
|         | Ca II            | 0.20           | 396.846        | 396.847                 |
| Karabala | Ca I             | 0.04           | 422.672        | 422.673                 |
|         | Ca I             | 0.04           | 445.478        | 445.477                 |
|         | N II             | 0.01           | 499.436        | 499.437                 |
|         | Si II            | 0.005          | 568.881        | 568.882                 |
|         | Na II            | 0.13           | 588.995        | 588.995                 |
|         | K II             | 0.02           | 612.027        | 612.028                 |
|         | P II             | 0.10           | 616.559        | 616.560                 |
XRF Measurements of Southern Region

Soil analyses of Southern region of Iraqi by technique of XRF are studied. The composition of Southern region Faw, Nasseria is shown in Table 2. It is noticed from the below table that the soil content ratio of the elements Ca, Fe and P in both Faw and Nasseria cities is proximity, while the soil content ratio of the elements Na, Si is not proximity. The soil content ratio of Si, Na in Faw city is the highest compared to other soil elements, while the soil content ratio of Si, Ca in Nasseria city is the highest compared to other soil elements.

Table 2: Ratio of elements of Southern region.

| Content | Elements | Region |
|---------|----------|--------|
| 27.11   | Na       | Faw    |
| 14.26   | Si       | Faw    |
| 0.121   | P        | Faw    |
| 1.254   | K        | Faw    |
| 10.61   | Ca       | Faw    |
| 3.393   | Fe       | Faw    |
| 3.972   | Na       | Nasseria |
| 29.39   | Si       | Nasseria |
| 0.443   | P        | Nasseria |
| 1.243   | K        | Nasseria |
| 13.15   | Ca       | Nasseria |
| 1.757   | Fe       | Nasseria |

LIBS analysis for the soil of Faw city indicates the abundancy of the element Ca and Si, While XRF analysis for the soil of Faw city indicates the abundancy of the element Ca and Si.

Conclusions

The soil of Samawah city features a good concentration of the elements P (0.18 a.u), Ca (0.7 a.u) while its N (0.003 a.u) is low. The reason for the H content of this soil is that the current experiment was conducted in air. The soil in Faw city features a good concentration of the elements Na II (1a.u), Ca II (0.27a.u). It is also noticed that the Si element is rare in this soil. The soil of Najaf governorate features a good concentration Ca II (0.97 a.u), P II (0.2 a.u) elements. The soil of Nasseria city contain abundant of the elements Ca II (0.21a.u), P II (0.3 a.u), Si making it suitable for agriculture. The soil of Karbala city contain acceptable amount of Ca II (0.21 a.u) element. In Basrah
governorate the concentration of Ca II (0.31 a.u) elements is accepted. The soil Babylon city contain small traces of P (0.06 a.u), K II (0.005 a.u) and Ca II (0.05 a.u) elements. From the above soil sample analysis of the southern cities of Iraqi, it is concluded that the rich soil element of P, Si, Ca elements make the Faw and Nasseria fit for cultivation.

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