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Phyllanthus Emblica Leaves and Fruits: A Spectroscopic Investigation Using Laser-Induced Breakdown spectroscopy (LIBS), x-ray Fluorescence (XRF) and Inductively Charged Plasma Mass Spectroscopy (ICP-MS)

Ubaid Ullah Khalil[1], Zahid Muhammad[1]*, Junaid Nisar[1], Nimra Shahzad[2], Madma Habib[3], Samra Naz[1]

[1]Department of Physics, Hazara University Mansehra, KPK Pakistan
[2]Department of Physics, University of Engineering and Technology Lahore, Pakistan
[3]Department of Chemistry, University of Sargodha, Punjab Pakistan

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***Corresponding author:** Zahid Muhammad
Department of Physics, Hazara University Mansehra, KPK Pakistan

Abstract **Original Research Article**

In this study we investigate Phyllanthus Emblica (Commonly Amla) leaves and fruits through different spectroscopic techniques, like calibration free LIBS (CF-LIBS), X-ray Fluoresces (XRF), and Inductively Coupled Plasma Mass Spectroscopy (ICP MS). The LIBS setup has Nd: YAG laser and is calibrated at its fundamental harmonic 1064nm and its energy is 400mJ. The basic principle of this spectroscopy is when laser interact with sample (Amla pellets in in this case) it generates plasma plume for the period of nanoseconds. The parameter of plasma such as Plasma Temperature was calculated by Boltzmann Plot Method. For our sample electron temperature calculated by this method is 8841.56021eV. The next step is the value of Full Width Half Maximum (FWHM) for a neutral line of Iron (Fe) by Stark Broadening. Electron Number Density value for our samples is 2.493nm. After getting all these values we put it all of these in the formula and get values for weight and percentage concentration for both samples (Fruits and leaves of Amla). Quantitative Analysis of these samples we get 16 elements of leaves and 18 elements in fruits. Percentage Concentration in Amla leaves are Yttrium, Titanium, Tungsten, Bromine, Sodium, Cobalt, Thulium, Lanthanum, Vanadium, Manganese, Chromium, and Platinum. For the characteristics of the fruits are as Carbon, Thulium, Strontium, Potassium, Chromium, Manganese, Yttrium, Bismuth, Technetium, Cobalt, Iron, Scandium, Lanthanum, Titanium, Nickel, Molybdenum, Manganese. The results find through is by shining XRF device on Phyllanthus Emblica Leaves and fruits directly on its pellets and we get the results on the screen of the device. In XRF Results we obtained are given here for amla fruits are ZrO_2 36.493%, MoO_3 35.695%, Nb_2O_5 34.844%, Zr 27.032%, Nb 24.366%, Mg 24.265%, Mo 23.797%. XRF Results for leaves are Fe_2O_3 68.083%, Fe_3O_4 65.693%, FeO 61.408%, Fe 47.603%, ZrO_2 22.337%, Mg 16.334%, Zr 16.543%, Nb_2O_5 10.406%, Mn 6.723% etc. Elemental concentrations ranging from parts per billion to parts per trillion are determined using inductively coupled plasma–mass spectrometry, or ICP-MS. The atomic elements are subjected to a plasma source, ionized, and categorized according to their e/m (mass-to-charge) ratio. This method possesses exceptionally low detection thresholds, a broad linear range, and the capability of determining the isotopic composition of elements. Due to these advantages we choose to cross check our results of LIBS and XRF with that of ICP MS results. The results for ICP MS (in Conc. μg/g) leaves are Ca 6539, K 3983, Mg 1191, P 578, S 451, Fe 136, Na 110, Mn 56.48, B 47.79, Cu 13.12, Zn 10.50, Ba 6.25 and some minor traces of other elements. While the results for Amla Fruits are K 9284, Ca 811, P 504, S 158, Fe 21.14, Na 19.94, Mn 7.42, Zn 9.87, B 3.59, Ba 0.94, Cu 0.85. The common elements which are common in all these experiments are Fe, Mn, Cr, Na, K.

Keywords: Phyllanthus Emblica, Laser-Induced Breakdown Spectroscopy, XRF, ICP-MS, Quantitative Analysis, CF LIBS.

INTRODUCTION

Using laser-induced breakdown spectroscopy (LIBS), one can investigate the optical emission spectra of solid, liquid, and gaseous materials. Assessing the elemental composition of any material, including solids and gases, is the main use for this technique. The combination of LIBS with other spectroscopic techniques, such Raman spectroscopy, can easily broaden its applications in other fields [1]. Any substance could create optical emission from formed plasma when exposed to a powerful laser beam [3], as was recognized with the creation of the Ruby laser in 1960 [2]. Compared with other spectroscopic techniques [4, 5], LIBS is a very dependable, fast, and fascinating technology because it provides rapid multi-elemental analysis with outstanding accuracy [6]. It is a dependable, cost-effective, and efficient approach that requires minimal sample preparation [7, 8]. To concentrate the pulsed laser's beam on the target, a convex lens with the proper focal length is employed. Generally, a sample holding stage is movable to rotate the sample without making a deep crater on its surface. Because the plasma is heated, excitation and de-excitation processes occur there, producing emission lines that offer both qualitative and quantitative analytical information [9]. There are three phases to the LIBS experiment. The first entails a sample being heated and ablation-treated by a laser beam. A pulsed laser interacts with the material to create plasma in the second phase, and the plasma plume expands adiabatically in the third.

Over the last few decades, the LIBS instrument has become a vital and competitive analytical method in the commercially available technologies. It is now used for elemental analysis of a variety of materials, including metals, minerals, plants, food, and advanced substances like copper oxide, gallium arsenide, and indium antimonide. Many samples have previously been analyzed using the LIBS approach. Iron in iron slag specimens collected from surrounding plants was examined by Hussain *et al*., [10]. The cartage of gold alloys was examined by Ahmed *et al*., [11], using this technique in combination with the laser-ablated time-of-flight mass spectrometer methodology [12]. Fayyaz and colleagues used the CF-LIBS approach to analyze a number of cement brands from Pakistan. Borduchi et al. used the CF-LIBS technique with a one-point calibration procedure to perform elemental analysis on five different soybean leaf samples [13]. To study target materials based on tungsten, Roldan *et al*., [14], looked at the CF-LIBS. Fayyaz *et al*., [15], looked at the deposition of impurities of degraded compounds as a result of plasma-wall contact using the CF-LIBS technique. Zhang *et al*., [16], monitored the environment using the CF-LIBS method.

EXPERIMENTAL ARRANGEMENT

Sample Preparation

We started by gathering samples of Amla leaves and fruits from Hazara University Mansehra's Department of Biochemistry. After that, we concurrently cleaned these samples with tap water and mineral water. We then ground it and let it dry for half an hour in a clean pot. We then formed it into pellets under extreme pressure. The fruit sample's dimensions are 1.6 mm in diameter, 1.7 mm in thickness, and 1.230 g in mass. In contrast, the Leaves sample's dimensions are 1.2 mm in thickness, 1.4 mm in diameter, and 0.555 g in mass.

No preparation is needed for XRF experimentation as we just plug in the pellets prepared for LIBS just plug and play in XRF. Shining X Ray on the pallet gets our results.

The standard procedure in ICP-MS for solid materials is digestion in hot, powerful acids. The acids themselves vary from hydrofluoric acid (for samples with a high silicon dioxide component) to simple nitric acid (for comparatively simple matrices). During the digesting step, hydrogen peroxide may also be introduced to samples that include organic matter because H2O2 effectively breaks down organic matter.

Experimental Setup

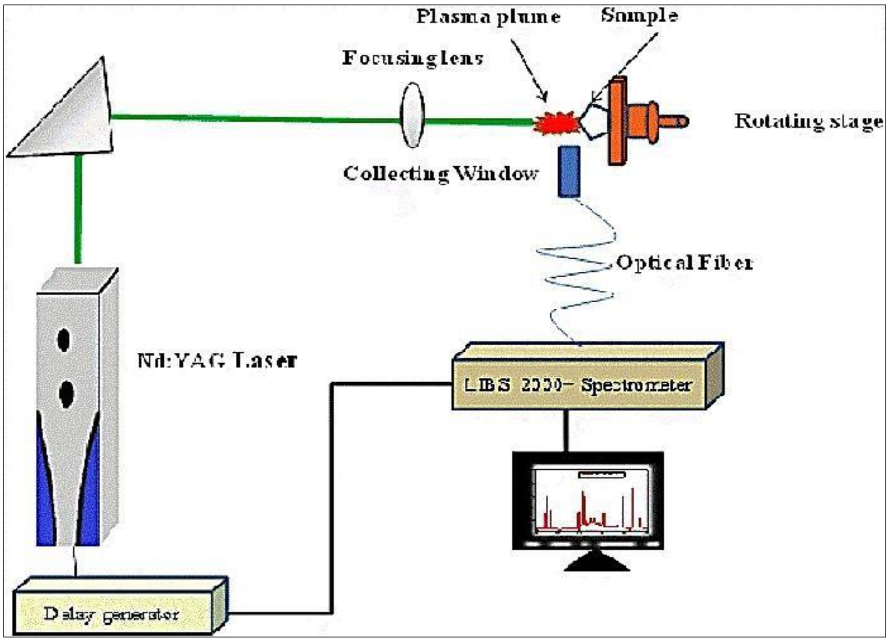
A pulse laser, sample, and spectrometer make up the LIBS system [17]. Our LIBS experimental setup, which was utilized for the materials' quantitative examination, is depicted in the figure. Our sample is ablated using a Q-switched Nd: YAG pulsed laser, which also produces plasma on the target's surface. Our laser source has a frequency of 10 Hz and a pulse length of 5 ns. Each pulse from our pulsed energy source may produce energy at 1064 nm (400 mJ), 532 nm (300 mJ), and 322 nm (100 mJ).

By varying the flash lamp Q-switch delay, the source energy controller allows us to modify the energy of our pulsed laser source. A Joule meter was used to detect the pulse's energy. The sample was exposed to the pulsed laser beam using a convex lens that was fixed to a sample holding platform. The lens has a 20 cm focal length. To prevent deep holes and give the sample a new surface for each laser fire, the sample holder was rotated following each laser blast.

Utilizing a collimating lens attached to an Ocean Optics LIBS2000 spectrometer, an optical fiber with a 600um core diameter positioned at a right angle.

Emitted plasma radiation is recorded using a fused silica window with a diameter of 5 cm. With the use of OOI-LIBS software, the undesired dark signals from the detecting unit were removed in order to extract the actual signal that was transmitted. Five spectrometers with a wavelength range of 200–700 nm and a slit width of 5µm make up the LIBS2000 detection system. A linear CCD, commonly referred to as a 2048-element linear CCD, with an optical resolution of 0.06 nm is a feature of every spectrometer.

All wavelengths between 200 and 720 nm are covered by the spectrums of neon, argon, and mercury, which are compared or recorded in order to calibrate our system. All five of the spectrometers that were placed in LIBS2000 were used to calibrate the "DH-2000-CAL" standard light source. The OOI LIBS software was used to store the data that spectrometers would be collecting simultaneously.



Experimental Set up Libs

RESULT AND DISCUSSION

We go over how to prepare and use laser-induced breakdown spectroscopy to quantitatively analyze Phyllanthus Emblica fruits and leaves. The Nd: YAG laser (1064 nm) is used in this technique to create plasma on the sample surface. Excitation and de-excitation of neutral or ionized atoms were used to determine the spectra for each sample. The species of the sample was confirmed by this plasma.

The CF-LIBS (calibration free) technique is used to calculate the quantitative analysis of Phyllanthus Emblica. To acquire elemental concentrations, this approach requires plasma properties such as electron temperature and number density. Plasma temperature can be determined using the Boltzmann plot approach, and electron number density can be determined using Stark broadening by utilizing the (FWHM) of any given spectrum line.

We make use of two Phyllanthus Emblica fruit and leaf samples. We designated them as sample leaves and sample fruit. We employ the calibration curve approach to determine the concentration. The following procedures are used to determine the elemental concentration.

We can break down LIBS data analysis into four steps:

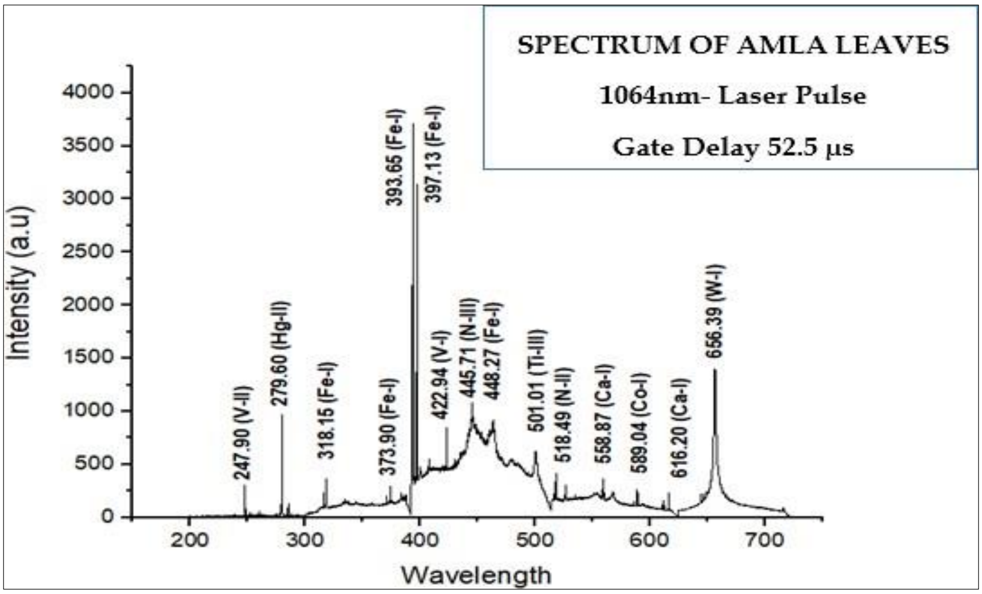
1. Temperature of Electrons
2. Density of Electrons
3. Maximum Full-Width Half
4. Analysis of Quantitative Data

Calibration-Free Method

All sample's atomic components can be found using CF-LIBS, which measures at least one of their distinctive spectral lines [20]. Using this method, elemental analysis and trace elements can be found in any sample [21]. F. Anabitrate *et al*., investigated how to determine each element's concentration and calibration curve without calibrating integral lines [19]. The CF-LIBS method uses line emission intensities and plasma temperature to determine the composition of the material.

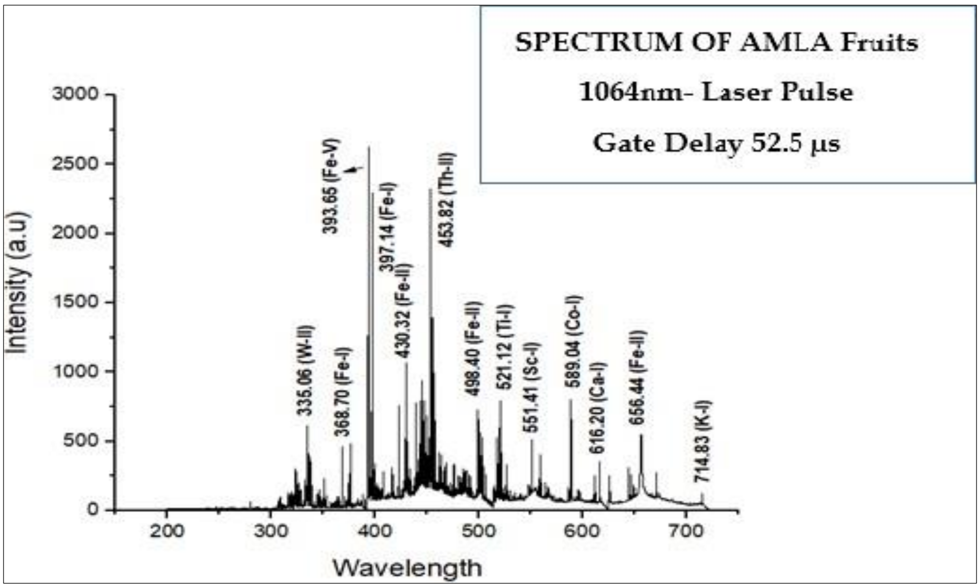
Analyzing the properties of laser-induced plasma, particularly temperature, LSD will help us better understand the laser-matter interaction. For optimal analytical performance, these parameters can be changed to optimize experimental conditions [22].

Examining the Sample



Two Phyllanthus Emblica fruit and leaf samples were pelletized and examined using a 1064 nm Nd: YAG laser. The sample was placed on a revolving stage and exposed to approximately 400 mJ of energy per pulse. The spectrum was produced by the excitation and de-excitation processes that occurred when the laser was shone on the sample plasma's surface. By modifying the delay gate, the LIBS 2000 spectrometer records this spectrum at a different delay time. Below is the spectrum that the sample fruit and sample leaves emanate.

Fundamental Examination of the Phyllanthus Emblica Leaf Spectrum



FRUIT SPECTRUM ELEMENTAL ANALYSIS OF PHYLLANTHUS EMBLICA

Identifying the Parameters of Plasma

The laser-induced plasma's number density and temperature are determined by its plasma characteristics. This allows us to determine plasma properties without knowing the plasma itself. In this study, the plasma parameters of fruits and leaves of Phyllanthus Emblica will be discussed [22].

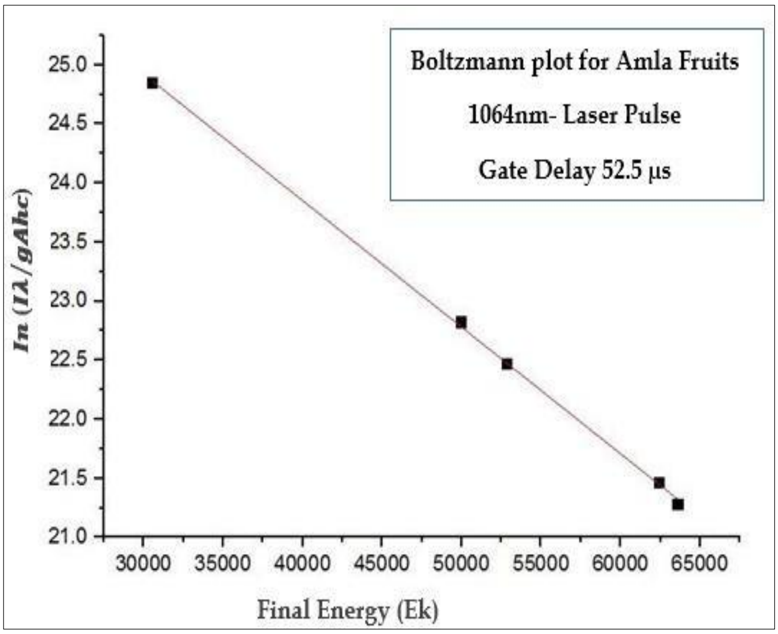
Calculating the Electronic Temperature

Electron temperature measurement allows us to determine the degree of ionization and excitation of plasma. The electron temperature can be numerically determined using the Boltzmann plot method. For the same thermometric species, the process determines the temperature and related intensities using the LTE condition [18].

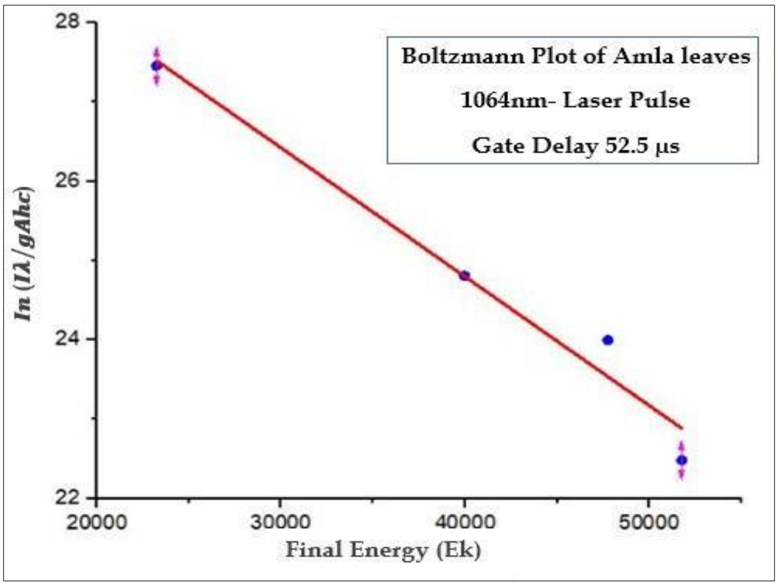
] The lines used for computations must have equal states of ionization, and all spectrometric parameters, including statistical weight g, transition probability, and ultimate energy, should be recorded [23].

The temperature in our estimation is 8841.56021K. The following formula can be used to determine the electron temperature:

$In\ (I\lambda\ /gAhc) = -\ (\Delta E/KT) + InC$



The Leaves Boltzmann Plot



Fruit's Boltzmann Plot

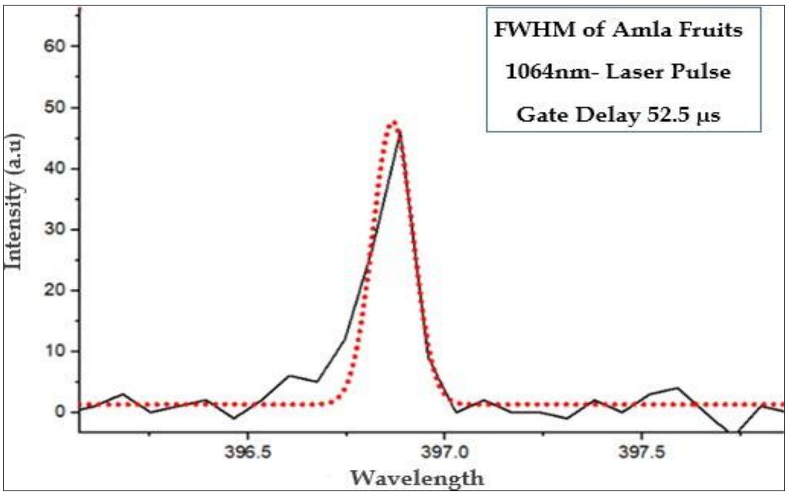
Calculating the Density of Electron Numbers

The electron Number density can be calculated by using neutral atom of an ion. Next is FWHM of spectral lines can be used to determine it [24, 25]. For accurate results in our scenario, Ion Broadening's 15% contribution can be disregarded.

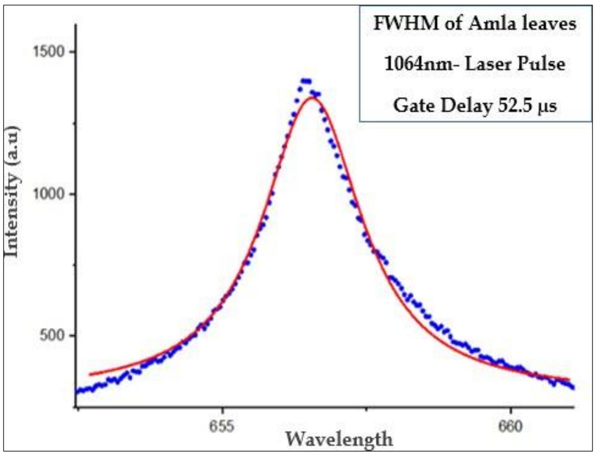
$$\Delta\lambda_{1/2} = 2\omega\,(N_e/10)$$

$$N_e = \frac{\lambda_{1/2}}{2\omega} \times 10^{16}$$

Here
The Number is Ne. The Electron Impact Parameter, or density 𝝀1/ FWHM, has a value of 1.3×1017 cm-3, according to literature [26].



The AMLA Fruit's FWHM



Fwhm for Amla Leaves

QUANTITATIVE ANALYSIS USING CALIBRATION FREE LIBS

In our work, we use the Boltzmann equation to get the atomic number density of the neutral atom in order to do a quantitative analysis of our sample using CF-LIBS:

$$FC^{Z} = I_K \frac{U_Z}{A_k g_k} e^{(E_k/KT)}$$

The Saha Boltzmann Equation is then used to determine the atomic number density of ionized atoms, which is

$$\frac{C^{Z}_{+1} N_e}{C^{Z}} = \frac{(2mKT)^{3/2}}{h^3} \frac{2U_{Z+1}}{U_Z} \exp\left[-\frac{E_{ion}}{KT}\right] cm^{-3}$$

Where $\frac{(2mKT)^{3/2}}{h^3} = 6.4\text{^}10^{21}$

Any sample's elemental makeup can be determined by adding the ionized and neutral atoms' atomic number densities.

$$C^{a}_{T} = C^{a}_{Z} + C^{a}_{Z+1}$$

One can determine the element's percentage composition by multiplying the total element concentration by 100.

$$C_{\%} = \frac{C^{a}_{T}}{C_T} \times 100$$

One can use the following formula to get the element's weight percentage composition:
$CWt\% = (CT \times A / \sum CT \times A) \times 100$

ELEMENTAL COMPOSITION

Any sample's elemental composition can be determined using CF-LIBS. We contrast our spectrum with the National Institute of Standards and Technology's (NIST) Standard Reference Database (SRD). Around the world, this is the accepted norm for data analysis. The relationship that may be used to assess the spectral line's intensity is

Intensity is proportional [‹ψf ׀er ׀ψi›]

Fruit of Amla Percantage concentration

Elements;	% Conc
(Co I)	0.0009911
(Fe I)	0.0020644
(Sc I)	9.26E-066
(La I)	2.58E-066
(Ti I)	0.0004744
(Ni I)	0.0003799
(Mo I)	0.0006666
(C I)	0.9177177
(Tm I)	0.0004333
(Sr I)	6.86E-066
(K I)	1.5143344
(Cr I)	0.0002666
(Mn I)	0.0002388
(Y I)	0.0003266
(Bi I)	0.0008022
(Tc I)	5.73E-077

Amla leaves' Percentage Concentration

Elements;	% Conc
(Pt I)	0.0053422
(Fe I)	0.0398588
(V I)	0.012544
(Mn I)	0.0069655

(Cr I)	0.0106311
(Y I)	0.005033
(Ti I)	0.0339199
(W I)	0.0044666
(Br I)	2.2851622
(Na I)	0.0147444
(Co I)	0.3350777
(Tm I)	0.0336811
(KI)	24.912633
(La I)	0.0009655

XRF RESULTS

Amla Fruits		
Elements	%	±
ZnO	0.0	21.535
ZrO2	36.493	11.547
Nb2O5	34.844	7.036
MoO3	35.695	10.958
Zn	0.0	17.367
Zr	27.032	8.553
Nb	24.366	4.92
Mo	23.797	7.305
Mg	24.265	1.194
Si	0.222	0.153
P	0.245	0.04
S	0.063	0.023

Amla Leaves		
Elements	%	±
Mn3O4	9.345	5.799
MnO	8.673	5.382
Fe2O3	68.073	8.586
Fe3O4	65.693	8.286
FeO	61.408	7.745
ZrO2	22.337	5.137
Nb2O5	10.406	6.353
MoO3	4.729	5.13
Mn	6.723	4.172
Fe	47.603	6.004
Zr	16.546	3.805
Nb	7.277	4.443
Mo	3.153	3.42
Mg	16.334	1.717
Si	1.926	0.22
P	0.21	0.048
S	0.218	0.026

ICP-MS RESULTS

	Elements	Amla Leaves (Conc. µg/g)	Amla Fruits (Conc. µg/g)
1	Al	77.42	12.12
2	B	47.79	3.59
3	Ba	6.25	0.94
4	Ca	6539	811
5	Cr	2.20	**ND**
6	Cu	13.12	0.85

7	Fe	136	21.14
8	K	3983	9284
9	Mg	1191	458
10	Mn	56.48	7.42
11	Na	110	19.94
12	Ni	0.52	**ND**
13	P	578	504
14	S	451	158
15	Sr	27.13	3.93
16	Zn	10.50	9.87

ND: Not Detected

CONCLUSION

Laser-Induced Disintegration Phyllanthus Emblica fruits and leaves collected by the Department of Biochemistry at Hazara University Mansehra have undergone spectroscopy. Using a LIBS system with a delay gate of 52.5 µs and a Nd: YAG laser at 1064 nm, the experiment was conducted outdoors.

A plume of plasma was released when the laser focused on the material, and this was subsequently examined using the Calibration-Free Laser-Induced Breakdown Spectroscopy method.

Four sections can be distinguished in the analysis. In order to compute the plasma temperature, we first ascertain the slope of the Fe-I lines.

Which let us to the second step of calculating Electron Temperature Te for the plasma which is in our case 8841.5601 eV. Next, we determine the number density Ne. Finally, we apply the Lorentzian fit to the H alpha line to determine the Full-Width Half Maximum (FWHM), which comes out to be 2.4393 nm. Following these procedures, we obtained 16 elements in the Amla plant's leaves and 18 elements in its fruit. All the results for amla fruits and leaves are discussed in the table above.

In XRF Results we obtained are given here for amla fruits are ZrO2 36.493%, MoO3 35.695%, Nb2O5 34.844%, Zr 27.032%, Nb 24.366%, Mg 24.265%, Mo 23.797%. XRF Results for leaves are Fe2O3 68.083%, Fe3O4 65.693%, FeO 61.408%, Fe 47.603%, ZrO2 22.337%, Mg 16.334%, Zr 16.543%, Nb2O5 10.406%, Mn 6.723% etc.

The results for ICP MS (in Conc. µg/g) leaves are Ca 6539, K 3983, Mg 1191, P 578, S 451, Fe 136, Na 110, Mn 56.48, B 47.79, Cu 13.12, Zn 10.50, Ba 6.25 and some minor traces of other elements.

While the results for Amla Fruits are K 9284, Ca 811, P 504, S 158, Fe 21.14, Na 19.94, Mn 7.42, Zn 9.87, B 3.59, Ba 0.94, Cu 0.85.

All these results are discussed in detail in the above tables.

The common elements which are common in all of these experiments are Fe, Mn, Cr, Na, K.

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