An Analytical Comparative Study of High-Resolution Laser Induced Breakdown Spectroscopy and X-ray Fluorescence in Children's Dental Tests

Wisam D. Jalal [1]
Mohanad A. Aswad [2]
Ahmed M. Ahmed [1]

[1] *Department of Physics, College of Science, University of Anbar, Ramadi, IRAQ*
[1] *Department of Physics, College of Science, University of Baghdad, Baghdad, IRAQ*

Here, we compared two popular analytical methods for studying children's teeth: X-ray fluorescence (XRF) and high-precision laser-induced plasma (LIBS). A total of three child tooth specimens were collected from a clinic in Ramadi, Iraq. An Nd:YAG Q-switched laser with the following specifications: a wavelength of 1064nm, a pulse time of 10 ns, and a pulse energy of 600 mJ was used to record optical emission spectra within the LIBS system. Thorlabs model CCS spectrophotometer was used to analyze dental specimens' emission spectra throughout the 300-1100 nm spectral range, with a precision of 100/M < 0.5 nm. Additionally, X-ray fluorescence imaging was employed to register the XRF spectrum. More accurate and comprehensive elemental analysis capabilities are offered by LIBS compared to XRF. For both realistic and academic dentistry functions, LIBS has proven to be a really useful analytical device. The outcomes confirm that LIBS has promise in dentistry.

***Keywords*:** LIBS; X-ray fluorescence; Dentistry; Teeth care
Received: 20 December 2023; **Revised:** 31 January; **Accepted:** 07 February 2024

1. Introduction

There has been promising evidence in latest years that picosecond or sub-picosecond pulsed lasers could update conventional mechanical approaches techniques with touch-less drills for the analysis of dental trace elements. This finding could have far-achieving implications for our understanding of human health [1-2]. Therefore, we will attempt to paintings on present day analytical techniques which could reliably compare dental specimens.

In the LIBS technique, elements' excited atoms and ions are detected via studying atomic emission spectra after plasma is created at the floor of a substance using focused pulsed lasers [3]. This approach allows huge-scale elemental imaging of calcified human tissues and different heterogeneous materials, supplying both quantitative and qualitative consequences [4]. LIBS method has been rapidly enhancing in current years for research of substances in all their states. It is preferred over traditional emission spectroscopy methods among the several now available because it provides information from micron-scale specimens without requiring specimen preparation. These benefits have led to widespread adoption of this technique in biomedicine [5]. The analysis by this technique successfully determines the chemical composition of solid, liquid, and gas specimens. In addition, of all biological tissues, hard tissues (tooth and bones) are the most amenable to LIBS analysis [4-6]. In contrast, x-ray fluorescence spectrometry (XRF) is a tried-and-true analytical atomic method for quantitative and qualitative chemical analysis over a broad variety of matrices and elements [7].

The incisor, or first tooth, will erupt into the jaw at about six months of age. When the first dental comes in at birth or during the initial few weeks of life, it can significantly alter the eruption schedule [8]. Dentine, root, and enamel cementum are the three main components of a human teeth. The root cementum is a mineralized, avascular tissue that covers the whole root surface of the tooth. Because of its intermediate position, it makes contact with the periodontal ligament and root dentine [9].

Hydroxyapatite is the primary building block of all human hard tissues, including teeth. The most heavily calcified tissue in a human tooth is the enamel, which consists of 4% organic matter and 96% inorganic material (mostly hydroxyapatite). As a percentage of total density, dentine is 70% inorganic minerals, 20% organic, and 10% water. Inorganic minerals make up 65% of cement's weight, while organic matter accounts for 23% and water for 12% [10]. The human body's most mineralized tissue is enamel, the protective covering on teeth. The primary component of this inorganic substance is a calcium phosphate that is associated with hexagonal hydroxyapatite [11].

In order to find the best and most accurate method for assessing trace elements in dental specimens from children, this research will compare LIBS and XRF as analytical methods.

2. Experimental Part

In this practical part of this research, three specimens of teeth from three different children were collected from a dental clinic in the city of Ramadi, Iraq and utilized as it is , for analysis using LIBS and XRF methods.

Optical emission spectra were recorded during plasma formation using the LIBS setup depicted in Fig. (1). A 1064nm-wavelength Q-switched Nd:YAG laser with a pulse time of 10 ns and a 6 Hz frequency was the main component of the system. We used a 10 cm converging lens to focus the 600 mJ laser beam onto the surface of the dental specimen. To create plasma, we angled a 45-degree optical fiber to focus the laser beam 10 cm away from the specimen.

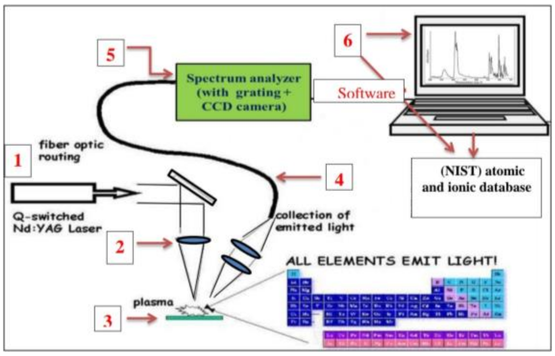

Fig. (1) LIBS experimental setup

A fiber optic cable was used to transport the plasma light produced by the interaction of the laser beam with the specimen to the spectrum analyzer, and then LIBS spectra were recorded in air at atmospheric pressure to get the spectral data. The spectra were captured using a Thorlabs model CCS spectrometer, which covers the wavelength range of 300-1100 nm. A personal computer could record the spectra using the spectrometer's (Thorlab) software, with a resolution of 100/M<0.5nm. The NIST database, used for elemental identification, calculated the emission spectra of dental elements.

Furthermore, we employed XRF imaging to capture spectra from tooth specimen using a model of Spectro-Xepos and German starch.

3. Results and Discussion

Figure (2) shows the 300-1100 nm plasma emission spectra acquired with the LIBS technique for the first tooth specimen. There was a long list of elements whose spectra were recorded, including PI, Ti I, Ti II, Zn II, Mg I, Mg II, Sc I, Fe I, Fe II, and Ca I, all of which emitted light at different wavelengths. The concentrations of Ca I at 616.21nm, Sc I at 631.39nm, P I at 1058.91nm, and Fe I at 653.37nm were the highest, while the concentrations of other elements were few.

Figure (3) shows the 300-1100 nm plasma emission spectra acquired with the LIBS technique for the second dental specimen. The spectral lines recorded elements such as PI, Mg II, Fe I, Sc I, Sc II, Ti I, and Ca III, all emitting at different wavelengths. The concentrations of Ca III at 620.7nm, Sc II at 635.86nm, and P I at 1058.91nm were the greatest, while the proportions of other elements were low.

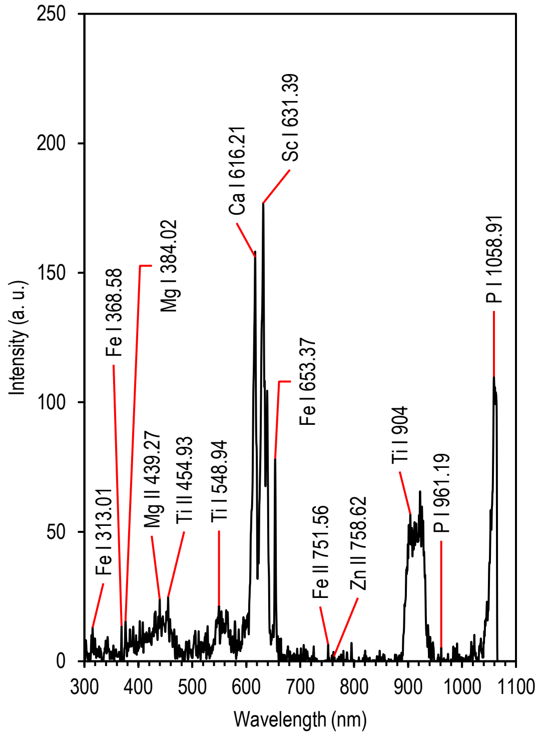

Fig. (2) LIBS spectra of children's teeth specimen 1 at 600mJ

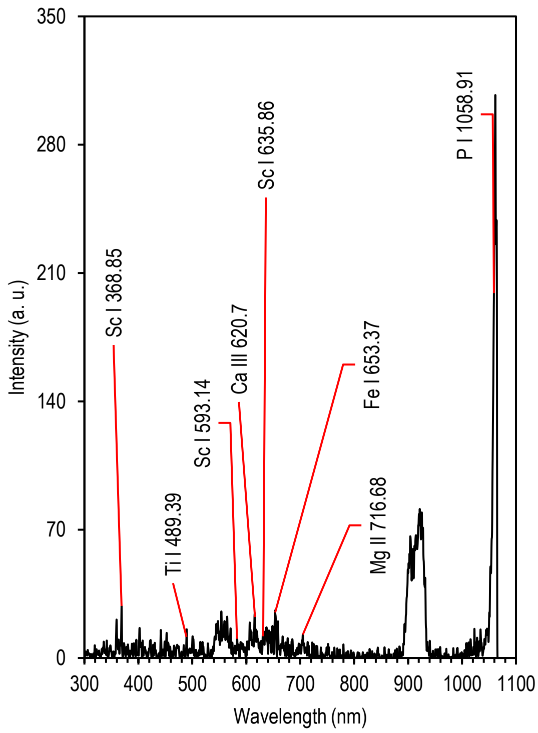

Fig. (3) LIBS spectra of children's teeth specimen 2 at 600mJ

The plasma emission spectra of the third dental specimen in the spectral range of 300-1100 nm, acquired using the LIBS technique, are shown in Fig. (4). The spectral lines record different components, each of which is emitted at a different wavelength. These elements included PI, Fe II, Sc I, Sc II, Ti I, and Ca II. The concentrations of Sc II at 635.86nm and Fe II at 633.13nm were the highest, while the concentrations of other elements were low.

However, in all the aforementioned samples, XRF analysis revealed the presence of only three elements: Ca, P, and Sc, where the concentration of Ca is the highest while the other two elements were slight.

These results indicate the superiority of the LIBS method over the XRF method for the identification and detecting of several elements in dental samples, including Ti, Zn, Mg, and Fe, which were not detected by XRF; in addition, while the XRF method detected much low concentrations of P and Sc, the LIBS method showed much higher concentrations for the same elements, which indicates that the LIBS method is accurate and highly elementally sensitive for more complete elemental analysis in pediatric teeth.

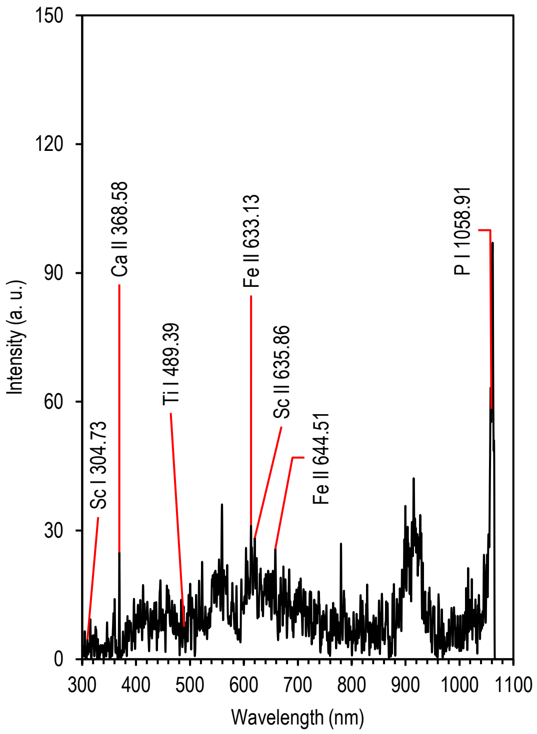

Fig. (4) LIBS spectra of children's teeth specimen 3 at 600mJ

The unique principle of LIBS to generate laser-induced plasma, excite different sample materials, and release unique spectral lines for fast and accurate analysis is what made the LIBS method superior to the XRF method in this study. XRF analysis, on the other hand, it depends on the ability of the material to absorb and re-emit X-rays, which provides information about the amount of elements in the sample but it may be less detailed than LIBS in determining the amount of various elements. Thus it is possible to determine the elemental composition by analyzing the spectral lines. XRF approach show only Ca, P, and Sc. This is a restricted study because, compared to the LIBS method, XRF has inherent limitations related to complete and accurate elemental composition.

Since the LIBS method is widely used to understand the elemental composition, it provides better statistics. This can be an opportunity to learn about the potential environmental, oral health and public outcomes of exposure from which many pediatric neurological researchers could benefit as well. The specific microscopic functions of the LIBS enable dentists to identify probable connections between particular micro-particles and oral issues, which make treatment and prevention methods possible.

In the end, as revealed by this study, for pediatric teeth evaluation a LIBS approach is clearly more accurate than an XRF method. The LIBS spectra revealed various materials on the basis of their spectrum emission lines. The XRF approach revealed only the presence of Ca, P, and Sc. These findings support the premise that LIBS holds a much promise to be an excellent analytical instrument in pediatric dental research and clinical demonstrations with great elemental sensitivity enabling complete composite of elements.

4. Conclusion

Based on the study and the results conducted in this research, the following facts were concluded. The LIBS efficiency surpassing the XRF efficiency by detecting a wide range of elements such as Ca, P, Sc, Fe, Mg, Zn, and Ti. The analysis of children's teeth by LIBS method gives a perfect and accurate diagnosis. The specimen preparation is not required and this decreases time, effort, and cost. The LIBS is a promising technique as an important analytical tool for the teeth analysis.

References

[1] O. Samek et al., "Clinical application of laser-induced breakdown spectroscopy to the analysis of teeth and dental materials", *J. Clinic. Laser Med. Surg.*, 18 (2000) 281-289.

[2] M.S. Doğan, "Relation of trace elements on dental health", in H. Saleh and E. El-Adham (Ed.), **"Trace Elements-Human Health and Environment"**, IntechOpen (2018) pp. 71-83.

[3] M.T. Hussein and W.D. Jalal, "The Short Laser Pulse to Create Micro-Plasma for Identification of Kidney Stone Elements", *Indian J. Nat. Sci.*, 8 (2018) 13537-13543.

[4] P. Pořízka et al., "Feasibility of laser-induced breakdown spectroscopy to elucidate elemental changes in human tooth ankylosis", *Spectrochimica Acta Part B: Atom. Spectro.*, 206 (2023) 106727.

[5] K. Keerthi et al., "Elemental analysis of liquid samples by laser induced breakdown spectroscopy (LIBS): Challenges and potential experimental strategies", *Opt. Laser Technol.*, 147 (2022) 107622.
[6] Priyanka et al., "Validity of laser induced breakdown spectroscopy (LIBS) in determining age and sex from tooth specimens", *Heliyon*, 8 (2022) 1-7.
[7] E. Marguí, I. Queralt and E.d. Almeida, "X-ray fluorescence spectrometry for environmental analysis: Basic principles, instrumentation, applications and recent trends", *Chemosphere*, 303 (2022) 1-18.
[8] A. Olszewska and A. Hanć, "The potential of trace elements mapping in child's natal tooth by laser ablation-ICPMS method", *J. Enviro. Health Sci. Eng.*, 19 (2021) 379-388.
[9] A. Khalid et al., "Laser-induced breakdown spectroscopy analysis of human deciduous teeth samples", *Lasers in Med. Sci.*, 30 (2015) 2233-2238.
[10] A. Hanć, A. Olszewska and D. Barałkiewicz, "Quantitative analysis of elements migration in human teeth with and without filling using LA-ICP-MS", *Microchemical J.*, 110 (2013) 61-69.
[11] M. P. Gutierrez-Salazar and J. Reyes-Gasga, "Microhardness and chemical composition of human tooth", Materials Research, 6 (2003) 367-373.

