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Fatima H. Salih
Department of Physics, College of Science for Women, University of Baghdad, Baghdad, Iraq, fatima.hadi2104m@csw.uobaghdad.edu.iq

Shaimaa S. Mahdi
Department of Physics, College of Science for Women, University of Baghdad, Baghdad, Iraq, shaimaa.saleh@CSW.uobaghdad.edu.iq

Alaa H. Ali
Ministry of Science and Technology, Baghdad, Iraq, alaa.spectro@hotmail.com

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RESEARCH ARTICLE

Employment of Laser Induced Breakdown Spectroscopy to Determine Elements of Human Hair

Fatima H. Salih [1,*], Shaimaa S. Mahdi [1], Alaa H. Ali [2]

[1] Department of Physics, College of Science for Women, University of Baghdad, Baghdad, Iraq
[2] Ministry of Science and Technology, Baghdad, Iraq

ABSTRACT

This paper presents an innovative approach for thyroid disease detection leveraging the capabilities of Laser-Induced Breakdown Spectroscopy (LIBS). Thyroid diseases are a global health issue with a significant impact on millions of people worldwide. Traditional diagnostic methods, such as blood tests, can be invasive, costly, and time-consuming. In contrast, LIBS provides a quick, non-destructive, and cost-effective approach to illness identification. The study uses LIBS to analyze human hair's elemental composition, revealing elemental signatures linked to thyroid disease through laser ablation and spectral emissions. This method provided data that demonstrated the distinction between the control and infected patients by precisely identifying elemental changes in hair samples. The spectra lines of copper (Cu), zinc (Zn) and selenium (Se) were determined to identify thyroid disorders. The diagnosis and therapy of thyroid problems may be completely changed with LIBS. This non-invasive method not only enhances patient outcomes but also creates opportunities for future developments in personalized medicine and medical spectroscopy.

Keywords: Blood analysis, Hair testing, Hypothyroidism, Laser-induced breakdown spectroscopy, Thyroid disease

Introduction

Thyroid disorders are among the most prevalent endocrine diseases globally, impacting millions of individuals across various age groups, genders, and ethnicities. The thyroid gland plays a crucial role in metabolism, growth, and development, thereby. Making thyroid disorders a significant public health concern.[1] Traditional diagnostic methods involve blood tests to measure thyroid-stimulating hormone (TSH), thyroxine (T4), and triiodothyronine (T3) levels. However, these methods can be invasive, expensive, and often fail to detect the disease in its early stages. There is, therefore, a pressing need for innovative, non-invasive, and accurate diagnostic techniques.[2]

This study explores the potential of Laser-Induced Breakdown Spectroscopy (LIBS) as an alternative diagnostic tool for thyroid diseases through the elemental analysis of human hair. Previous studies represent the cornerstone of this research, several studies have investigated the use of LIBS to analyze hair samples for diagnosing and screening thyroid disorders. Abnormal thyroid hormone levels in patients can alter the uptake and secretion of various elements by the body, potentially changing their levels in hair. Researchers have hypothesized that LIBS detection of changes in the elemental composition of hair samples could differentiate patients with thyroid disorders from healthy individuals,[3] another study published in Spectrochemical Acta Part B in 2017 analyzed hair samples from 50 patients with

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* Corresponding author.
E-mail addresses: Fatima.Hadi2104m@csw.uobaghdad.edu.iq (F. H. Salih), shaimaa.saleh@csw.uobaghdad.edu.iq (S. S. Mahdi), alaa.spectro@hotmail.com (A. H. Ali).

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hyperthyroidism, 50 patients with hypothyroidism and 50 healthy controls using LIBS. Another study reported that LIBS could differentiate patients with thyroid disorders from healthy controls based on differences in the levels of various elements in their hair, including magnesium, calcium, potassium, iron and copper. The researchers achieved 90% accuracy in classifying patients and controls based on their LIBS spectra, suggesting potential for thyroid screening.[4]

Biological significance of hair

Hair, often regarded as a mere physical attribute, in fact holds a complex biological structure offering an abundance of information about an individual's health status, nutritional profile and environmental exposure.[5] Deepening our understanding of human hair lies in elucidating the primary minerals found within its cortex, medulla and cuticle layers. Elements such as zinc, copper and selenium form part of the basic mineral composition of hair, playing critical roles in maintaining healthy hair growth and integrity.[6]

Recent scientific findings highlight the notion that deficiencies in these key minerals may directly contribute to the prevalence of thyroid gland disorders.[7] For instance, low zinc levels have been associated with hypothyroidism and subclinical hypothyroidism. Copper deficiency has also been linked to impaired thyroid hormone metabolism and abnormal thyroid function.[8] Similarly, selenium inadequacy is implicated in the etiology of Hashimoto's thyroiditis. These findings underscore the need for accurate, efficient techniques to quantify zinc, copper and selenium concentrations in human hair, offering valuable insights into an individual's mineral status and potential thyroid dysfunction.[9]

Among available techniques, laser-induced breakdown spectroscopy (LIBS) stands out as a promising option for trace element analysis of human hair. LIBS operates on the principle of generating a micro plasma on the sample surface using an intense laser pulse, allowing for rapid, minimally invasive determination of elemental composition. Despite its numerous advantages, LIBS remains underutilized for human hair analysis.[10]

Purpose of the research and expected outcomes

The purpose of this research is to exploit LIBS for analyzing human hair, specifically focusing on quantifying the levels of zinc, copper, and selenium. The overarching aim is to unravel the correlation between the mineral composition of hair and the incidence of thyroid gland disorders. This endeavor is set to bridge the gap between LIBS technology and medical diagnostics, thereby offering a novel approach to non-invasive health monitoring.

Employing LIBS in this context opens up an innovative avenue for understanding and diagnosing health disorders. We anticipate that the outcomes of this research will not only augment the existing body of knowledge but also set the stage for new opportunities in the application of LIBS in medical diagnostics and preventive-healthcare.

Materials and methods

The research was carried out for people with hypothyroidism diseases in addition to the control. The laboratory blood test was required to ensure that they have only a hypothyroidism problem, which means that the thyroid hormone is below the normal range. Hair samples were obtained from women with hypothyroidism between the ages of 40, 50 years, and the control (30 years old) that does not suffer from hypothyroidism. For the purpose of conducting the examination accurately, the samples that were taken from the hair roots were prepared, ground, and a layer of KBr was pressed to be used as a base on which to place the sample for the purpose of giving the possibility of lifting the sample and placing it in front of the laser beam and to avoid the process of disintegration of the sample when bombarded with high energy. In practice, the hair sample was placed on a layer of potassium bromide salt (KBr) and re-pressed, thus obtaining a sample with a solid and transparent base covered by the hair material from the upper surface.

Laser-induced breakdown spectroscopy (LIBS)

Against the backdrop of the need for efficient elemental and isotopic analysis techniques, Laser-Induced Breakdown Spectroscopy (LIBS) emerges as a compelling option. The fundamental principle of LIBS revolves around focusing a high-intensity laser pulse onto the target material, resulting in the ablation and vaporization of surface atoms.[11] When the laser radiation exceeds the material's breakdown threshold, an intensely hot plasma is generated. This plasma consists of free electrons, ions, atoms and photons that are elevated to excited states.[12] As the plasma cools down, the excited species return to lower energy levels by emitting electromagnetic radiation in the form of light. The emission spectrum of this light consists of discrete spectral lines that uniquely identify the elemental composition of the sample.[13]

$$\Delta E = h\nu = hc/\lambda \quad (1)$$

Where h is Planck's constant, ν is the frequency, λ is the wavelength, and c is the speed of light. Each element emits light at specific wavelengths. By analyzing the intensities and wavelengths of the spectral lines using calibration curves, the concentrations of elements within the sample can be quantified. Additionally, the isotopic composition may be deduced from subtle wavelength shifts in the spectral lines.[14] Some of the key advantages of LIBS include its simplicity of use, rapid analysis times, minimal sample preparation requirements, and its versatile application to solid, liquid and gaseous samples. In addition, LIBS is capable of multi-element and spatially-resolved detection, features that have enabled its application across diverse fields.[15–17]

Plasma parameters

The core operation of LIBS involves measuring the light emitted from the plasma. Each element in the plasma emits light at a unique set of wavelengths, thereby enabling the determination of the elemental composition of the sample based on the detected spectral lines. Owing to its rapid, multi-elemental analysis capabilities and minimal sample preparation requirement, LIBS has found extensive applications in diverse fields including archaeology, industrial process control, and environmental monitoring.[18–20] The intensity of the emission lines follows the Boltzmann distribution equation:[21]

$$N_2/N_1 = \left(g_2/g_1\right)\exp\left(-\Delta E/kT\right) \tag{2}$$

Where N_1 and N_2 are the population densities of the upper and lower states, g_1 and g_2 are the degeneracies, ΔE is the energy difference between levels, and T is the plasma temperature. Furthermore, the electron density in the plasma is related to the temperature via the Saha equation:[22]

$$n_e = (2\zeta i/\zeta a)^{3/2}\left(2\pi mekT/h^2\right)^{3/2}\exp\left(-\Delta E_i/kT\right) \tag{3}$$

Where n_e is the electron density, ζi and ζa are the partition functions, ΔEi is the ionization energy, me is the electron mass, and the other variables are as previously defined. To achieve local thermodynamic equilibrium (LTE) for straightforward analysis, the plasma must meet density and temperature thresholds:[23]

$$n_e \geq 1 \times 1016\,\text{cm}^{-3}, T \geq 10{,}000\,\text{K} \tag{4}$$

Under LTE conditions, the plasma transitions follow a Boltzmann distribution. By incorporating these fundamental equations, the relationships between the measured LIBS emission spectra, plasma conditions, and elemental composition are highlighted.

Electron temperature (T_e) and plasma density (n_e), which are interrelated according to the plasma equation:[24]

$$n_e * T_e = P/(k * (1 + n_e * \lambda_D^3)) \tag{5}$$

where n_e is the electron number density, T_e is the electron temperature, E ion is the ionization energy, electron temperature signifies the average kinetic energy of free electrons, reflecting the excitation and k is the Boltzmann constant and C is a constant. The ionization levels within the plasma. Higher electron temperatures lead to greater excitation and ionization, resulting in stronger emission intensities. The plasma density, indicating the concentration of free electrons, also governs plasma properties such as its degree of ionization and optical characteristics. Higher electron densities tend to enhance emission intensities by providing more excited species. Thus, optimizing both the electron temperature and density of the plasma generated during LIBS is essential for achieving optimum spectral emission and analytical performance. Various experimental parameters such as laser fluence, pulse width and ambient gas can be manipulated to tailor the plasma parameters and obtain enhanced LIBS signals.[25,26]

LIBS setup

The main component of the LIBS setup is Passively Q-switch Nd:YAG laser with fundamental wavelength 1064 nm and pulse duration 10 ns, pulse energy 100 mJ with single pulse operation. This type of laser is commonly used in LIBS due to its ability to deliver high-energy pulses in a short duration, which is necessary to generate the plasma from the sample. The laser was paired with a spectrometer from OPTOSKY ATP2400, with spectral rang 190–1110 nm: lowest 190 nm ± 10 nm, highest 110 nm ± 10 nm, SNR > 500:1 emphasize incident light to near saturation average energy LRMS and optimal resolution: measuring the Characteristic peak half width of mercury argon lamp. Reference value: should be better than the following values 190–1110 nm: 546.08 nm (1 nm), a device that separates the light emitted by the plasma into its component wavelengths, much like a mirror separates white light into a rainbow as shown in the Fig. 1. Each element in the sample emits light at specific wavelengths, allowing for their identification. A computer was connected with the spectrometer for data acquisition and analyzing.

The laboratory examination of the patients was verified, which gave the percentages of the blood

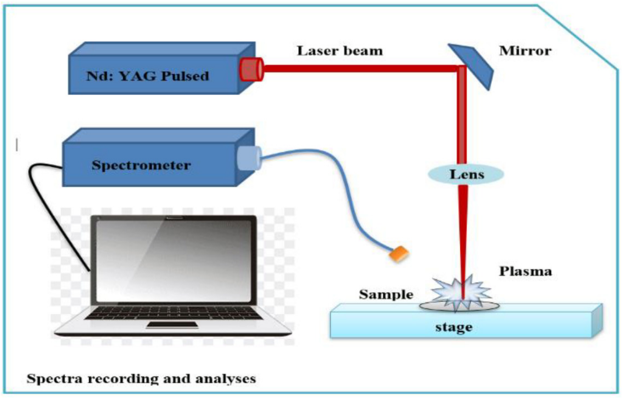

Fig. 1. Shows represent LIBS Setup.

Table 1. It represents the selected samples with the percentage of thyroid hormone in the blood.

The samples	The age	Thyroid blood tests results (T.S.H)	Normal Range
Sample 1(control)	30	1.517	0.38–4.31
Sample 2(Hypothyroidism)	40	5.32	0.4–4.0
Sample 3(Hypothyroidism)	50	14.120	0.38–4.31

test for thyroid hormone. It also includes the normal range that proves the presence of a disease condition and the extent of the disease gradually getting worse from one person to another Table 1. It represents the selected samples with the percentage of thyroid hormone in the blood.

It can't be denied that the blood drawing procedure in laboratories hasn't been efficient over the last years, but the need to discover new technologies that provide scientific and practical benefit, more accurate results, and ease of use is not harmful. This research shed light on the use of physics medically, in various cases as a lifesaver for conducting examination. If the patient suffers from amputation of limbs, or suffers from severe burns. There is also a difference in terms of the time taken, as experience has shown that the blood drawing process takes half an hour until the result is known. In this method, the time is shorter, as one hair is placed in the device and the result appears immediately after storing the database of hair elements that are affected by thyroid diseases. From a financial standpoint, the cost of laboratory materials is more due to the need for disposable materials for each patient. The accuracy of the work is less when analyzing blood due to the need to re-examine more than once or the use of expired materials. Sometimes the sample is destroyed simply because it was forgotten for a short time and the need to repeat the blood draw often causes the patient to fear the pain of the needle prick. This technique is more accurate because the process depends on the vital elements in human hair.

Table 2. Shows Electron temperature T_e (K) and plasma density n_e (cm^{-3}) of the generated plasma of the first sample.

The Elements	Te (K)	n_e (cm^{-3})
Copper (Cu)	1106.64	1.05653E+17
Zinc (Zn)	8277.85	9.16678E+16
Selenium (Se)	1292.33	8.60702E+16

Table 3. Shows Electron temperature T_e (K) and plasma density n_e (cm^{-3}) of the generated plasma of the second sample.

The Elements	Te (K)	n_e (cm^{-3})
Copper (Cu)	1112.94	1.05953E+17
Zinc (Zn)	8536.41	9.30884E+16
Selenium (Se)	1274.61	8.54781E+16

Table 4. Shows Electron temperature T_e (K) and plasma density n_e (cm^{-3}) of the generated plasma of the third sample.

The Elements	Te (K)	n_e (cm^{-3})
Copper (Cu)	9487.32	3.0935E+17
Zinc (Zn)	8665.74	9.37909E+16
Selenium (Se)	1263.67	8.51105E+16

Results and discussion

Plasma, defined as an ionized gas consisting of free electrons, ions and neutral species, exhibits specific properties captured by the plasma equation. Crucial parameters in describing plasma generated during LIBS. The electron temperature T_e (K) and plasma density n_e (cm^{-3}) can be calculated by Eqs. (2), (3) and (5) and the result show in Tables 2 to 4.

Calculation of plasma parameters

Electron temperature T_e (K) and plasma density n_e (cm^{-3}) are the most important properties. These properties are affected by many conditions, such as wavelength, the effect of laser energy, and the target material. Since the creation of high-potential-power lasers, numerous theoretical and empirical studies have concentrated on laser-produced plasma. This plasma is created by focusing a beam of intense laser on a target's surface and then evaporating the target, resulting in very hot and extremely dense plasma.[27] A method for measuring the interaction between laser and plasma was presented and all measurements were performed at room temperature. The plasma spectrum was made by comparing the

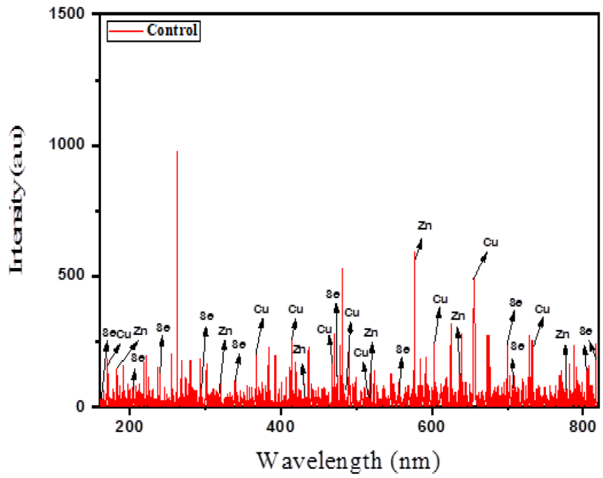

Fig. 2. Shows LIBS spectra of control hair fiber of sample 1.

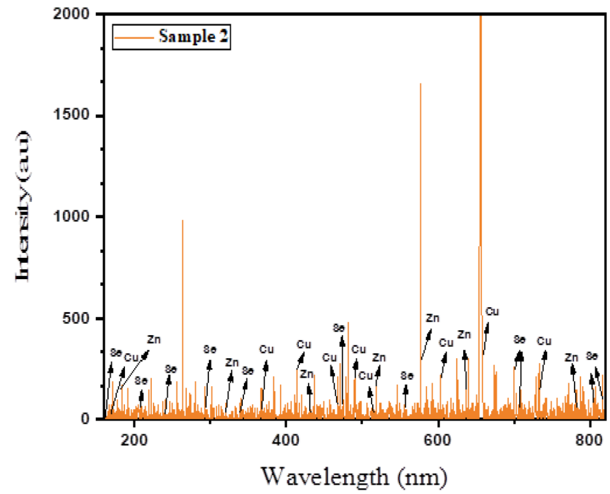

Fig. 3. Shows LIBS spectra of Hypothyroidism hair fiber of sample 2.

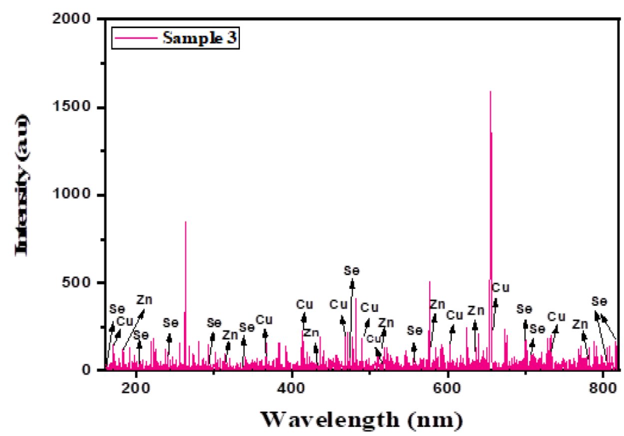

Fig. 4. Shows LIBS spectra of Hypothyroidism hair fiber of sample 3.

recorded wavelengths with National Institute of Standards and Technology (NIST) data. Tables 2 to 4 show the electron temperature and plasma density proving the generation of plasma after laser bombardment of the samples. The emitted intensity increases with laser of energy increases because of the abstraction of laser radiation by plasma, thus increasing radiation and eventually increasing the spectral lines intensity. As the laser energy increased, intense plasmas with higher temperatures were attained. However, only a moderate increase in the density of electrons were perceived.

LIBS spectra

The results of examining hair samples using laser-induced breakdown are analyzed below. Focus was placed on three basic and important elements due to their direct impact on hypothyroidism to detect mineral changes.

Fig. 2 shows the atoms assignments of sample 1 (Control) single hair fiber and the LIBS spectra of which represents the basis for comparison between infected samples.

Figs. 3 and 4 show LIBS spectra of Hypothyroidism hair fiber. Human hair is a biomarker, providing a non-invasive way to reflect nutritional, environmental, and physiological factors. Because of its unique structure and growth pattern, it can serve as a biological record, including the main elements found in the hair root during the growth phase. It remains stable there for a long time. Changes in their levels indicate a defect in the health condition. Therefore, it can be used to detect hypothyroidism. The most important of these elements are zinc (Zn), copper (Cu), and selenium (Se), referred to in Tables 5 to 7. These elements play crucial roles in various physiological processes, such as enzyme activity, immune function, and antioxidant defense. Zinc is involved in DNA synthesis and cell division, while copper is essential for the formation of connective tissues and iron metabolism. Selenium acts as a cofactor for antioxidant enzymes and plays a vital role in thyroid hormone metabolism. Monitoring the levels of these elements in hair can provide valuable insights into an individual's nutritional status and potential health issues related to hypothyroidism.[28]

The comparison was made in terms of the difference in intensity between one sample and another, as shown in Fig. 5. A gradual decrease in the intensity of the elements was observed between the control condition and the affected conditions for the same wavelengths. As the element zinc (Zn) at wavelengths 183.227–317.204 (nm) then the severity gradually decreases for cases with hypothyroidism, according to the incidence rate mentioned in Table 1. As for selenium (Se), at a wavelength of 293.009 (nm), it

Table 5. LIBS measurements show the atoms assignments of sample 1 (Control) single hair fiber.

S. No	Wavelength (nm)	The Elements
1	162.281, 205.616, 241.368, 295.176, 339.594, 473.933, 474.655, 556.631, 699.998, 706.137, 803.641, 816.281.	Se
2	170.226, 367.401, 413.986, 468.155, 489.823, 515.463,603.216, 655.58, 733.222.	Cu
3	183.227, 317.204, 429.153, 518.352, 577.215, 636.44, 779.085.	Zn

Table 6. LIBS measurements show the atoms assignments of sample 2 single hair fiber.

S. No	Wavelength (nm)	The Elements
1	162.281, 205.616, 241.368, 295.176, 339.594, 473.933, 474.655, 556.631, 699.998, 706.137, 803.641, 816.281.	Se
2	170.226, 367.401, 413.986, 468.155, 489.823, 515.463,603.216, 655.58, 733.222.	Cu
3	183.227, 317.204, 429.153, 518.352, 577.215, 636.44, 779.085.	Zn

Table 7. LIBS measurements show the atoms assignments of sample 3 single hair fiber.

S. No	Wavelength (nm)	The Elements
1	162.281, 205.616, 241.368, 295.176, 339.594, 473.933, 474.655, 556.631, 699.998, 706.137, 803.641, 816.281.	Se
2	170.226, 367.401, 413.986, 468.155, 489.823, 515.463,603.216, 655.58, 733.222.	Cu
3	183.227, 317.204, 429.153, 518.352, 577.215, 636.44, 779.085.	Zn

was noted that the intensity for the control sample was 183.6471 (a.u). As for the second sample, it decreased to reach 168.281 (a.u). As for the third sample, it gave an intensity of 153.8324 (a.u). The same is true for copper (Cu) at wavelengths 481.227–603. 216–728.888 (nm). It is observed in Fig. 5 that cases of severe copper deficiency led to poor metabolism of thyroid hormone, which ultimately leads to hypothyroidism. Scientific evidence has proven that people with high levels of selenium suffer from fewer thyroid problems. The thyroid gland plays a role in the normal use of zinc. Many studies have found that zinc helps convert thyroid hormone into an active and beneficial form in the body, which in turn directly affects hair. Furthermore, zinc deficiency has been associated with hair loss and impaired

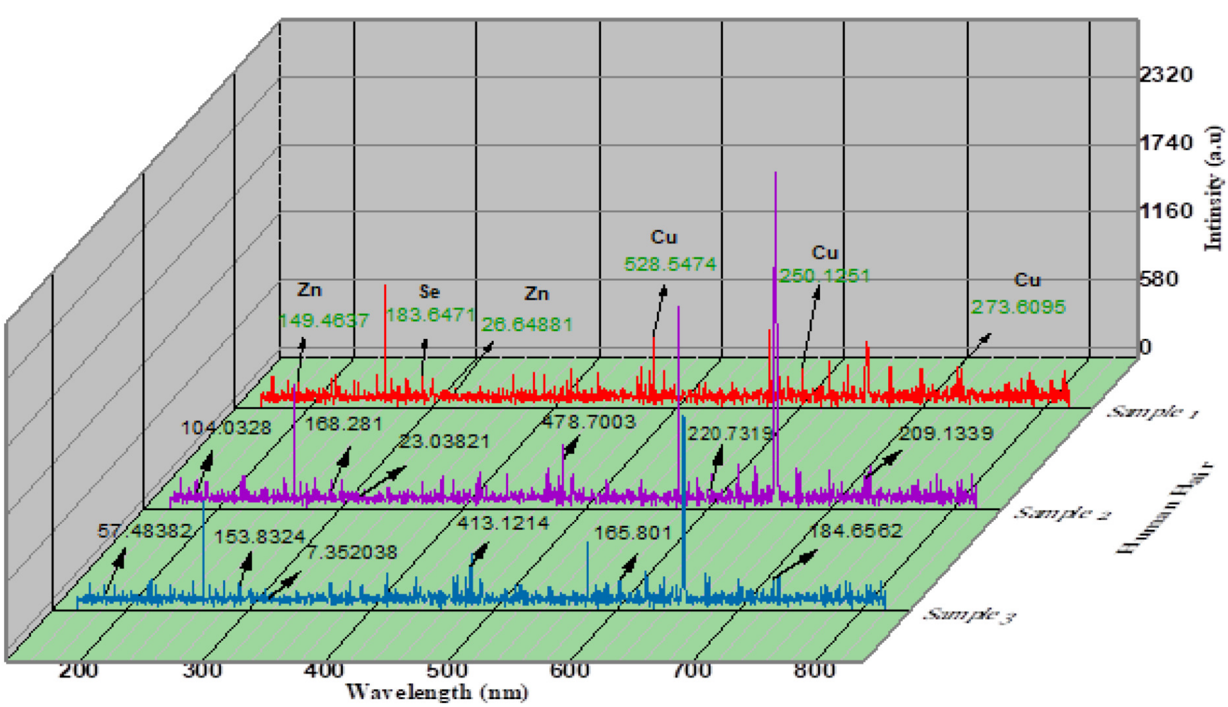

Fig. 5. Shows 3D plot spectra of LIBS measurements.

hair growth. Therefore, maintaining adequate levels of zinc is crucial for optimal thyroid function and healthy hair. Additionally, selenium has been shown to have antioxidant properties that protect the thyroid gland from oxidative stress, further supporting its role in thyroid health and hair growth.[29]

Conclusion

This research set out to explore the potential of LIBS analysis of hair samples as a diagnostic tool for thyroid diseases. The steps involved included participant recruitment, hair sample collection and preparation, LIBS analysis, and elemental analysis. He provided insights into the feasibility and potential of using LIBS as a non-invasive diagnostic tool for thyroid diseases. The spectral lines of the elements copper, zinc, and selenium were determined, and the electron temperature and plasma density were studied. The difference was reached between the control and the two cases with hypothyroidism, by comparing the intensity of the elements that most affect the healthy and affected person where are the Low levels of copper, zinc, and selenium associated with lower element intensities for each sample lead to poor metabolism of thyroid hormone which leads to an underactive thyroid gland. This physical technique has proven its efficiency and accuracy in diagnosing disease, and it could have a promising future in the medical domain and treatment.

Author's declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication, which is attached to the manuscript.
- Authors sign on ethical consideration's approval.
- No animal studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Considerations: All the participants involved in the study provided informed consent before the collection of hair samples. This ensured that they were fully aware of the nature and purpose of the study, the procedures involved, and their rights as participants. The study was conducted in accordance with the ethical guidelines of the Helsinki Declaration. This includes principles related to voluntary participation, informed consent, confidentiality, and the right to withdrawal.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Authors contributions

The study conception and design: A. H. A.; data collection: F. H. S.; analysis and interpretation of results: F. H. S., S. S. M.; draft manuscript preparation: F. H. S., S. S. M. All authors reviewed the results and approved. the final version of the manuscript.

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تحديد مكونات شعر الإنسان باستخدام مطياف الانهيار الناجم عن الليزر

فاطمة هادي صالح [1] ، شيماء صالح مهدي[1] ،علاء حسين علي[2]

[1] قسم الفيزياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق.
[2] وزارة العلوم والتكنلوجيا، بغداد، العراق.

الخلاصة

يقدم هذا البحث طريقة مبتكرة للكشف عن أمراض الغدة الدرقية من خلال الاستفادة من قدرات التحليل الطيفي للانهيار المستحث بالليزر (LIBS). تعد أمراض الغدة الدرقية مشكلة صحية عالمية ولها تأثير كبير على ملايين الأشخاص في جميع أنحاء العالم. يمكن أن تكون طرق التشخيص التقليدية، مثل اختبارات الدم، غازية ومكلفة وتستغرق وقتًا طويلاً. في المقابل، يوفر LIBS طريقة سريعة وغير مدمرة وفعالة من حيث التكلفة لتحديد المرض. تستخدم الدراسة LIBS لتحليل التركيبة الأولية للشعر البشري، والكشف عن التوقيعات الأولية المرتبطة بمرض الغدة الدرقية من خلال الاستئصال بالليزر والانبعاثات الطيفية. قدمت هذه الطريقة بيانات أظهرت التمييز بين المجموعة الضابطة والمرضى المصابين عن طريق تحديد التغيرات العنصرية في عينات الشعر بدقة. تم تحديد الخطوط الطيفية للنحاس (Cu) والزنك (Zn) والسيلينيوم (Se) لتحديد اضطرابات الغدة الدرقية. قد يتم تغيير تشخيص وعلاج مشاكل الغدة الدرقية بالكامل باستخدام LIBS. لا تعمل هذه الطريقة غير الجراحية على تحسين نتائج المرضى فحسب، بل تخلق أيضًا فرصًا للتطورات المستقبلية في الطب الشخصي والتحليل الطيفي الطبي.

الكلمات المفتاحية: تحليل الدم، اختبار الشعر، قصور الغدة الدرقية، التحليل الطيفي للانهيار الناجم عن الليزر، مرض الغدة الدرقية.