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Using Laser - induced breakdown spectroscopy system to determine the fertility of middle Iraqi soil

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Abstract

In this work, different soil samples were brought to study and analyse the element concentrations from different middle regions of Iraq (such as, Habbaniah, Garmah, Fallujah, and Tarmiah cities). Using laser-induced breakdown spectroscopy (LIBS) has been documented as an atomic emission spectroscopy (AES) technique. Laser-induced plasma utilized to analyse elements in materials (gases, liquids, and solids) in order to analyse elements in materials (gases, liquids and solid). The Nd:YAG laser excitation source at 1064nm with pulse width and pulse duration of 9ns is used to generate power density of 5.5 x 10^{12} MW/mm^2, with optical spectrum in the range 320 -740 nm.

The soils of Habbaniyah and Garmah cities features have small concentrations of P II (0.08 a.u), K II (0.01 a.u), SiII (0.004 a.u) and Ca II (0.04 a.u) elements. However, the Fallujah city features have a good concentration of Ca II (0.14a.u). It is concluded that the soil in Fallujah city is better than Habbaniah, Tarmiah and Garmah cities of concentration elements.

Keywords: Laser-induced breakdown spectroscopy (LIBS), XRF, and soil fertility.

أستخدام منظومة طيف الانهيار المحتث بالليزر لتحديد خصوبة تربة وسط العراق

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الخلاصة

في هذا البحث تم الحصول على اربع عينات من اتربة او اطيان مختلفة وذلك لدراسة وتحليل تركيز العناصر المختلفة المكونة لها ، ولمناطق مختلفة من وسط العراق مثل الحبانية والكرمة والفلوجة والطارمية. وذلك بأستخدام نظام طيف الأنهيار المحتث بالليزر (LIBS)، بوجود تقنية طيف الأنبعاث(AES). للبلازما المحتثة بالليزر للحالات (الغازية،السائلة والصلبة) ، حيث يستعمل ليزر النيوديميوم . ياك بعامل نوعية ذات فتح عند الطول الموجي 1064 نانومتر ، وبزمن نبضة قدره 9 نانوثانية ، لتوليد شدة نبضة بقيمة 5.5×10^{12} ميكا واط /ملم^2 ، ضمن الطيف البصري ذي المدى 320 . 740 نانومتر .

التربة في مدينتي الحبانية والكرمة تتميز بمحتواها قليل التركيز للعناصر مثل الفسفور(0.08a.u) والبوتاسيوم (0.01 a.u) والسيلكون (0.004 a.u) والكالسيوم (0.04 a.u). بينما تربة مدينة الفلوجة تتميز بتركيز جيد للكالسيوم (0.14 a.u) . يستنتج من ذلك ان تربة مدينة الفلوجة افضل من ترب او اطيان مدن الحبانية ،الطارمية والكرمة لتركيز العناصر .

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Introduction

Laser-induced breakdown spectroscopy (LIBS) is an analytical technique that allows for the determination of a sample's elemental composition. It is 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 [1,2,3].

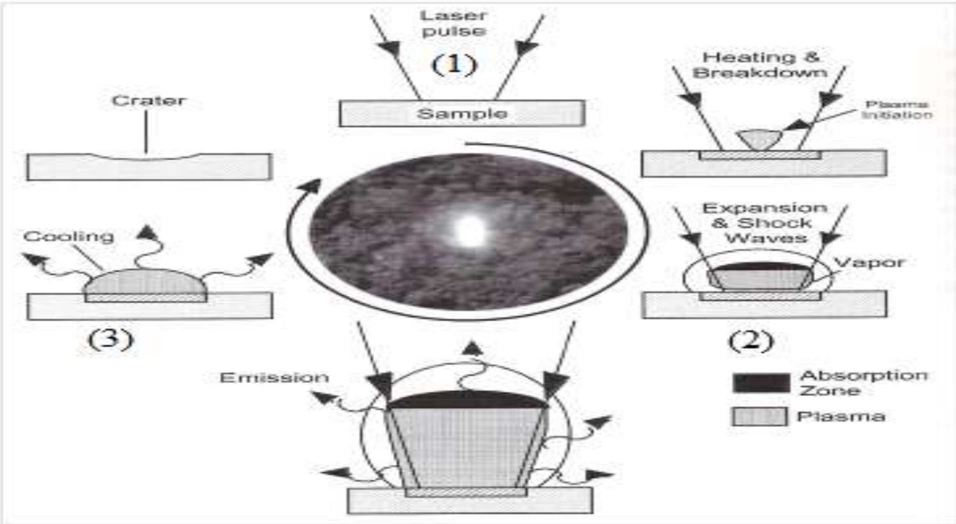

Figure 1-Life cycle diagram showing of the main events in the LIBS process [4].

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 that may take just a second of time [5]. LIBS is also applicable to the analysis of extremely hard materials that are difficult to digest or dissolve, such as ceramics and semi or super-conductors [6].

There are many applications of LIBS for qualitative and quantitative elemental measurements in a wide range of samples such as various environmental samples ,nonmetallic solids, liquid samples and gases samples. Among the applications of LIBS in biological and medical samples include calcified tissue materials (e.g. teeth, bones, sea shells), soft tissue materials (e.g.human skin, plant parts like leaves and wood) [7.8].

LIBS has been used for the materials detection and analysis in various applications, such as steel. [9] wood pre-treatment. [10] bacteria [11], molds, pollens, proteins [12], space exploration [13] and books [14].

The X-ray fluorescence XRF technique is a very powerful, portable and easy tool compared to the LIBS technique. The ionising electromagnetic radiations, which are X. Furthermore, gamma-rays are also used to move the electron away from its orbital (energy level) causing ionisation. In other words, Both X and gamma ray photons are able to eject an electron from its orbit in an atom ("are ionising radiation"), instigating ionisation, 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 [15].

a. Soil: Soil aggregation is defined as the group of soil particles in which the forces held the particles together. The aggregation is formed by some processes like physical, chemical and biological forces which are mainly responsible for their stabilization [15].

b. Soil fertility: The fertility of soil "its ability to provide nutrients (in adequate amounts and in proper balance) for the growth of plants, when other important growth factors are favorable. For optimum crop production, a soil that enables deep rooting, provides aeration, has a good water holding, capacity and consists of an adequate and balanced supply of plant nutrient is considered a productive and fertile soil [15]. The aim of this work is to employ the high power laser-induced plasma spectroscopic analysis techniques and 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. The process 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/mm^2, 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.

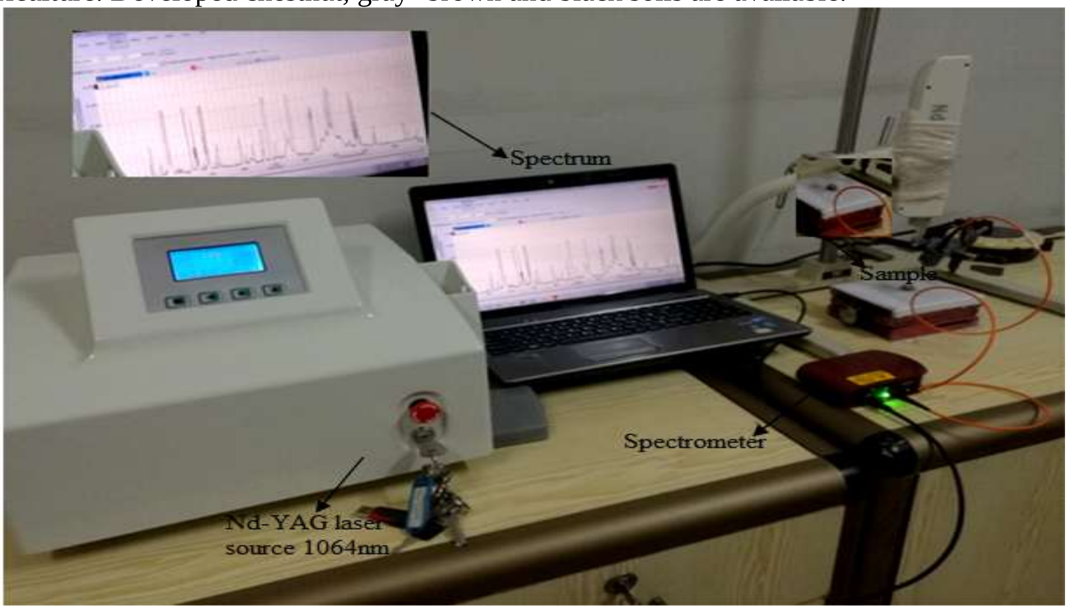

Figure 2-Laser-induced breakdown spectroscopy system

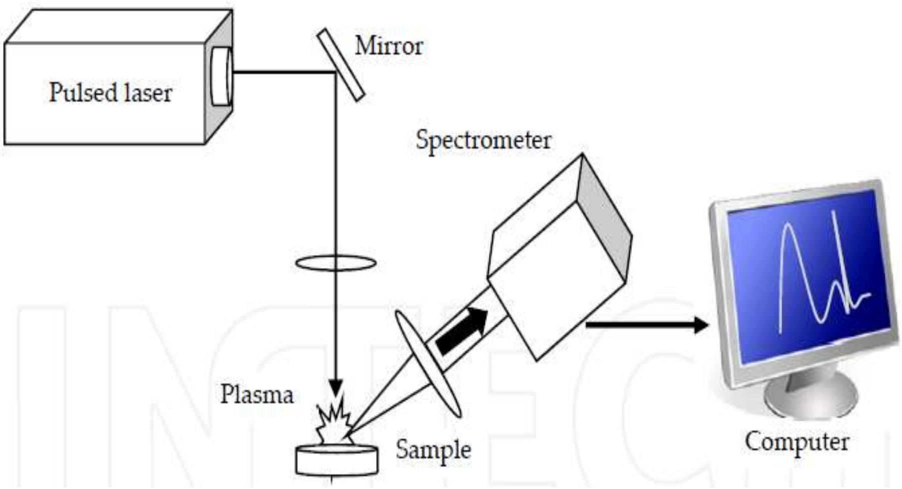

Figure 3-A schematic diagram of the LIBS system.

A total of four various depth levels down to 30 cm soil samples were brought from different northern regions of Iraq, as shown in Figure-4. Habbaniyah, Garmah, Fallujah and Tarmiah. 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.

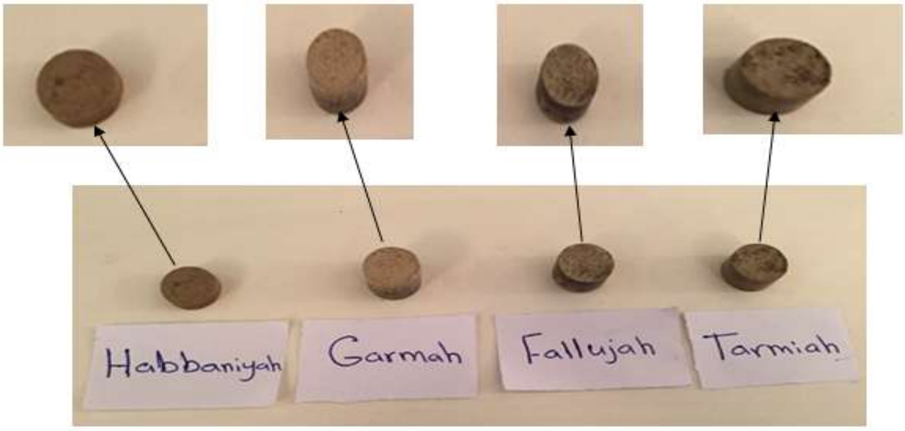

Figure 4-The samples used in the experimental work, middle

Results and discussion

In Al- Habbaniyah region, the emission peaks that present as shown in Figure-5 are Fe I at wavelength 383.422 nm, intensity 0.003 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.04 a.u and 396.846 nm, intensity 0.05 a.u, two peaks of Ca I 422.672 nm, intensity 0.02 a.u and 445.478 nm, intensity 0.03 a.u, N II 499.436nm, intensity 0.004 a.u, Si II 568.881 nm, intensity 0.004 a.u, Na II 588.995 nm, intensity 0.07 a.u, K II 612.027nm, intensity 0.01 a.u, P II616.559 nm, intensity 0.08 a.u.

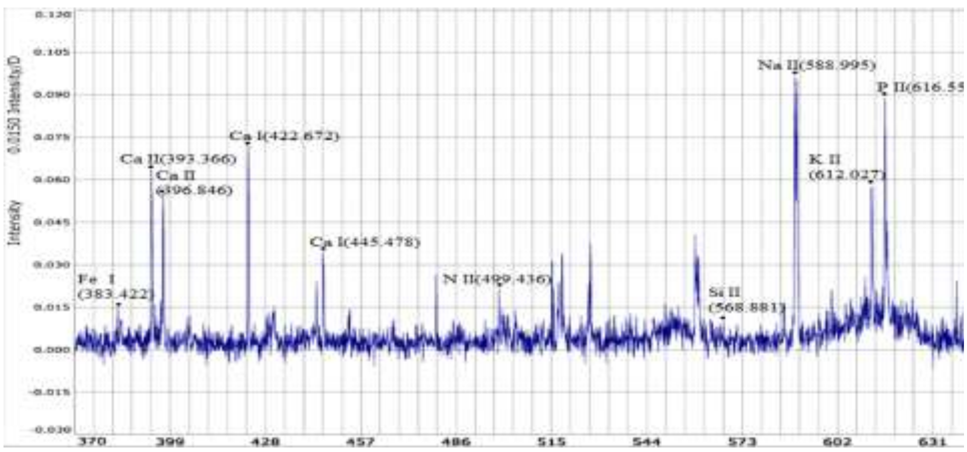

Figure 5-LIBS spectra recorded from Habbaniyay soil.

In Al- Garmah region, the emission peaks that present as shown in Figure-6, are Fe I at wavelength 383.422 nm, intensity 0.004 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.08 a.u and 396.846 nm, intensity 0.09 a.u, two peaks of Ca I 422.672 nm, intensity 0.005 a.u and 445.478 nm, intensity 0.01 a.u, N II 499.436nm, intensity 0.006 a.u, Si II 568.881 nm, intensity 0.001 a.u, Na II 588.995 nm, intensity 0.17 a.u, K II 612.027nm, intensity 0.001 a.u, P II616.559 nm, intensity 0.07 a.u

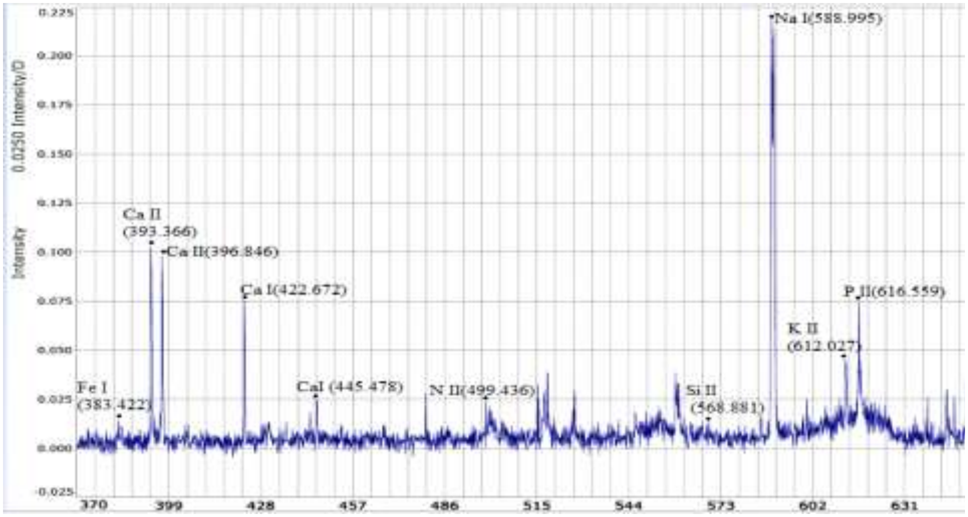

Figure 6-LIBS spectra recorded from Garmah soil.

In Fallujah region, the emission peaks that present as shown in Figure-7, are Fe I at wavelength 383.422 nm, intensity 0.008 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.14 a.u and 396.846 nm, intensity 0.13 a.u, two peaks of Ca I 422.672 nm, intensity 0.01 a.u and 445.478 nm, intensity 0.02 a.u, N II 499.436nm, intensity 0.004 a.u, Si II 568.881 nm, intensity 0.007 a.u, Na II 588.995 nm, intensity 0.07 a.u, K II 612.027nm, intensity 0.01 a.u, P II616.559 nm, intensity 0.08 a.u.

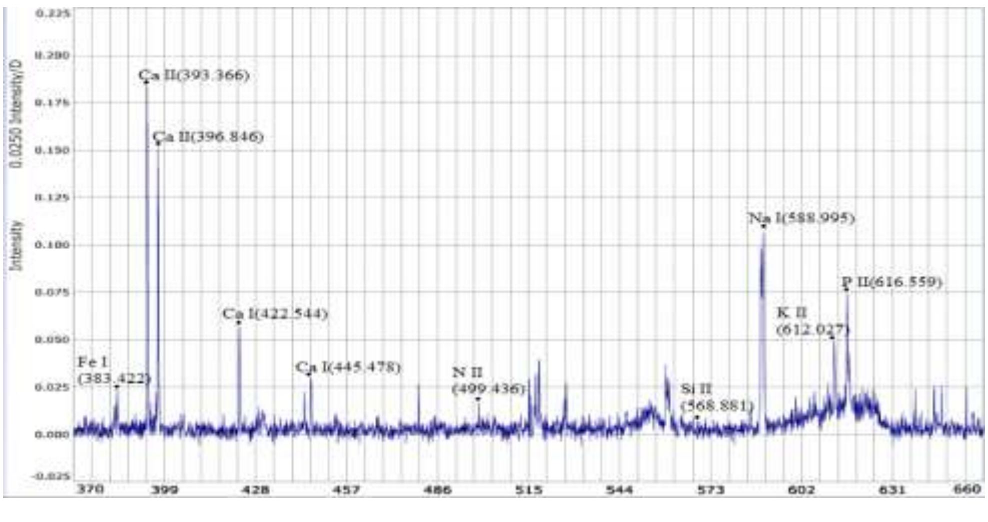

Figure 7-LIBS spectra recorded from Fallujah soil.

In Tarmiah region, the emission peaks that present as shown in Figure-3, are Fe I at wavelength 383.422 nm, intensity 0.03 a.u, two peaks of Ca II at wavelength 393.366 nm, intensity 0.21 a.u and 396.846 nm, intensity 0.18 a.u, two peaks of Ca I 422.672 nm, intensity 0.04 a.u and 445.478 nm, intensity 0.05 a.u, N II 499.436nm, 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 612.027nm, intensity 0.01 a.u, P II616.559 nm, intensity 0.09 a.u.

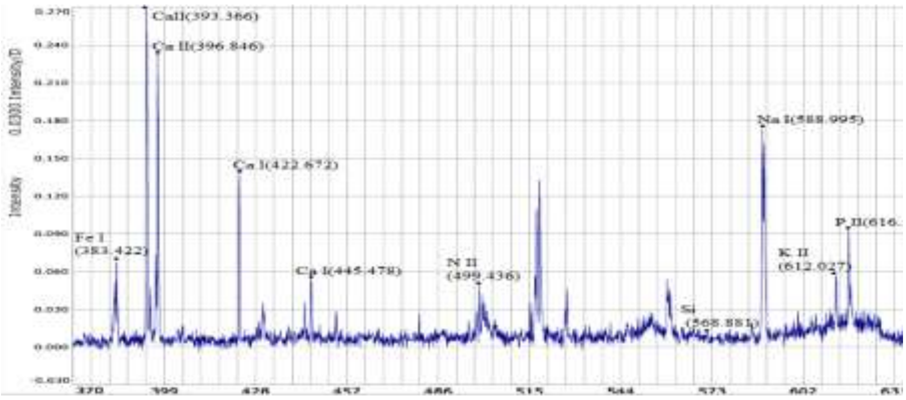

Figure 8-LIBS spectra recorded from Tarmiah soil.

The Figure-9 illustrates all spectra of the middle region for comparison between spectra for identify soil components and fertility.

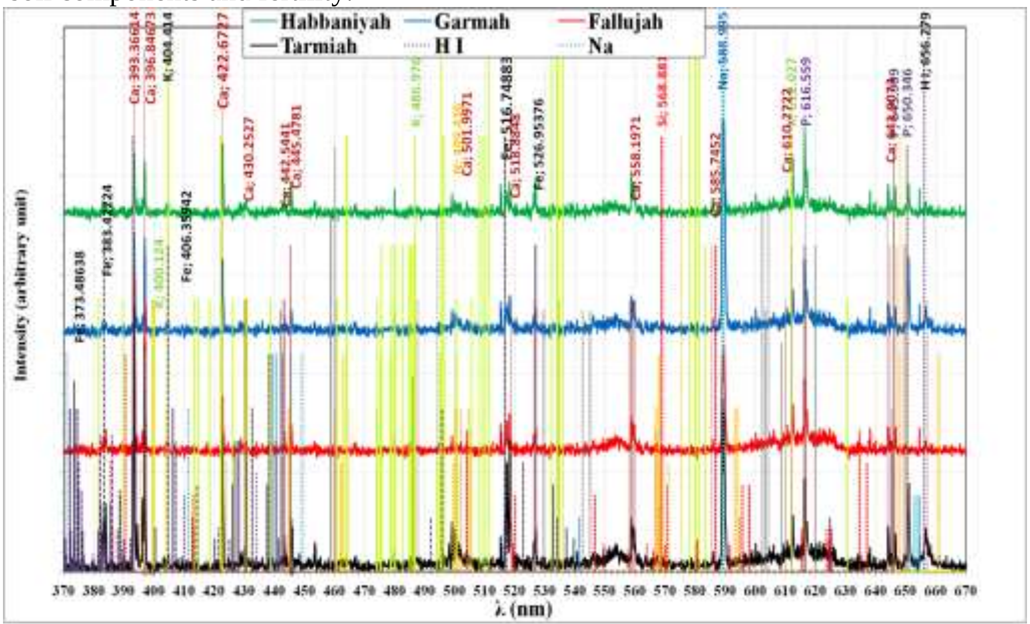

Figure 9-The LIBS emission spectra recorded of the different samples of soils.

Table-1 illustrates the analysis and composition of soil samples for middle regions of Iraq, by LIBS system. It is shown that the soil content ratios of Fe I, Ca II, , K II and P II elements are similar for both Habbaniyah and Garmah cities. But all elements Ca II, N II, Si II, Na II, K II and P II are ionic elements for soil samples of middle regions, while Fe I is an atomic element. Also, the soil content ratio of Ca II and Na II are the highest compared to the soil elements for the middle regions in Iraq.

Table 1-Soil content Ratios of elements for middle regions

Region	Elementsof soil	Intensity (a.u.)	Wavelength (nm)
	Fe I	0.003	383.422
	Ca II	0.04	393.366
	Ca II	0.05	396.846

Habbaniyah	Ca 1	0,02	422.672
	Ca I	0.03	445.478
	N II	0.004	499.436
	Si II	0.004	568.881
	Na II	0.07	588.995
	K II	0.01	612.027
	P II	0.08	616.559
Garmah	Fe I	0.004	383.422
	Ca II	0.08	393.366
	Ca II	0.09	396.846
	Ca I	0.005	422.672
	Ca I	0.01	445.478
	N II	0.006	499.436
	Si II	0.001	568.881
	Na II	0.17	588.995
	K II	0.001	612.027
	P II	0.07	616.559
Fallujah	Fe I	0.008	383.422
	Ca II	0.14	393.366
	Ca II	0.13	396.846
	Ca I	0.01	422.672
	Ca I	0.02	445.478
	N II	0.004	499.436
	Si II	0.007	568.881
	Na II	0.07	588.995
	K II	0.01	612.027
	P II	0.07	616.559
Tarmiah	Fe I	0.03	383.422
	Ca II	0.21	393.366
	Ca II	0.18	396.846
	Ca I	0.04	422.672
	Ca I	0.05	445.478
	N II	0.01	499.436
	Si II	0.005	568.881
	Na II	0.13	588.995
	K II	0.01	612.027
	P II	0.09	616.559

The XRF technique is used to analyse the soil samples of middle regions of Iraq. Therefore, the compositions of middle regions for both Garmah and Habbaniyh are shown in Table-2. It denotes that the soil content ratio of the elements ,K, Na ,and P for Garmah and Habbaniyay cities are proximity, while the soil content ratio of the elements Ca and Si are not a proximity. But, the soil content ratio of Si and Ca are the highest compared with the other soil elements. The samples were taken from middle regions of Iraq for comparison between XRF and LIBS measurements for identifying the similarity and difference points for two measures of these regions as shown below.

Table 2- Ratio of elements of the Middle region

Region	Elements	Content
Garmah	Na	0.518
	Si	36.92
	P	0.184
	K	1.138

	Ca Fe	21.76 5.263
Habbaniyay	Na Si P K Ca Fe	2.951 34.08 0.219 0.975 22.19 3.530

Conclusions

The soil sample analyses of the middle cities of Iraq concluded that the soil in Fallujah city is better than Habbaniah, Tarmiah, and Garmah cities. The LIBS analysis for the soil of Garmah city indicates low abundance elements Si and Ca, While XRF analysis indicates the abundance of the elements Ca and Si. The LIBS is low cost and high-efficiency system to analyze the Iraqi soil, LIBS has shown high susceptibility to identify the elements (such as, Iron, Calcium, nitrogen, silicon, sodium, potassium, and phosphorus).

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