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Laser-Induced Breakdown Spectroscopy in Vegetable Analysis: Contaminants and Nutrients

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ABSTRACT

Vegetables are rich in minerals, but pollutants and wastewater, which introduce heavy metals into the soil, heavily impact their cultivation. As an efficient and effective methodology, scientists prefer using Laser-Induced Breakdown Spectroscopy (LIBS) as a light-based technique to determine the elemental constituents of vegetables. It aids in safety and quality assurance by allowing them to image nutrients and hazardous metals, such as cadmium. This study explored the application of LIBS for detecting contaminants, such as Cd, and profiling essential nutrients in vegetables. Unlike conventional methods such as ICP-MS and AAS, LIBS offers fast, on-site, and multi-element analyses with minimal sample preparation. This review consolidates recent studies on carrots, potatoes, spinach, broccoli, and other leafy greens, emphasizing enhancements using nanoparticles and chemometric tools to improve sensitivity and accuracy. According to the results, LIBS has also been effectively employed to analyze the components of vegetables, enhancing the control and safety of food quality surveillance. The results also prove that LIBS can be a better method for monitoring food quality and safety. LIBS is more consumer-and environmentally friendly because it is portable, fast, and capable of simultaneously analyzing various components.

Keywords: *Cadmium, Food, LIBS, Metals, Safety, Vegetables*

1. Introduction

Vegetables are sources of Potassium, Calcium, Magnesium, and Iron, which are important for health. Yet, when toxic metals like lead and cadmium contaminate them, their safety and nutritional value is compromised [1]. Concentrated metals invade the agricultural ecosystem mainly through contaminated soil and water [2]. The application of raw sewage for irrigation in some Asian developing countries results in certain crops containing alarmingly high levels of cadmium, lead, chromium, mercury, arsenic, and nickel, well over international safe limits [1].

Common methodologies for the quantification and detection of heavy metals in vegetables include Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Atomic Absorption Spectrometry (AAS). Although these techniques are efficacious, they are frequently costly, time-intensive, and require highly skilled personnel because of the intricate nature of the laboratory apparatus. Additionally, they require extensive sample preparation, which may affect the accuracy of the results. Consequently, these methods are not conducive to decision-making in field settings or for immediate assessment [3]. The substantial time investment required renders these techniques ineffective for rapid decision-making and field applications [4]. Zhao et al. (2019) demonstrated that the incorporation of nanoparticles in Laser-Induced Breakdown Spectroscopy (LIBS) enhanced the sensitivity of detecting pesticides and heavy metals by a factor of two compared to conventional LIBS [5].

1.1 Fundamentals of LIBS and Laser-Matter Interaction

The quality and intensity of plasma used in LIBS is dependent on the intensity of pulse, the duration, pulse energy and roughened surface. LIBS systems normally involve nanosecond Lasers, picosecond/ femtosecond lasers, and broadband spectrometers simultaneous multi-element analysis [6]. The types of lasers often used in LIBS systems include nanosecond lasers, but picosecond and femtosecond lasers are becoming more common to produce higher resolution and reduced thermal effects [7].

Recent comparative studies have revealed that shorter pulse durations decrease continuum emission by restricting avalanche ionization [8]. The increase in plasma lifetime and signal strength offers an effective tool for utilizing double-pulse LIBS (DP-LIBS) with signal enhancement 2-32 times greater than that of single-pulse LIBS spectra [9]. Quantification is typically performed with broadband spectrometers, such as Echelle or Czerny-Turner systems, with a wide wavelength range (200-900 nm) and will yield a multi-element analysis [10].

Recent advances in dual-spectrometer LIBS (DS-LIBS) systems have led to the implementation of both wideband and narrowband spectrometers capable of collecting major and trace element spectra in a synchronous manner, with characteristics enabling the classification of more than 90% of the datasets (specifically 92% vs. 84% and 73% when single-spectrometer LIBS) [11]. These principles constitute the core of the application of LIBS techniques in the analysis of vegetables, as the technique is quick, sensitive, and non-destructive in the detection of elements. LIBS has achieved significant success in the detection and mapping of trace elements in a variety of plant matrices, and uses is increasingly widespread across the plant science, agriculture, and food technology fields [12]. Recent research has proven that LIBS can be used to examine the nutritional elements in medicinal herbs with great precision, identifying over 90 emission lines of elements and effectively quantitatively detecting nutritive elements in different production regions

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[13].

2. Laser-Induced Breakdown Spectroscopy in Food Analysis

LIBS has become a useful research tool in the field of food science, and it can be used to determine the elemental composition and check the quality parameters [14]. In the case of LIBS, materials are bombarded with laser light, which forms plasma, including atoms, ions, and electrons that excite, collide, and emit light with specific wavelengths that can be measured. It is applied in the food industry to determine and identify products owing to its accuracy and affability concerning cost-effectiveness [15].

Real-time analysis of trace elements in food can be conducted using LIBS, which allows for quality assurance and quick decisions [16]. The measurement accuracy of LIBS can be compromised by changes in the plasma and sample matrix. Therefore, researchers are attempting to augment the system with other spectroscopic techniques to enhance its precision [17].

The integration of LIBS with machine learning and chemometrics enables the creation of automatic systems for food classification, origin determination, and adulteration detection [18]. These advances further enhance the usefulness of LIBS for detailed monitoring of the supply chain.

2.1 Detecting Contaminants in Vegetables

Currently, the focus of LIBS has shifted to farming and the food safety analysis. Researchers have conducted studies on plant sample analysis using LIBS [19]. Using LIBS, researchers were able to detect Cd-containing cocoa powder, which confirmed that spot testing is efficient in field work; the results improved significantly under proper sample handling and background removal [20]. Thus, it has been proven that LIBS can be used for monitoring contamination in plant materials.

2.2 Nutrition Profiling

Iron (Fe), calcium (Ca), potassium (K), and other essential trace elements can be measured using LIBS in vegetables, and contamination can also be examined. Researchers using LIBS have demonstrated that organically grown and mid-grown conventional vegetables have different nutritional values [21]. With this method, detecting several nutrients is very easy, which enables producers and consumers to choose the right food. Indeed, LIBS can detect almost all possible problems related to food safety and quality issues [22]. The use of LIBS nutrient data aids in differentiating the nutritional values of organically and conventionally grown vegetables [21].

Table 1 presents the identification of food constituents, such as heavy metals and other chemical elements, using the LIBS technique along with other integrated methods.

Table 1: LIBS Applications in Elemental Analysis of Vegetables

Ref.	Vegetable	Factor	Method	Results
[23]	Potato	Trace mineral elements	The investigation uses LIBS to define and examine the fine vegetable mineral elements, in our case, we study the plasma from the Nd:YAG laser ablation of potato skin and flesh.	For both potato flesh and skin, the following mineral constituents were identified along with their estimated relative concentrations: magnesium, calcium, aluminium, potassium, sodium, copper, iron, manganese, titanium, lithium, silicon, and some others.
[14]	Spinach and rice samples	Nutrient elements and contamination by pesticides	Using a Q-switched Nd:YAG laser to LIBS, spinaches and unpolished rice samples' nutrient elements (Mg, Ca, Na, K) were measured spectroscopically with light emitted from the sample while it was excited by the laser.	Nutrient elements (Mg, Ca, Na, and K) in spinach and unpolished rice samples, with limits of detection (LODs) for these elements in spinach being 29.63 mg/kg for Mg, 102.65 mg/kg for Ca, 36.36 mg/kg for Na, and 44.46 mg/kg for K, while in unpolished rice, the LODs were 7.54 mg/kg for Mg, 1.76 mg/kg for Ca, 4.19 mg/kg for Na, and 6.70 mg/kg for K.
[24]	Carrot	Organic and inorganic elements	Recorded emission spectrum of the carrot root pellet to identify the presence of various inorganic elements. The produced plasma during the LIBS process helped in understanding the characteristics of the emitted spectrum.	Inorganic elements such as K, Na, Fe, Ca, Ti, Zn, Hg, Cu, Mg, Sr, Co in the carrot root pellet were identified. The emission spectrum of fresh raw carrot also indicated the presence of additional organic elements, specifically C, H, N and O, alongside the inorganic elements detected in the carrot root pellet.
[21]	Cauliflowers and broccolis	Nutrient elements	LIBS to identify and compare the presence of major nutrient elements in organic and conventional vegetables, specifically focusing on different parts of cauliflowers and broccolis as working samples.	Analysed and compare the presence of major nutrient elements in organic and conventional vegetables, specifically focusing on different parts of cauliflowers and broccolis. The analysis involved acquiring laser-induced breakdown spectra at optimized parameters and performing both univariate and multivariate analyses.
[25]	Leafy vegetables	Heavy metals	LIBS was applied to analyse the presence of cadmium (Cd) in fresh leafy vegetables, demonstrating its capability for green analysis of toxic heavy metals in agricultural products.	Effectively monitor cadmium (Cd) levels in fresh leafy vegetables, although initial direct calibration methods using single Cd lines showed limitations in accuracy. PLSR analysed the data, the researchers improved

			Partial least-squares regression (PLSR) was utilized for predicting Cd concentrations	the prediction accuracy of Cd concentrations, meeting the necessary requirements for food safety determination.
[26]	Freshly cut carrot samples	Nutrient elements	LIBS technique was employed utilizing a Nd:YAG laser with a wavelength of 1064 nm for the ablation process.	Detected 18 chemical elements in fresh carrot samples, including essential nutrients such as Mg, Al, Fe, Mn, Ti, Ca, and Mn

2.3 Comparative Evaluation of Chemometric Models in LIBS

Several studies have highlighted the reality of chemometric techniques in improving LIBS analytical capability, especially under complex food matrices such as vegetables. Principal component analysis (PCA) has been extensively exploited to carry out an initial visualization of the data and to determine natural clusters in multivariate LIBS data [27, 28]. PLSR has demonstrated incredible predictive ability to measure the scales of elemental levels, such as cadmium and calcium, in leafy greens [25]. Linear discriminant analysis (LDA) has been found to be very useful in the identification of organically and conventionally grown vegetables using weak spectral gradation [29]. Artificial neural networks (ANN) and other machine learning algorithms have recently become popular because of their better performance in modeling nonlinear relationships and high classification accuracy when applied to large LIBS datasets [27, 30].

Figure 1 shows a flow chart that summarizes a comparative overview of widely used models PCA, PLSR, LDA, and ANN and their key characteristics, applications, data requirements, strengths, and limitations.

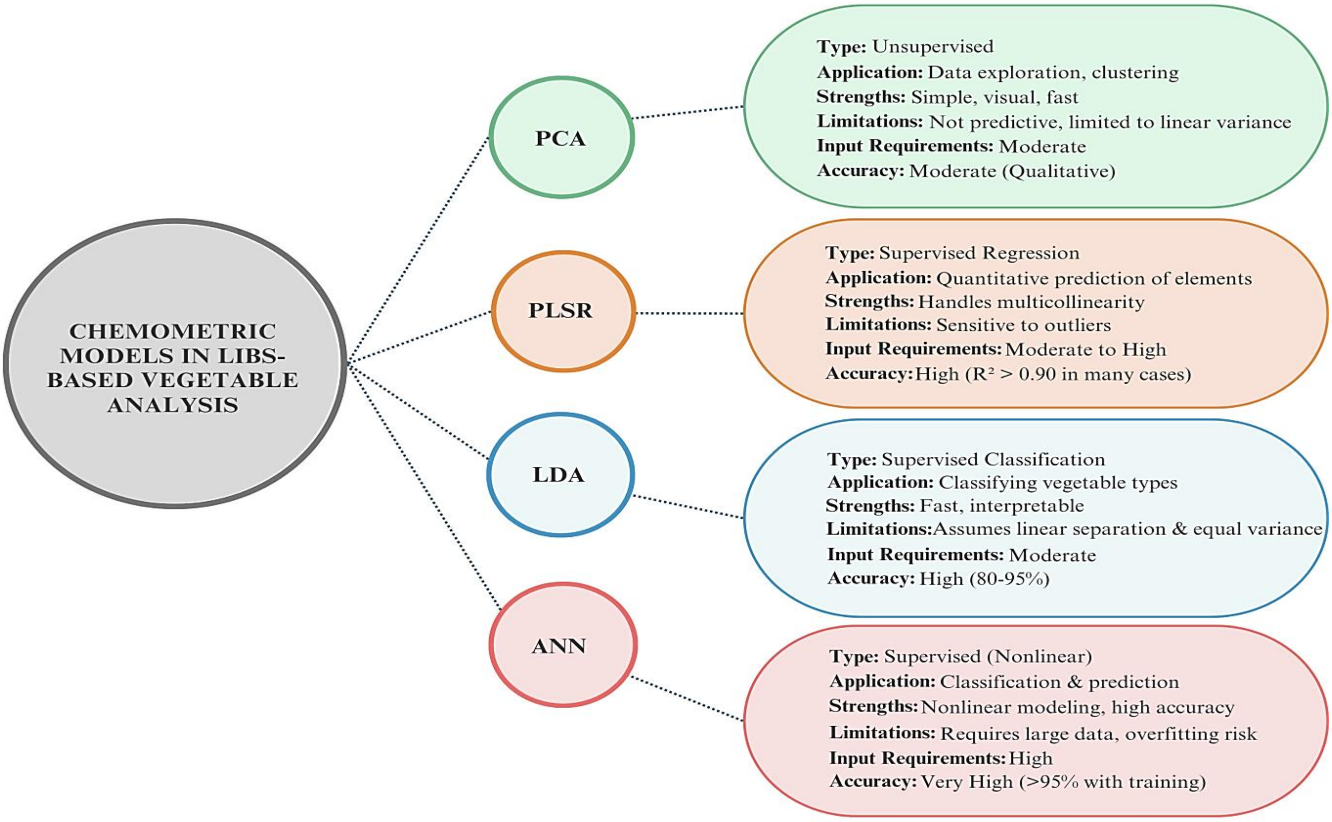

Fig. 1: Flow Chart of Chemometric Models Used in LIBS-Based Vegetable Analysis.

3. Feasibility and Challenges

Other recent findings have pointed out that portable LIBS systems, although possessing rapidity of analysis and low sample preparation, have serious infrastructural constraints when used in the developing world [31]. The commercialization of ruggedized underwater LIBS, such as the LIBSea II system, has made it possible to realize the technical viability of portable field-deployable tools [32]. Nevertheless, these systems still require advanced laser devices and spectroscopy equipment, which may pose a financial obstacle in limited-resource environments [33].

Barriers to implementation also differ across regions and contexts. Investigations into the adoption of technology in Africa and Asia have found some uniform issues, such as a shortage of funds, poor infrastructure, and a lack of technical support systems. To achieve equitable access to, and wide application of, the technology it is necessary to respond to the region-specific challenges by the use of cost-reduction strategies, local training programs, and simplification of LIBS interfaces.

3.1 Matrix Effects and Calibration Challenges in LIBS along with Solutions

By cyclic culture, vegetables will have considerable sample heterogeneity because moisture content, surface texture, density, and inner composition will vary in the samples [34, 35]. These chemical and physical differences may influence the homogeneity of the plasma generated in the LIBS analysis, where there is variation in the emitted spectral signals [36].

Vegetable matrices are heterogeneous materials, making LIBS analysis problematic. Very strong physical and chemical baseline variations characterize agricultural products, such as vegetables, which influence the repeatability of spectral measurements [35, 36]. This heterogeneity occurs in several aspects: moisture content variations alter the laser-matter interaction and plasma formation, surface texture variations alter the ablation patterns, and internal variations cause localized matrix effects [37].

Vegetable analysis is one area where moisture effects are particularly serious. It was found that the optimal laser absorption properties and plasma temperature distribution substantially depend on the moisture content [38]. Atmospheric conditions, including pressure, composition, and confinement effects, significantly influence LIBS performance by altering plasma formation, the signal intensity, and the spectral resolution [39].

Spatial variations in the surface texture also complicate the LIBS analysis of vegetables. Studies show that the surface roughness influences the ablation performance and craters formation patterns. This nonuniform distribution of the laser energy may occur due to irregular surface topography and will cause nonuniform generation of plasma and produce changes in the spectral signals [40]. This is especially applicable to vegetables that grow with naturally bumpy or textured surfaces, such as root vegetables or coated vegetables with a waxy policing.

To overcome these matrix-induced challenges, several strategies have been developed to improve the performance and consistency of LIBS in vegetable analysis. Calibration-free LIBS (CF-LIBS) is a self-contained technique that computes elemental concentrations implicitly based on plasma parameters with few external dependencies on calibration standards [41]. Internal standardization methods involve standardizing the spectral signal with respect to elements within the matrix sample to demodulate the variation caused by matrix effects [42]. Optimized experimental procedures, such as spatial confinement, have shown significant increases in the detection limit and precision of the analytical method [43]. Plasma intensity can be increased and its duration extended using a dual-pulse LIBS system, leading to improved stability and increased amplification of the emission signal. Such systems work as follows: the first pulse ablates material and establishes initial plasma conditions, whereas the second pulse reheats and strengthens the plasma, resulting in a significant signal enhancement [44]. The method of multi-spot averaging, which implies the gathering of spectra of different points on one sample, has also proved useful in mitigating the effects of localized heterogeneity and better reproducibility [45]. The aspects of the matrix effects in LIBS are evidenced by the sample heterogeneity and the fluctuations of the signals, as well as the ways out discussed in Figure 2.

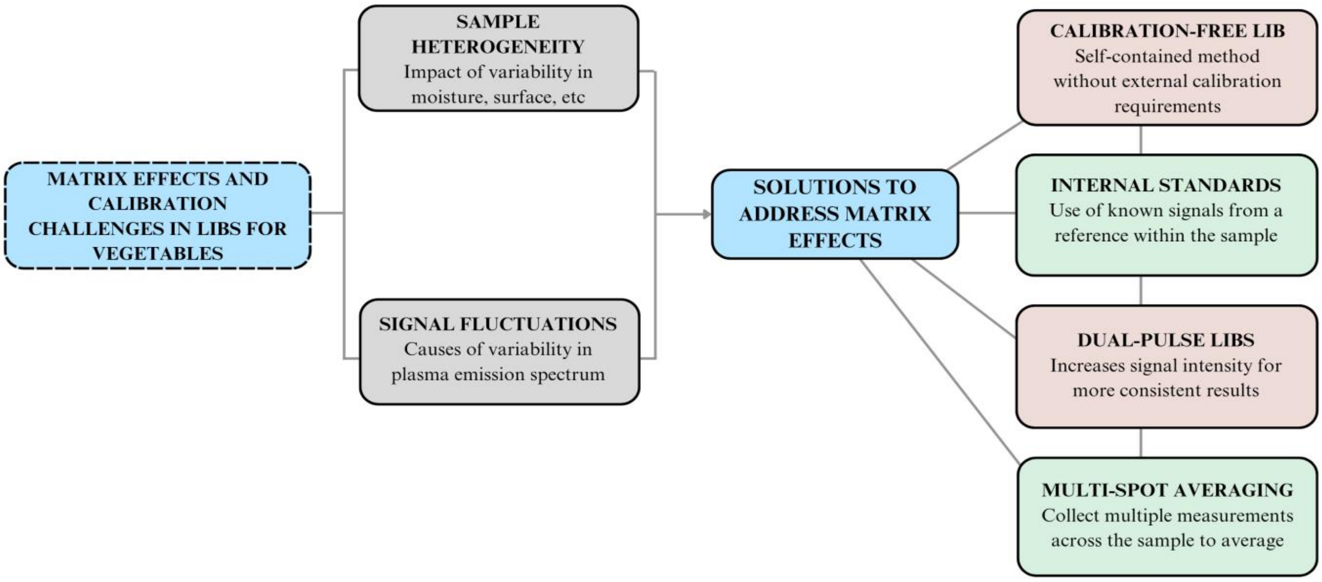

Fig. 2 Challenges in LIBS for Vegetables Analysis and Its Solutions

4. Recent Advancements in LIBS Technology (2020-2025) for Food Safety Applications

Recent innovations in LIBS technology from 2020 to 2025 have largely increased the range of its application in food safety, especially in the non-destructive manner of food safety analysis of vegetables. One of the most revolutionary advances has been the advent of handheld and portable LIBS instrumentation, which allows the on-site detection of elements in real time without laboratory infrastructure [46]. Simultaneously, other forms of deep learning have been developed to work with LIBS, including convolutional neural networks (CNNs) and autoencoders, to enhance the interpretation of the spectrum. Such models are very effective in identifying complex nonlinear trends in high-dimensional spectral data, resulting in improved classification accuracies and increased detection of subtle differences in compositions [47]. Nanoparticle-enhanced LIBS (NELIBS) is another important innovation in which metal nanoparticles are deposited on sample surfaces, increasing the signal arising from the plasma. NELIBS has especially been able to reduce the limits on the detectability of trace heavy elements such as cadmium and lead [48]. Moreover, 3D mapping and imaging methodologies have been investigated in LIBS to obtain the spatial regions of the elemental distribution in raw vegetable tissues. The commonly defined spatially resolved analysis allows better insights into the localization of nutrients and infiltration of contaminants, which can be used to evaluate food quality and study agriculture [49]. The combination of these technological advances is a paradigm shift in LIBS applications for food safety, given the ability to make portable deployments, perform enhanced analytics through the incorporation of artificial intelligence, enhance sensitivity through nanoparticle reproducers, and provide full spatial analytics capabilities that were unavailable in earlier LIBS systems.

4.1 Future Directions

To maximize the potential of LIBS in food analysis, future efforts are required to miniaturize the equipment and build portable, field-ready systems with real-time observation procedures. New developments indicate that there have been significant advances in this field, with commercial handheld LIBS instruments exhibiting very strong performance in food authentication [31]. The analysis of portable LIBS systems has also proven their usefulness in the analysis of various food matrices, such as European Alpine-style cheeses, coffee, spices, balsamic vinegar, and vanilla extracts, where little or no treatment is given to the sample [50].

4.2 Limitations and Advancements in LIBS Application

Despite the usefulness of LIBS, it is disadvantaged by the fact that LIBS is prone to matrix effects, low sensitivity levels on the trace elements and the inexistence of standardized calibration techniques, which contributes to quantitative reproducibility and accuracy. This variation in sample surface composition makes the generation of plasma even more confounded with the heterogeneous surface being more fluctuating in the plasma. To survive these challenges, researchers were exposed to NELIBS to enhance the signal, and also to include chemometric techniques in order to fully analyze the data, namely, PLSR and PCA. Machine learning algorithms that facilitate the accuracy of classification and real-time food safety monitoring in a field are also part of the recent development.

5. Conclusion

LIBS is a useful method for analyzing the nutritional and contaminant content of vegetables. The reviewed studies confirm that it can accurately determine the levels of Cd and nutrients such as potassium, calcium, and iron in various types of vegetables. Unlike traditional methods, LIBS is quicker, does not require much sample preparation, and allows for testing at the site, which is great for immediate food safety checks. As new progress is made in enhancing signals, processing data, and calibration, LIBS is set to become vital for sustainable farming, food safety, and health services.

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