International Journal of Trend in Scientific Research and Development (IJTSRD)
Volume 7 Issue 1, January-February 2023 Available Online: www.ijtsrd.com e-ISSN: 2456 – 6470

Detection of Heavy Metals Contents in Some Sudanese Vegetable Oil using Laser Induced Breakdown Spectroscopy

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ABSTRACT

Vegetable oils manufacturing now became one of the most important food industries in any country, some of these oils contain natural compounds, which are classified as high purity oils, and others contain harmful components which are classified as law purity oils because they contain heavy metals components and chemical additives. This paper reviews the use of Laser induced break down spectroscopy for the analysis and characterization of edible oils, including four types of oils (Naama, sabah, shams, afia.) purchased from local Sudanese markets The results showed that the spectra of oil samples are similar, but they show some differences in the concentration of some elements. And the results showed that they contain toxic heavy metals (Pb, Cu, As, Hg, Sr) which are harmful to health that may can cause cancer diseases, and can also cause great harms health. The recorded spectra samples were analyzed using [NIST data].

KEYWORDS: *heavy metals, Sudanese vegetable oils, Concentration (ppm) laser induced breakdown spectroscopy*

How to cite this paper: Nedal Abdulazim | Abdelmoneim Awadelgied | Sohad Saad Elwakeel | Ahmed Abubaker Mohamed "Detection of Heavy Metals Contents in Some Sudanese Vegetable Oil using Laser Induced Breakdown Spectroscopy" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-7 | Issue-1, February 2023, pp.488-493, URL: www.ijtsrd.com/papers/ijtsrd52726.pdf

IJTSRD52726

INTRODUCTION:

Vegetable oils are widely used in food (edible oils), cosmetic, pharmaceutical and chemical Industries. Regarding the world-wide food production, the edible oils derived from various plants and seeds, have an important role in human nutrition due to cholesterol reducing property [1, 2]. The quality of the edible oils, regarding their freshness and toxicity, can be evaluated through the determination of several trace metals, this being one of the most important criteria for the quality assessment of the oils from the health point of view [3-4]. Metals play important negative and positive roles in human life [5]. Vegetable oils and fats contain trace levels of various metals depending on many factors, such as species, soil used for cultivation, irrigation water, variety, and stage of maturity, pollution [6, 5].

Heavy metals in the soil and contaminants reduces the quality of food products and food safety, and they also poses great risk to the health of animals and humans since heavy metals could be transferred into the chain food (soil-plant human or soil-plant-animal-human)[7]. Elements such as copper, zinc, magnesium, and iron have been reported to increase oil oxidation, while copper and iron catalyze the decomposition of hydroperoxides, thereby leading to the formation of hazardous substances in oils. Lead, arsenic, mercury, and cadmium are toxic heavy metals found in oils [8]. It is well established that heavy metals have adverse effects on the nutritional value, toxicity, and oxidative stability of oils. Therefore, it is critical to determine the concentrations of heavy metals in various oils [9].

Laser-induced breakdown spectroscopy (LIBS) is atomic analytical technique a multi elemental and with simultaneous detection of all the elements in real-time in any type of sample matrix including solid, liquid, gas, and aerosol [10].

LIBS is one of the atomic emission techniques that has found wide interest in many fields such as research, industry and others because it has many advantages over other analysis techniques [11].

LIBS technique is based on collecting atomic emissions in a plasma spark. All elements are emitted in the spectral range (200-900) nm and therefore by using a detector that covers the entire spectral range we can detect and identify the elements by their relative abundance [12, 13]. (LIBS) technology is used in quantitative and qualitative analysis, and it is a rapid technique to know the components of samples and is used in all kinds of materials, solid, liquid, gas and others.in this work we used (LIBS) to detection the heavy metals in Sudanese vegetable oil samples.

Experimental Samples:

Four types of Sudanese edible oils (Naama, sabah, Shams, afia.) This edible oils produced from (corn oil, peanut, and sunflower oils) were selected as the experimental samples. The oil samples used in this work were purchased as commercial products from a local supermarket in Khartoum, Sudan.

Experimental setup:

The main physical process that forms the essence of LIBS technology is the formation of high-temperature plasma, induced by a short laser pulse. When the short-pulse laser beam is focused onto the sample surface, a small volume of the sample mass is ablated (i.e. removed via both thermal and non-thermal mechanisms) — in a process known as Laser Ablation. This ablated mass further interacts with a trailing portion of the laser pulse to form a highly energetic plasma that contains free electronics, excited atoms and ions. Many fundamental research projects have shown that the plasma temperature can exceed 30,000K in its early life time phase.

When the laser pulse terminates, the plasma starts to cool. During the plasma cooling process, the electrons of the atoms and ions at the excited electronic states fall down into natural ground states, causing the plasma to emit light with discrete spectral peaks. The emitted light from the plasma is collected and analyzed by using a detector module for LIBS spectral analysis. Each element in the periodic table is associated with unique LIBS spectral peaks. By identifying different peaks for the analyzed samples, their chemical compositions can be rapidly determined. Often, information on LIBS peak intensities can be used to quantify the concentration of trace and major elements in the sample.

With the advancement of powerful chemometric software for LIBS data analysis, and with steady progress in understanding laser ablation fundamentals, today's analytical researchers are applying LIBS effectively for both quantitative and material discriminatory analysis for a wide range of sample matrices.

The experimental setup consisted of Q- switched Nd: YAG nanosecond laser provides pulses of 8 ns duration at 266 nm, with pulse energy 30 milli joule .The USB 2000 spectrometer ocean optical company made in USA usable range 2400-1150 a detector 2048 element silicon CCD away integration time 1ms-60se optical resolution 0.3 -10nm FWHM [14]. The emission is observed at a 90°angle to the laser pulse as shown in Figure 1. The recorded spectra of the samples were analyzed using NIST data.

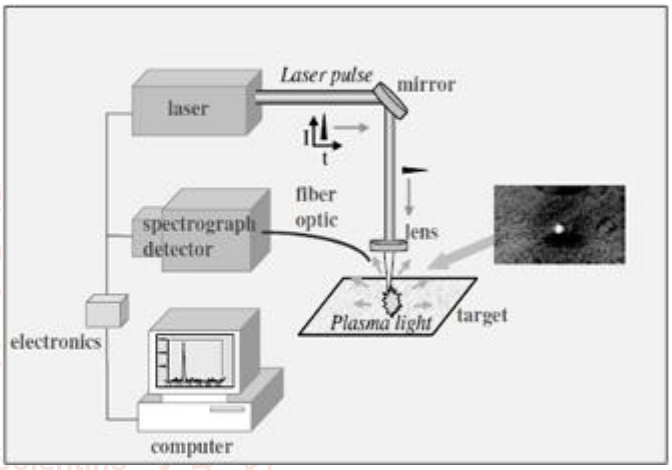

Fig (1): A schematic of a simple apparatus for laser-induced breakdown spectroscopy illustrating the principal components. The main components are:

1. pulsed laser that generates the powerful optical pulses used to form the microplasma
2. The focusing system of mirror and lens that directs and focuses the laser pulse on the target sample;
3. Target holder or container (if needed);
4. The light collection system (lens, mirrors or fiber optic) that collects the spark light and transports the light to the detection system;
5. Detection system consisting of a method to spectrally filter or disperse the light such as a spectrograph and a detector to record the emission light.
6. Computer and electronics to gate the detector, fire the laser, and store the spectrum. The basic elements of any (LIBS) system are the same but the component specifications are dedicated to the particular application. These specifications include physical parameters such as size, weight, packaging, power and utilities required for operation as well as technical specifications for operational performance. [15]

Software Origin Lab is software that specializes in data analysis and processing (LIBS) spectra.

Results and Discussion:

The LIBS emission spectra for the 4types of Sudanese edible oils (Naama .sabah shams, afia.) this edible oil produced from (corn oil, peanut, and sunflower oils) shown in Figures from 2 to 5 after irradiation with 30 mJ pulse energy, the spectra were recorded in the region from (300 nm to 1100 nm) nm.

LIBS spectrum of sample Naama in the range of (300 to 1100) nm as figure 2 shows. It shows clear peaks and by comparison with the wavelengths recorded in some references, we found that these wavelengths describe the components of Naama oil as listed in Table 1.

Table 1 Analysis of LIBS Emission Spectrum of Naama oil

Measured λ (nm)	Intensity (a.u)	Elements	Reference	Concentration (ppm) LIBS
488.132	131	Li II	MH59	0.0813
511.319	132	Ti I	F91	0.27121
514.949	136	Hg II	SR01	1.65836
520.144	132	Pb I	WA68	1.7
534.548	132	Nb I	SCM75	1.50926
652.766	135	Ba I	KL99	0.72
664.139	131	Cu II	R69	14.8
672.484	131	CSII	S81	303
677.507	132	RbII	R75	481
709.231	137	Zr II	JDBA90	0.53
718.289	133	In II	BC38	6.4
730.949	132	SrI	SCM75	3.3
757.891	135	S II	KM93	271
764.189	131	Kr II	MHD33	2.7
1048.778	134	K I	R56	7.5

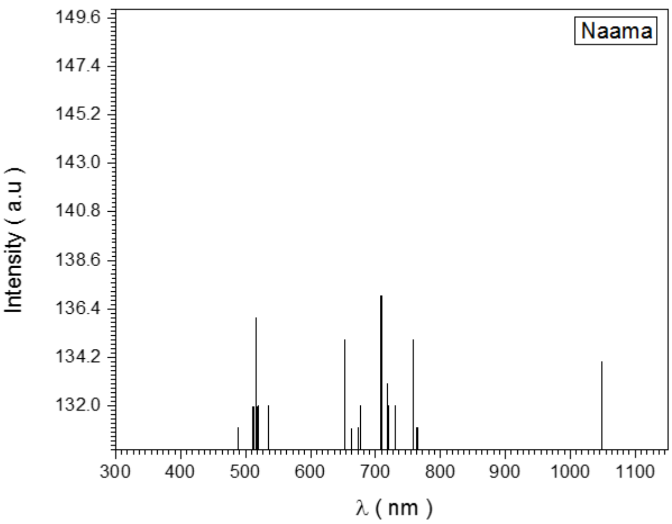

Fig 2: LIBS emission spectrum of Naama oil

LIBS spectrum of sample Sabah oil in the range of (300 to 1100) nm. Figure 3 shows clear peaks and by comparison with the wavelengths recorded in some references, we found that these wavelengths describe the components of Sabah oil listed in Table 1.

Table 2 Analysis of LIBS Emission Spectrum of Sabah oil

Measured λ (nm)	Intensity (a.u)	Elements	Reference	Concentration (ppm) LIBS
419.655	131	La II	MCS75	1.7
424.492	131	Pb II	WRSH74	3.9
488.559	143	Rb II	R75	514
489.991	135	Ti I	F91	0.41
542.525	132	Hg I	SR01	2.2
612.731	135	Zr I	J98	0.19
629.538	133	Ce I	BWCC91	3.3
637.847	131	Cu II	R69	20.7
652.766	131	Ba II	KL99	0.56
664.139	132	Cu II	R69	19.78
696.012	131	Pa I	BW926	0.006

709.231	134	Zr II	J98	0.23
752.666	131	Kr I	DHM33	0.021
757.576	132	S II	KM93	243
765.886	133	Mg I	KM91a	101

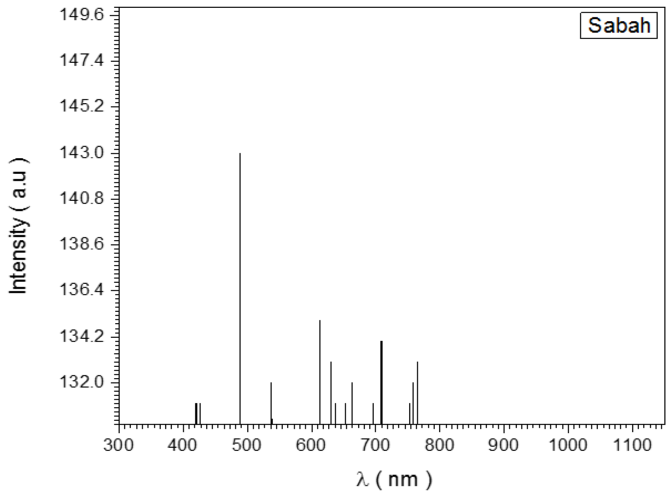

Fig3: LIBS emission spectrum of Sabah oil

LIBS spectrum of sample Shams oil in the range of (300 to 1100) nm. Figure 4 shows clear peaks and by comparison with the wavelengths recorded in some references, we found that these wavelengths describe the components of Sabah oil listed in Table 2.

Table 3 Analysis of LIBS Emission Spectrum of Shams oil

Measured λ (nm)	Intensity (a.u)	Elements	Reference	Concentration (ppm) LIBS
426.339	132	Ti I	F91	0.43
438.646	131	Pb II	WRSH74	16.3
449.756	131	Na I	R56	322
452.847	132	Ce II	C73	5.2
488.132	140	Li I	MH59	0.12
542.525	131	Hg II	SR01	2.1
652.766	135	Ba II	KL99	0.69
687.702	132	Sr I	SCM72	8.3
696.012	131	Pa II	WB926	0.0023
706.012	134	Zr I	J98	0.20
719.240	131	Rb I	B59	667
740.817	132	N I	M75	0.056
1048.778	132	K I	R56	7.8
1055.388	131	U I	WB926	8.12

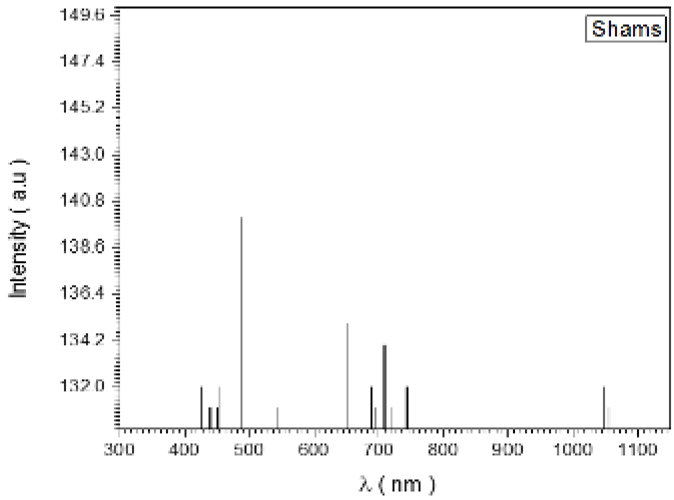

Fig 4: LIBS emission spectrum of Shams oil

LIBS spectrum of sample Afia oil in the range of (300 to 1100) nm. Figure 5 shows clear peaks and by comparison with the wavelengths recorded in some references, we found that these wavelengths describe the components of Sabah oil listed in Table 2.

Table 4. Analysis of LIBS Emission Spectrum of afia oil

Measured λ (nm)	Intensity (a.u)	Elements	Reference	Concentration (ppm) LIBS
384.33	131	V I	AD78	0.033
386.492	133	VI	ADA78	0.033
481.188	130	Sr I	MCS71	5.5
567.711	130	Hg II	SR01	4.6
566.274	133	Ti II	F91	0.35
677.507	130	Rb II	R75	444
696.012	130	Pa II	WB926	0.09
697.330	132	Cs I	K62b	371
751.722	131	Ar I	N73	0.0002
755.897	131	Pb II	S75	5.6
807.432	130	Zr I	J98	0.45
849.583	130	Ce I	BWCC91	1.9
890.219	130	K I	T63	5.9
997.786	131	In I	LJ67	7.1
1000.622	132	As I	AH85	11.6
1003.832	133	Sr II	S389	5.2

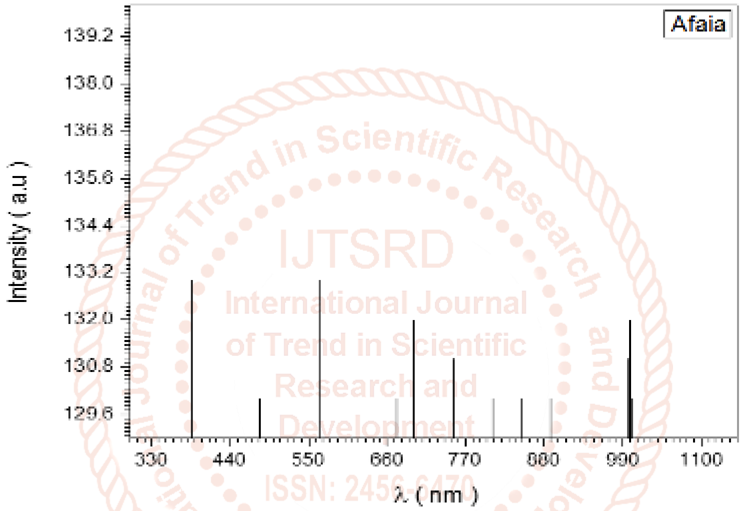

Fig 5: LIBS emission spectrum of afia oil

Discussion;

In this study, 5 elements (Cu, Pb, As, Sr, and Hg) in edible vegetable oil samples were studied using LIBS as shown in table1,2,3,4 and Fig.2,3,4,5

Copper (Cu)concentrations present in table {1,2} 14.8mg/kg and20.7mg/kg respectively the concentration was high than that reported in similar data (0.05mg/kg[16] 0.1mg/kg (Codex2011)

Lead (Pb) concentrations present in table {1,2,3,4} 1.7mg/kg 3.9mg/kg, 16.3mg/kg, 5.6mg/kg, respectively the concentration were high than that reported in similar data 0.02mg/kg [16] 0.1mg/kg) [17] FAO/WHO 0.1 mg/kg (Codex S 2011). recommended limit by the EU and National Iranian Standards Organization (0.1 mg/kg).

Arsenic (As) concentrations present in table {4} 11.6 mg/kg the concentration was high than that reported in similar data 0.01mg/kg[16] 0.1mg/kg (Codex 5) the recommended limit by the National Iranian Standards Organization (0.1 mg/kg).the level recommended by (Food and Agriculture Organization, World Health Organization FAO/WHO) of 0.5 mg/kg (Codex S 2011). The control sample concentration was 0.01mg/

Strontium (Sr) concentrations present in table {1,3,4} 3.3mg/kg, 8.3mg/kg, 5.5mg/kg respectively the concentration was high than that reported in similar data (0.1mg/kg) (16)

Mercury (Hg) concentrations present in table {1,2,3,4} 1.66 mg/kg, 2.2mg/kg, 2.1mg/kg, 4.6mg/kg respectively the concentration was high than that reported in similar data 0.003mg/kg [16].

Conclusion;

The determination of the elemental concentration in edible vegetable oil is vital, since several elements play major roles in various metabolic processes in the human body. Moreover, some of these elements are toxic if consumed in excessive quantities. In this study, 5 elements (Cu, Pb, As, Sr, and Hg) in edible vegetable oil samples were studied using LIBS

Copper (Cu) concentrations was high than that reported in similar.

Lead (Pb) concentrations was high than that reported in similar.

Arsenic (As) concentrations was high than that reported in similar.

Strontium (Sr) concentrations was high than that reported in similar.

Mercury (Hg)) concentrations was high than that reported in similar.

The laser induced break down spectroscopy (LIBS) technique is very accurate LIBS is capable to detect almost all the elements and ions in the oils samples and can be used as diagnostic technique for investigation of elements in liquid samples.

Recommendations;

From the results, the followings can be suggested as future work:

laser-Induced Breakdown spectroscopy, should be focused to improve the LIBS system for analysis and examination of crude oil grains before the manufacturing process to determine if there are toxic elements (Cu, Pb, As, Sr and Hg) to avoid the manufacturing process.

LIBS method can be considered in the future as an analytical technique of many classes of all types of oil compounds.

Diode pulse lasers can be used to overcome the laser flash lamp background.

Study the percentage of the heavy elements in oils.

Study the effect of the chemical additives on the toxicity of the oils

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