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Correlation between the Mechanical Properties and Laser-Induced Breakdown Spectroscopy for Human Teeth

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Abstract

The present study investigates the correlation between mechanical properties and laser-induced breakdown spectroscopy (LIBS) for different tooth samples. A set of Fourteen samples collected from the Al-Kut Specialized Dental Centre in Wasit Governorate was analysed. Tooth samples were divided into groups depending on age and gender (men, women). The optical emission of the ionic-to- atomic intensity ratio of the Ca spectral line has been correlated with Leeb hardness test (LHT) values, compressive strength, and Young's modulus measured by standard tools. The ratio ionic line to atomic line CaII / CaI between the ionic calcium line at 396.84 nm and the atomic line at 558.87 nm for teeth is obtained using LIBS technique employing Nd: YAG laser with a wavelength of 1064 nm, an energy of 200 Mj, and a pulse duration of 10 ns. A linear relationship was observed between compressive strength, Young's modulus and hardness, and the concentrations of the elements (Ca). The small relative errors for hardness, compressive strength, and Young's modulus ranged from (1.44-5.57) %, (1.94-6.07) %, and (0.243-4.01) %, respectively. These findings validate applying LIBS to check out some important mechanical properties.

Keywords: Human tooth, hardness, LIBS, mechanical properties

ارتباط الخصائص الميكانيكية مع التحليل الطيفي للانهيار الناجم عن الليزر لأسنان الإنسان

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الخلاصة

تبحث الدراسة الحالية في ارتباط الخواص الميكانيكية ومطياف الانهيار الناتج عن الليزر (LIBS) لأجزاء مختلفة من عينات الأسنان. تم تحليل مجموعة مكونة من اربع عشر عينة تم جمعها من مركز الكوت التخصصي لطب الأسنان بمحافظة واسط. تم تقسيم عينات الأسنان إلى مجموعات حسب العمر و الجنس (رجال ، نساء). تم ربط الانبعاث البصري لنسبة الكثافة الأيونية إلى المتعادلة للخط الطيفي Ca بالقيم المشتركة لاختبار الصلادة LEEB وقوة الانضغاط ومعامل Young المقاسة بواسطة الأدوات القياسية. يتم الحصول على نسبة CaII / CaI بين خط الكالسيوم الأيوني عند 396.84 نانومتر والخط المتعادل عند 558.87 نانومتر للأسنان ، باستخدام LIBS (ليزر Nd: YAG) بطول موجة 1064 نانومتر وطاقة 200 مللي جول ومدة نبضة تبلغ 10 نانوثانية. لوحظ وجود علاقة خطية بين مقاومة الانضغاط ومعامل يونغ والصلادة وتركيزات العناصر (Ca). تراوحت الأخطاء النسبية الصغيرة للصلادة وقوة الانضغاط ومعامل يونغ

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من (4.52–1.44)٪ و (6.07–2.89)٪ و (2.12–0.455)٪ على التوالي. تحقق هذه النتائج من صحة تطبيق LIBS لفحص بعض الخصائص الميكانيكية المهمة.

1. Introduction

Laser-Induced Breakdown Spectroscopy (LIBS) is a multi-element analytical technique that can determine the elemental composition of almost all materials [1]. This technique relies upon the spectroscopic analysis of the radiation emitted by laser-induced plasma [2]. LIBS provides a precise, fast and in situ analysis with a minute or no sample preparation [3]. Multiple applications of LIBS have constantly been growing and extended from lab to field for qualitative and quantitative elemental analysis in solids, studying the trace elements contamination of soils, and more, recently, fingerprinting for characterization of materials, as examples, but not limited [4-6]. Even though extensive studies have been published concerning industrial applications [7], the focus on the biomedical applications of LIBS remains rare [8,9]. LIBS technique can examine the chemical composition of biological samples such as bones, blood, and tissues. It can also identify hazardous materials as well as mineral excesses or deficiencies in tissues, teeth, nails, or bones. Similarly, LIBS is able to identify cancer and serves as a clinical equipment that simultaneously detects and eliminates tumor cells [10, 11]. Samek et al. [12] highlighted the applicability of the LIBS technique for the examination of minerals and potentially harmful components in calcified tissues like bones and teeth. Using lasers in dentistry is considered a new technology used in clinical dentistry to address some of the issues with traditional dental procedures [13].

It is important to understand the properties of teeth since they have significant clinical ramifications in dentistry. In particular, understanding the mechanical characteristics of teeth aids in predicting the stress and strain that teeth experience when in contact with other materials. The hardness of a tooth, as one of the mechanical properties, has been studied using different techniques by several researchers. Surface hardness, defined as the resistance of the surface to wear, is one of the most important general characteristics [14]. The main tests most frequently used in determining hardness are Knoop, Brinell, Shore, Vickers and Barcol [15]. Abdel-Salam et al. have estimated human teeth hardness via magnesium and calcium ionic to atomic intensity of line ratio in LIBS spectra; their results suggested the feasibility of the quantitative estimation of hardness for sample tissues [16]. A recent article described the measurement of dentin's Young's modulus using a nanoindentation technique. This technique comprises recording the indentation depth during the loading and unloading cycles. The load-displacement curve derived from these values can be used to determine Young's modulus automatically. Inaccurate optical measurements of the diagonal lengths of minor indentations are eliminated [17].

This work aimed to evaluate the mechanical properties of human teeth samples of different ages and gender using the LIBS technique. The peak intensities of calcium were analysed to establish the correlation between the spectral characteristics and mechanical properties (hardness, compressive strength, and Young's modulus) via estimation of the intensity ratio of ionic to atomic spectral lines in LIBS spectra. This will serve as the basis for developing a rapid detection technology.

.2. Materials and Methods

In this study,Fourteen extracted human molars from the Al-Kut Specialized Dental Centre in Wasit Governorate were used after obtaining the informed consent of patients were used. These samples were washed in distilled water, each tooth was wiped to remove any debris, and then dried at room temperature; all samples were stored in numbered sealed pots. Six teeth, as listed in Table 1 and shown in Figure 1, were arbitrarily selected to be used in drawing a calibration curve of the ratio of the calcium spectral lines intensity against teeth hardness values measured using the conventional Leeb method . Energy Dispersive X-ray spectroscopy (Model: INSPECT S50) was used to analyse the elemental composition of the examined teeth.

Table 1 : Distribution of tooth samples by age and gender.

No. Sample	Age	Type	Teeth name
T1	60	Male (smoker)	Molar
T2	31	Male (smoker)	Molar
T3	43	Male (non -smoker)	Molar
T4	40	female	Molar
T5	20	female	Molar
T6	13	Male	Molar

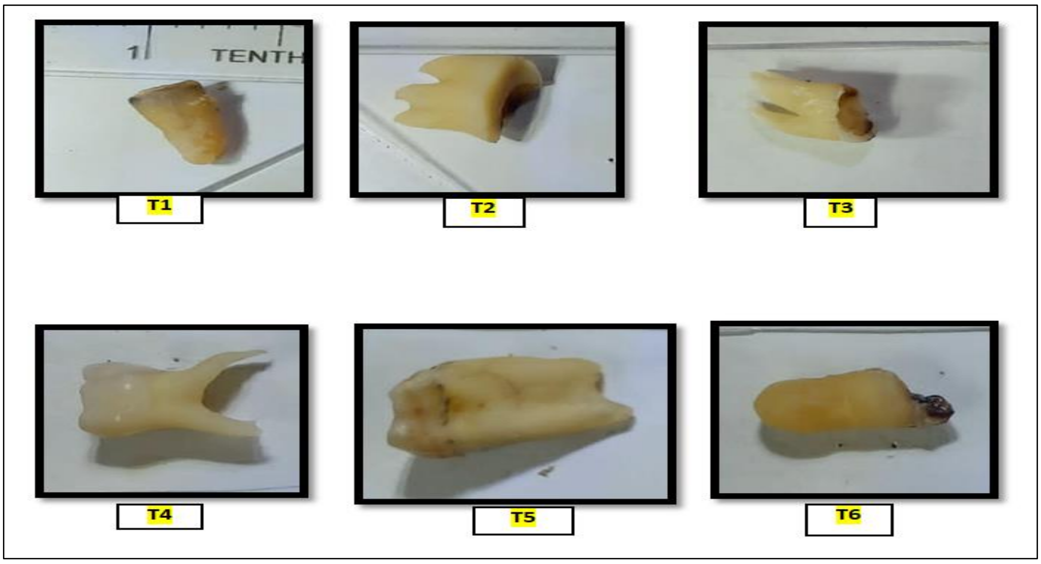

Figure 1: photographs of tooth Samples

A schematic of the LIBS system used in this work is shown in Figure 2. This setup consists of

- Nd: YAG laser operating at wavelength 1064 nm with a pulse width of 9 ns and a pulse energy of 200 mJ,
- Focusing and collecting optics.
- Sample platform.
- LIBS detection system (spectrometer and detector).

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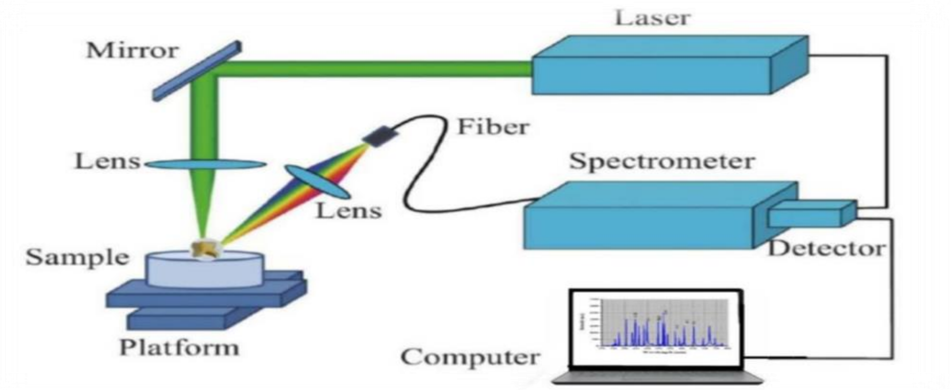

Figure 2 : Experimental set-up for LIBS measurements

3. Results and Discussion

3.1 Energy Dispersive X-ray spectroscopy (EDS)

The teeth samples were examined with the Energy Dispersive X-ray spectrometer to determine their elemental composition. The EDS is a powerful technique to determine the chemical composition. The element distribution in the tested sample is shown in a spectrum containing peaks corresponding to the elements in the examined sample. The EDS spectrum of teeth T1 is shown in Figure 3, where peaks corresponding to the elements (Ca, Mg, and P) are noted. The EDS spectra of the six teeth samples were recorded, from which the concentration percentage of the constituent elements in the teeth were extracted, as shown in Table 2.

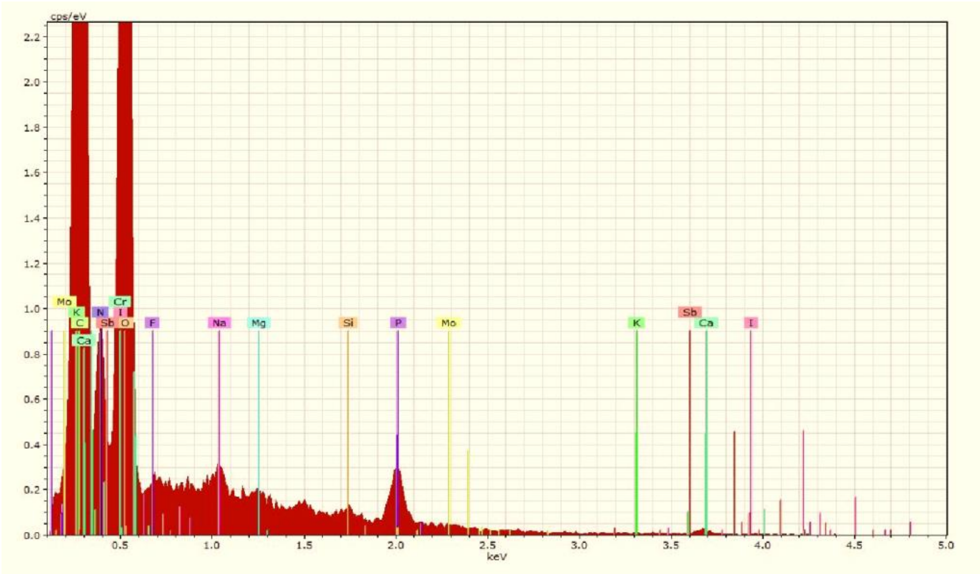

Figure 3: EDS spectrum of tooth T1

Table 2: Element content values (concentration %) of main elements Ca , P, and Mg EDS analysis

No. of Sample	Ca	Mg	P
T1	23.55	0.35	1.30
T2	25.5	0.05	7.19
T3	32.2	0.83	16.70
T4	34.5	0.27	4.83
T5	39.9	0.02	0.79
T6	42.3	0.28	8.66

3. 2 Qualitative Analysis of a tooth

The spectrum of one tooth's sample (T1) using the LIBS technique was recorded at the region of spectral (250-800 nm) for qualitative analysis. The major elements found in the sample T1 examined were C, K, Cr, P, Mn, Ca, F, Si, Zn, Na, and Sn. Amongst many detected lines that appeared in the spectra, the lines of Ca at (396.84, 422.67, 520, 573) nm, Mg at (448.13, 518.36) nm, and P at (602.14, 645.07, 255.29) nm were with the maximum intensity. To get a reliable result, these lines were chosen precisely because they are relatively free from other spectral interferences and were extracted with the help of (the NIST) database [18]. The peak intensities recorded for the selected element's lines were further analyzed to establish a calibration curve (C.C.) for the other examined teeth samples [19]. The LIBS spectrum for the tooth sample (T1) is shown in Figure 4. The LIBS spectrum of the other teeth were also recorded which gave similar results but with differences in the peaks intensity.

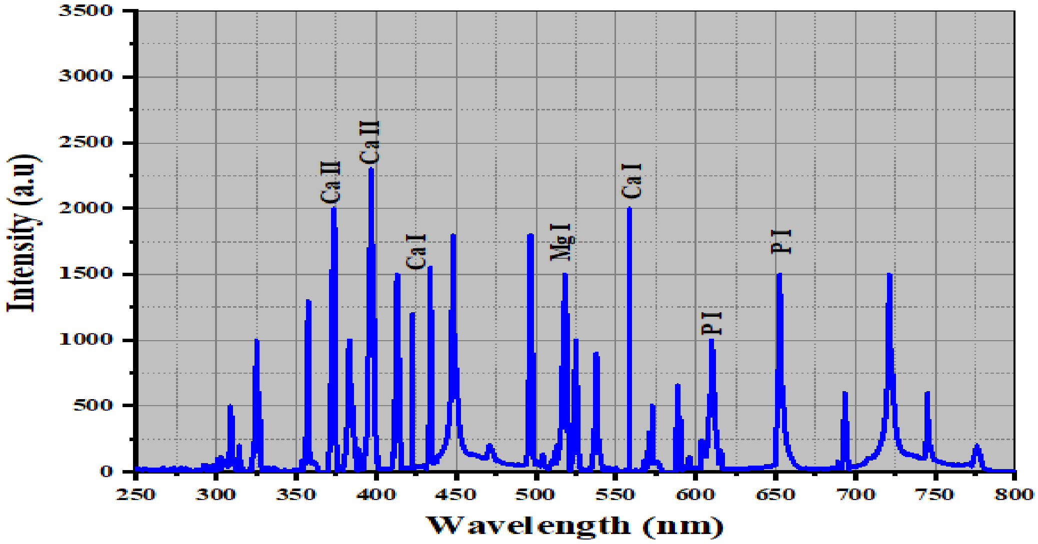

Figure 4: LIBS spectrum of tooth sample (T1) in the region of (250nm-800nm) at 200 mJ.

3.3. Relationship between the spectral intensity and mechanical properties

The hardness test of teeth, as well as other mechanical characteristics like Young's modulus and compressive strength, were carried out with the Leeb hardness tester (HM. 6561). It is a portable, easy-to-carry, and low-cost device. It was used for hardness testing according to (ASTM-E384) method, which is the standard method for measuring the hardness of materials. The surface is subjected to a standard pressure for a standard length of time. The teeth were subjected to pressure along their central axes. Hardness, Young modulus and compression strength are listed in Table.3

Table 3: Hardness, Young modulus and compression strength for the examined teeth samples

No. of Sample	Hardness (HV)	Young modulus (GPa)	Compression strength (MPa)	(Ca II /Ca I)
T1	125	0.164	129	1.9
T2	145	0.163	131	2.1
T3	156	0.164	131.9	2.4
T4	169	0.168	248	2.5
T5	322	0.176	454	2.7
T6	376	0.164	129	2.88

The Ca lines are of interest in the LIBS spectra of the human teeth since they were of the highest intensity, proving that Ca is the major constituent in all samples. Figure 5 shows the relation between Ca concentration calculated from the EDS analysis (Table 2) and the corresponding hardness values measured by the Leeb hardness tester (Table 3). It can be easily noticed that there is a linear link between Ca concentration and hardness, as the hardness values increase with increasing the concentration of calcium in the teeth.

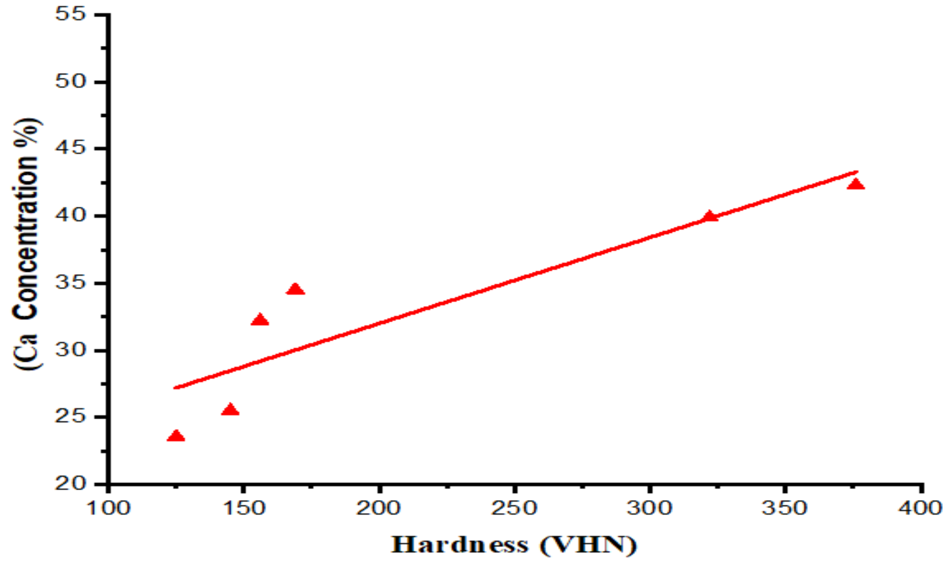

Figure 5: The correlation between the hardness of teeth and Ca concentrations.

The relation between the ratio of ionic to atomic of Ca emission lines (CaII/CaI) from the LIBS spectra of the six samples and their corresponding hardness values is shown in Figure 6. In this case, the preferred spectral line was displayed by plotting the three intensity ratios. The intensity ratio of the CaII at (396.84 nm) to the CaI (422.67 nm) is the first spectral line. The second spectral line shows the CaII at (373.60 nm) ionic line's intensity ratio to CaI at (422.67 nm). CaII at (396.84 nm) and the CaI at (558.87 nm) is the third spectral line. The corresponding values of R^2 are (0.72, 0.95, and 0.99), respectively. As a result, when comparing these three spectral lines, the third one is the best since it has the highest R^2 value of 0.99, so it was employed to determine the mechanical characteristics of the other teeth.

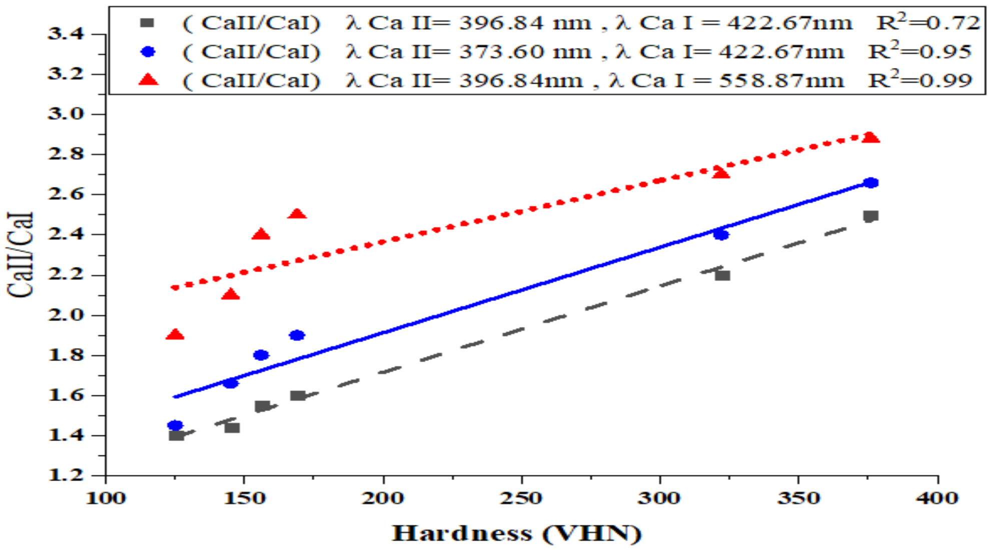

Figure 6: The relationship between CaII/CaI ratio and hardness.

Young's modulus is the most important property that measures the compressive stiffness or tensile of a solid sample when the applied force is below the proportional limit. It indicates the amount of deformation that occurs in a sample tissue when a load is applied to it [19]. In Figure 7, Young's modulus is plotted versus the best spectral line the CaII ionic line in the (396.84 nm) wavelength to the CaI neutral line at (558.87 nm), with (R^2 = 0.94). It was found that the relation is direct; the higher the concentration of calcium, the greater the Young's modulus.

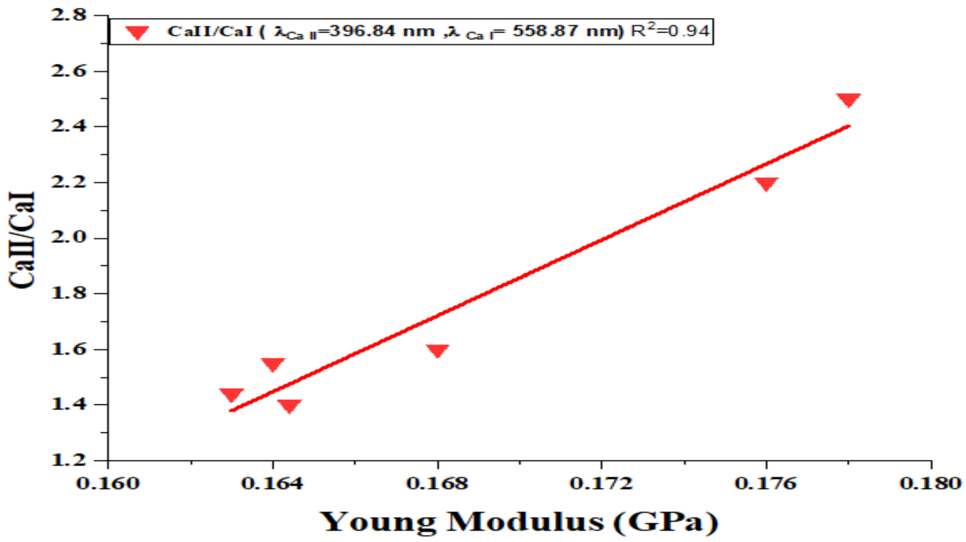

Figure 7: The relationship between CaII/CaI and Young Modulus

Compressive strength is generally defined as the maximum resistance of the material to the applied stress. It is considered a critical indicator of success because a high compressive strength is necessary to resist masticatory and parafunctional forces. The compression strength is shown in Figure 8 against the CaII/CaI ratio (R^2 =0.91) between the Call ionic line at 396.84 nm and the Cal atomic line at 558.87 nm. It turns out that when the ratio of the CaII/CaI increases, compression strength increases.

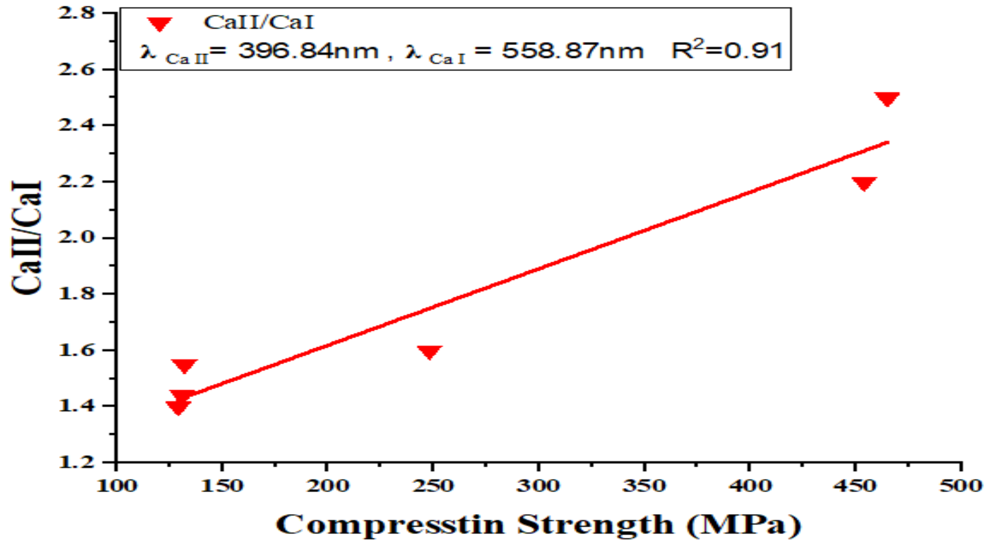

Figure 8: The relationship between CaII/CaI and compression strength

The graphs of Figures. 6, 7, and 8 were used to estimate the mechanical properties of the dental samples. From these figures, the linear relationship was used to estimate the mechanical properties values of unknown samples from their Ca spectral line intensity ratios deduced from their LIBS spectra. The comparison between mechanical properties (hardness, compressive strength, and Young's modulus) measured by Leeb hardness and LIBS procedure are tabulated in Table 4, which were calculated for eight teeth samples. The predicted results were highly acceptable with the relative error for hardness, compressive strength, and Young's modulus, which were in the range (1.44-5.57) %, (1.94-6.07) %, and (0.24-4.01) %, respectively. This observation can have practical importance even though the underlying physical mechanisms still need some clarification.

Table 4: The comparison between mechanical properties (hardness, compressive strength, and Young's modulus) measured by standard tools and by LIBS procedure

Sample	YOUNGS MODULUS IN (GPa)		Error%	COMPRESSION STRENGTH (MPa)		Error%	Vickers HARDNESS IN (HV)		Error%
	LBIS-Y (GPa)	Mec.-Y (GPa)		LBIS -CS (MPa)	Mec.-CS (MPa)		LBIS -H (HV)	Mec.-H (HV)	
T7	0.1610	0.1602	0.499	135	141	4.25	140	138	1.44
T8	0.1640	0.1621	1.172	138	145	4.82	162	155	4.52
T9	0.1661	0.1644	1.040	154	150	2.66	187	195	4.10
T10	0.1681	0.1646	2.126	170	181	6.07	210	204	2.94
T11	0.1723	0.1711	0.701	335	345	2.89	213	219	2.74
T12	0.1788	0.1762	1.475	420	412	1.94	225	220	2.27
T13	0.1723	0.1766	0.243	411	425	3.29	245	233	5.57
T14	0.1701	0.1772	4.01	350	342	2.33	254	261	2.68

4. Conclusion

In this experiment, some mechanical properties of human teeth, such as (hardness, compressive strength, and Young's modulus) were measured by standard tools. At the same time, these teeth were inspected using EDS to determine the elemental composition. The intensity ratios of calcium spectral lines were identified to establish the C.C. by correlating the intensity ratios with the measured mechanical properties. The linear correlations between the measured mechanical properties and intensity ratios and the best spectral, the CaII ionic line in the (396.84 nm) wavelength to the CaI atomic line at (558.87 nm) with (R^2 = 0.94), was utilized to estimate the unknown sample properties. The predicted results were satisfactory, with relative error of (1.44-5.57) %, (1.94-6.07) %, and (0.24-4.01) %. Thus, the obtained results demonstrated a direct proportionality of Ca emission lines with mechanical properties and introduced the LIBS technique as a reliable evaluation method.

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