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# ADVANCEMENTS IN SOIL NUTRIENT AND CONTAMINANT DETECTION USING LASER-INDUCED BREAKDOWN SPECTROSCOPY

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**Abstract**
*Laser-Induced Breakdown Spectroscopy (LIBS) has emerged as a fast, versatile, and efficient analytical technique with increasing applications in soil analysis, contributing to sustainable agricultural practices. This review provides a comprehensive overview of recent advancements in LIBS for soil characterization, emphasizing its role in detecting and quantifying essential nutrients and contaminants. Key developments focus on addressing limitations such as matrix effects, moisture content, and particle size variability, with optimized techniques including spatial confinement, addition of conductive materials, and laser-induced fluorescence (LIF) enhancing detection limits and analytical precision. Integration of machine learning, deep learning, and chemometric approaches has further improved predictive modeling, enabling robust analysis across diverse soil matrices. Additionally, portable and handheld LIBS systems facilitate real-time, in-field soil monitoring. Standardization efforts using certified reference materials and interlaboratory protocols are enhancing reproducibility and broader scientific acceptance. Collectively, these innovations establish LIBS as a powerful tool for precise soil nutrient management, contaminant detection, and environmentally sustainable agriculture.*

## INTRODUCTION

Sustainable agriculture is an integrative current practice, which seeks to fulfill the needs of expanding population with regard to food and resources without compromising the environmental integrity, economic sustainability, and societal equity. It has loomed large in the late 20$^{th}$ century, as affirmed after the United Nations Conference on the Human Environment held in 1972 [1]. LIBS has also found its use in the detection of dangerous components like the dangerous element lead in the soils, with new innovations increasing sensitivity of the signal by enhancing the use of conductive materials and chemometric analyses to support both food safety and environment quality [2]. Moreover, LIBS-based methods were devised to identify the presence of hydrocarbon contamination and atmospheric microplastic, indicating the flexibility of the tools in solving a range of environmental issues associated with sustainable agriculture and overall ecosystem health [3]. Precision agriculture relies on the portability, but also the timely acquisition of data that is a characteristic feature of LIBS instruments, which can be used to map the dynamics of soil parameters in any field, as a result, saving resources and increasing their output and productivity over the long term [4].

### LIBS for Soil Nutrient Detection

LIBS has become a useful research tool in the field of food science, using which it is possible to determine the elemental composition. The recent findings have also proved that LIBS has the ability to detect strongly emitting elements like calcium, magnesium, potassium and iron. K I lines and K I lines are

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commonly used spectral lines and they can be used to achieve good quantitative analysis with wavelengths of 404.72 nm and 766.49 nm respectively in element analysis [5]. In a similar manner, calcium and magnesium detection is useful; since these elements are characterized by emission lines with high signal- to- noise ratio thus potential measurements of concentration in a wide variety of soil matrices [6]. Most recently, techniques have been developed to combat these issues, such as the pelletization of samples and simulated soil matrices in order to normalize signals [5].

Advances in the use of complicated chemometric techniques with LIBS has considerably improved analytical abilities of using LIBS as a means of quantifying the nutrients in soil. Partial least squares regression (PLSR) has recently proved to be very efficient to deal with intricate soil matrices and spectral interferences. Research has concluded that PLSR models can use the R 2 to reach prediction results greater than 0.90 in major nutrients [4].

The latest addition of machine learning has also boosted the performance of LIBS. Convolutional neural networks were used efficiently to predict soil nutrients as well as artificial neural networks [7]. Such procedures have the capacity to extract the complex spectral features, and process the non-linear association between the spectral measurements and nutrient levels automatically [8].

Spatial confinement techniques have shown promise for improving LIBS performance in soil analysis. Cylindrical cavity confinement combined with inert gas atmospheres ($N_2$) has achieved enhancement factors of up to 3.25 times for certain elements [9]. The technique enhances stability of plasma and eliminates the effect of matrix that typically interferes with soil determination. More sensitive measurements have been achieved and explored, especially to target the difficult elements, such as phosphorus and nitrogen, using double-pulse LIBS systems [6]. The systems are also able to enhance ablation efficiency and characteristics of plasma via sequential laser pulses and thereby there are high intensities of emissions and vastly wide detection limits. **Table 1** summarizes key studies that have explored the application of LIBS in soil analysis, highlighting the benefits, limitations, and specific focus areas of each investigation in relation to nutrient detection, contamination assessment, and agricultural applications.



**Table 1: Key Studies on LIBS Applications for Soil Nutrient Detection**

| Ref. | Description | Benefits | Limitations |
|---|---|---|---|
| [10] | Reviews LIBS applications for soil carbon, nutrients, and classification. Highlights advantages, limitations, and potential for real-time soil analysis | Real-time soil analysis at low cost. Requires little or no sample preparation. | Its detection limit for elements like Mo and Ca is 0.3 mg $kg^{-1}$ for Mo and 89 mg $kg^{-1}$ for Ca, potentially insufficient for all soil types. |
| [11] | Review of LIBS applications in agriculture (2010-2019). Focus on soils and fertilizers analysis techniques. | Enhanced precision and accuracy in soil analysis. Ability to analyze multiple elements simultaneously. | The high limit of detection for certain elements, like potassium, necessitates internal standardization or ionizable elements for improved LIBS. |
| [12] | LIBS analyzes elemental composition using laser-induced microplasma. Suitable for soil, minerals, and environmental analysis. | Fast multi-elemental response with less sample preparation. Suitable for remote in-situ analysis in hostile environments. | Challenges in optical resolution and calibration, which may impact accurate results based on the quality of spectral data collected. |

| | | | |
|---|---|---|---|
| [13] | Systematic study of soil elemental and chemical composition.<br>LIBS technology applied for soil contamination detection. | Rapid detection of soil contamination and nutrients.<br>Green analytical technique for sustainable agriculture. | - |
| [14] | LIBS measures nutrients in greenhouse soil samples.<br>Rapid, efficient, and precise nutrient analysis method. | Rapid and efficient nutrient measurement in soil.<br>Excellent detection limits and precision for nutrient analysis. | The study found that the precision of LIBS measurements for greenhouse soil samples is influenced by sample complexity, homogeneity, and laser shot reproducibility. |
| [15] | In situ analysis of polluted soils using LIBS.<br>Provides semi-quantitative pollution information efficiently. | Time-saving and low-cost soil contamination analysis.<br>Optimizes sampling operations, reducing unnecessary laboratory analyses. | The paper highlights the limitations of in situ LIBS as a quantitative analytical method compared to traditional methods like ICP-AES, with detection limits for lead and copper being limited, and not for other heavy metals or elements. |
| [16] | LIBS enables rapid, non-destructive soil element analysis.<br>Accurate multi-element detection in watershed environments. | Non-destructive testing and real-time analysis capabilities.<br>Simultaneous multi-element detection with shorter analysis time. | The LIBS spectral signal is unstable due to system parameters, environmental changes, and sample components inhomogeneities. This instability affects quantitative detection, necessitating multi-pulsed data averaging. Matrix effects in soil types affect measurement accuracy. |
| [17] | LIBS rapidly determines soil fertility for agriculture.<br>Evaluates spatial variability in integrated agricultural systems. | Rapid determination of soil fertility improves agricultural production.<br>Evaluates spatial variability of soil fertility attributes accurately. | The study found that LIBS can accurately predict soil fertility parameters, but its model for labile P was less effective. The study identified two clusters, indicating that while LIBS can differentiate soil fertility levels, variability in soil types and conditions may hinder consistent predictive accuracy. |
| [18] | Rapid quantification of soil organic matter using LIBS.<br>Self-adaptive calibration improves prediction accuracy and robustness. | Rapid quantification of soil organic matter (SOM).<br>Improved prediction accuracy with optimized calibration strategy. | The LIBS technique's prediction performance is limited by its calibration strategy, which can lead to poor predictions due to soil background interference and low prediction accuracy due to low variances in target properties. |

**LIBS for Soil Contaminant Detection**

The recent research has contributed much as regards to the LIBS technology in the detection of heavy metals in the soils. Based on a thorough investigation of researchers of the University of Alabama, they used LIBS to identify arsenic (As), lead (Pb), and manganese (Mn) in the soil of Superfund sites in North Birmingham [19]. Modern studies have been concentrated on LIBS signal sensitivity enhancement means and methods. One of the significant improvements includes the conduction materials like Sodium Chloride (NaCl) and graphite to support

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the detection of lead in soil samples [2]. These studies proved that, conductive materials have the potential of enhancing quality of the signal and detections in analysis of heavy metals in soils. Also, there have been designs of novel methods of spatial confinement and resin enrichment as applied on cadmium detection in soil [20]. Such an approach was able to detect cadmium at the level of 0.132 mg/kg, which is far better than the normal LIBS techniques.

The current trends have led to an extension of LIBS use to a whole soil-plant interaction analysis. A 2024 study presented the new system based on the combination of LIBS with machine learning in analyzing garlic polluted with soil and discovering the source of contamination [21].

The recent advances of LIBS enhancement consist in the emergence of the nanoparticle-based methods and surface-enhanced methods. Research has also proved that gold nanoclusters when applied to a filter paper can increase the detection of heavy metals. These methods symbolize a novel era in the LIBS sensitivity increase, whereby it provides the hope of ultra-trace sensitivities of environmental chemicals with metals [22]. **Table 2** provides an overview of important studies on the application of LIBS in soil analysis, outlining the main objectives, benefits, and limitations of each, with a focus on contamination detection.

**Table 2: Key Studies on LIBS Applications for Soil Contaminant Detection**

| Ref. | Target Contaminants | Description | Benefits | Limitations |
|---|---|---|---|---|
| [19] | Heavy metals | Analyzes heavy metal contamination in North Birmingham soil.<br>Utilizes LIBS for detection. | Detects heavy metals in soil samples effectively.<br>Validates findings with ICP-MS measurements for accuracy. | Limit of detection for heavy metals specified.<br>Specific parameters required for accurate LIBS measurements. |
| [23] | Pollutants | LIBS detects pollutants in soil using laser-induced plasma.<br>Analyzes elemental composition through emitted light spectra. | Rapid analytical speed and easy sample preparation.<br>Multi-element detection capability in various mediums. | Signal intensity and stability inadequate for trace components.<br>Environmental conditions affect plasma lifespan and properties. |
| [2] | Pb | Study on Pb detection in soil using LIBS.<br>Signal enhancement with NaCl and graphite materials. | Accurate detection of heavy metals in soil.<br>Enhanced LIBS signal using conductive materials. | Soil matrix effect interferes with LIBS signal accuracy.<br>Quantitative detection accuracy is compromised by signal interference. |
| [24] | Cadmium (Cd), Argon (Ar) | Rapid detection of Cd in soil using LIBS.<br>LS-SVM under Ar condition shows best performance. | Rapid detection of Cd prevents soil heavy metal pollution.<br>Accurate quantitative detection aids environmental monitoring. | - |

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|---|---|---|---|---|
| [25] | Heavy metals | Novel method for detecting heavy metals in soil.<br>Uses gold nanoparticles-modified ion exchange membrane with LIBS. | Simple, rapid, and sensitive detection method.<br>High-efficiency enrichment of heavy metals in soil. | - |
| [26] | Heavy metals | Calibration-Free Picosecond Laser-Induced Breakdown Spectroscopy (CF-PS-LIBS) detects heavy metals in clover plants.<br>Analyzes plasma characteristics for environmental monitoring. | Enhanced accuracy in spectrochemical analyses.<br>Aids in developing effective remediation strategies. | - |
| [27] | Heavy metals | Mobile LIBS system detects heavy metals in soil.<br>Two methods: fixed measuring and handheld | Rapid, on-site monitoring of heavy metals in soil.<br>Effective detection with minimal sample pretreatment required. | Handheld mode shows decreased spectrum intensity and stability.<br>Matrix effects complicate quantitative analysis of soil samples. |
| [28] | Trace heavy metals | Detects heavy metals in soil using LIBS.<br>Solid-liquid-solid transformation improves detection sensitivity. | Improved detection sensitivity for heavy metals in soil.<br>Low-cost sample pretreatment enhances analysis efficiency. | - |

**Recent Advances and Future Directions in LIBS for Soil Analysis**

Schematic diagram to demonstrate the matrix effects, optimization and detection methods, and future directions in LIBS for soil analysis with detailed description is depicted in **Figure 1**.

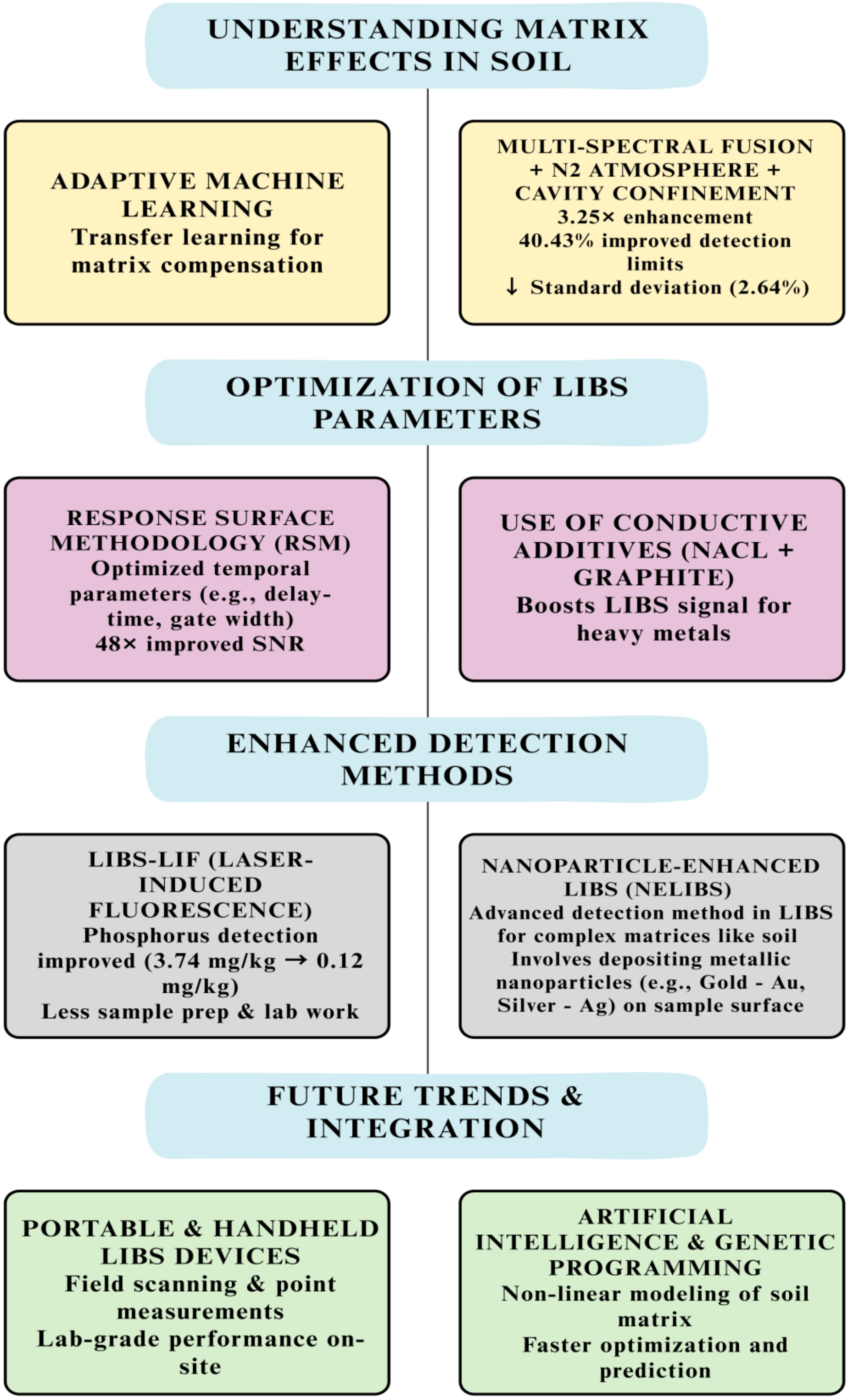


**Figure 1: Recent Advances and Future Directions in LIBS for Soil Analysis**

New studies have made a great step toward knowledge of the matrix effects in LIBS soil analysis. Machine learning (ML) techniques have come in as effective methods towards solving these issues. As recent study has been reported in 2024, it was found out that by utilizing adaptive learning structure involving transfer learning, matrix

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variation can be effectively compensated between different soil categories [29].

Response Surface Methodology (RSM) has now come up as a successful optimizing tool in LIBS parameters. In 2024 a study demonstrated the RSM optimization of temporal parameters (delay-time, interpulse delay, gate width and accumulated pulse) improved signal-to-noise ratio up to 48-fold over non-optimal conditions. The latter is important as it gives a systematic way in meeting the critical need to optimize the system temporal parameters that are in charge of signal collection and plasma evolution [30].

Recent scholarly research has shown better results about the multi-spectral fusion internal standard analysis models. Demonstrations made through research proved that N2 atmosphere enhancement coupled with confinement within a cylindrical cavity had an enhancement factor of 3.25 and enhanced reproducibility with relatively small standard deviation values of 2.64%. This two-fold improvement mechanism led to 40.43 % increase in the detection limits of strontium in soil samples [9].

The relatively new solutions to approach the problems of moisture content effects have been targeting the direct-focused laser ablation methods. In 2022, it was shown that direct-focused techniques of LA-LIBS have the ability to overcome the differences in the matrix more effectively, including the effect of moisture in particular [31]. This approach eliminates the need for carrier gas and provides more robust performance under varying moisture conditions.

Improvement of conductivity material has become a new technique. According to research conducted by Kim et al. (2024), incorporation of NaCl and graphite used in the study as a conductive agent were proven to boost LIBS signals when used in detection of heavy metals in soil. The method overcomes the dilemma in which "the signal of the target elements obtained in LIBS is susceptible to many interferences when detecting heavy-metal in soil type targets" [2].

Fluorescence aided by Laser has demonstrated a spectacular progress in detection limits. A recent study (2023) revealed that the LIBS-LIF methods enhanced the limits of phosphorus detection in clay soils in comparison to 3.74 mg/kg to 0.12 mg/kg. This mixed method greatly decreased preparatory sample and laboratory treatment as compared to traditional procedures [32]. Nanoparticle-Enhanced Laser-Induced Breakdown Spectroscopy (NELIBS) has emerged as an advanced technique in analytical chemistry, particularly for sensitive detection in complex matrices like soil. This approach involves depositing metallic nanoparticles commonly gold (Au) or silver (Ag) onto the sample surface before laser ablation [33].

Prospects of future advancements of LIBS are in development. The current development in the field of LIBS technology, data processing, and standardization have been making a mark in the role of LIBS in soil analysis in terms of field portability, analytical performance, and predictability. Handheld and portable LIBS equipment are getting very advanced. More recent innovations are platform spectrometers to be used in field scanning and handheld spectrometers to be used in point measurements. These systems are capable of laboratory grade performance in field portability [34].

AI involves developing systems that replicate aspects of human intelligence using techniques such as machine learning, language processing, and decision-making frameworks [35]. The application of artificial intelligence is transforming beyond the conventional machine learning. This new method has the capability to speed up the process of developing optimized formulations and process parameters [36]. Genetic programming strategies and the ensemble are investigated into the recent research to predict the soil property in a better manner. These are sophisticated algorithms that are able to make out non-linear relationships in soil matrix effects [37].

Assignments on standardization are reducing the challenges on reproducibility in LIBS systems. Further method reliability and acceptance is being advanced by the development of certified reference materials particular to LIBS analysis and interlaboratory comparison protocols [38].

## CONCLUSION

LIBS has become an intense and flexible technique used in soil science with speed, multi-elemental capability and minimal sample preparation. It is noted in this review that the application of LIBS, both as a method of detecting soil nutrients and soil contaminants, has seen great progress, which further statistically shows the urbanity of LIBS application in promoting sustainable agriculture. Improved methods such as spatial confinement, addition of conductive materials and multimodal set ups such as LIBS-LIF have further boosted its analysis capabilities specifically with regards to trace elements. Reproducibility issues are being resolved due to standardization efforts and the production of certified reference materials, and LIBS in soil science and precision agriculture are beginning to be accepted.

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