Ind. J. Pharm. Edu. Res., 2025; 59(1):30-36.
https://www.ijper.org

Review Article

Laser Induced Breakdown Spectroscopy: A Promising Analytical Technique

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ABSTRACT

Laser Induced Breakdown Spectroscopy (LIBS) is a rapid analytical technique. It has become an established analytical atomic spectrometry technique for the analysis of various samples. This method is widely used for determination of the elemental composition of various solids, liquids and gases as well as for the characterization and identification of the material. This technique is based on the optical detection of certain molecular and atomic species. Present review article aims to provide all the basic information related to Laser induced breakdown spectroscopy. In this review article, all the aspects of LIBS such as history, principle, instrumentation, advantages, limitations, as well as applications are described in brief.

Keywords: Laser Induced Breakdown Spectroscopy, Calibration Free Laser Induced Breakdown Spectroscopy, Analytical technique, Laser Induced Plasma Spectroscopy, Laser Spark Spectroscopy.

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Received: 14-07-2023;
Revised: 07-03-2024;
Accepted: 18-11-2024.

INTRODUCTION

Laser was invented in the year 1963; shortly after which Laser-induced breakdown spectroscopy was developed.[1] Many modern analytical techniques work on the basis of atomic spectroscopy to achieve the typical vaporization and excitation. Laser-Induced Breakdown Spectroscopy (LIBS) is one of them. Elemental analysis is done by using the rapid analytical technique which is Laser-Induced Breakdown Spectroscopy (LIBS) and this technique has been widely applied for various industrial applications. Short pulse of high energy radiation is used by LIBS that is generated by an analyser.[2] LIBS provides various advantages such as chemical free technique, portability, spatial information and rapid detection.[3] But its relatively low measurement repeatability is the major drawback of LIBS technique.[4] LIBS is also called as atomic emission spectroscopy. When atoms are in the high energy state, they are excited from lower to higher energy levels.[5] LIBS is also a straight forward and versatile spectroscopic technique from laser-induced plasmas that analyses spectral emission.[6] Provision of simultaneous multi-species measurements is the capability of LIBS so it is an emission technique.[7] LIBS are also called as Laser Spark Spectroscopy (LSS) and Laser Induced Plasma Spectroscopy (LIPS). By monitoring emission signals from the laser-induced plasma, the phenomena of Laser induced breakdown spectroscopy were first reported by Maker *et al*. in 1963. LIBS technique is based on the optical detection of certain molecular and atomic species. The principle of laser induced breakdown spectroscopy strongly depends on the conventional plasma atomic emission spectroscopy. It is a very useful technique for the determination of the elemental composition of various gas, solid and liquid samples. Choice of experimental condition is the parameter on which the analytical performance of the laser induced breakdown spectroscopic technique is strongly depend.[8] Nowadays, for qualitative and quantitative analysis laser induced breakdown spectroscopy is strongly used with the possibility of fast measurement in conjunction with no or small sample preparation. Laser induced breakdown spectroscopy has various types of industrial as well as security applications. It is found that LIBS is an emerging technology for real time chemical analysis. LIBS is an established instrumental analytical technique appearing in teaching labs of university.[9] LIBS has been applied for various analysis such as environmental, geochemical, archaeological and biological analysis to achieve great progress in understanding of the mechanism and detection performance. LIBS applications in agricultural field are more challenging as compared to other areas. These challenges are due to adverse working conditions and complex interaction between sample and sensors.[10] The major aim of this review paper is to provide a critical overview of the laser induced breakdown spectroscopy. It mostly focuses on the all aspects of LIBS like history, principle, instrumentation, advantages, limitations as well as applications.

DOI: 10.5530/ijper.20254665

HISTORY

Few groups worked on LIBS in the early 1980s.[11,12] In the year 2000 the 1st international conference on LIBS was conducted in Pisa, Italy. Later, meetings focused on work have been held in the European and Mediterranean areas on the odd years and international meetings every 2 years.

Year	History
1970-1960	In the year 1960 the 1st pulsed laser was developed by Ted Maiman. After that Brech and Cros demonstrated the first useful laser induced plasma on a surface in year 1962. The first analytical use of laser plasma on surfaces was found in the year 1963 which led to the invention of laser-induced breakdown spectroscopy. In the same year, use of laser plasma in a gas and laser micro-spectral analysis was demonstrated. Also, the initial investigation of laser plasma in liquids is done in year 1963. In 1964, time resolved laser plasma spectroscopy was introduced.
	In 1966, study of characteristics of laser-induced air sparks and direct analysis of molten metal with the laser spark was done. In the year 1970 continuous optical discharge was reported.
1971-1980	In 1971, the results of Q-switched laser were compared with normal laser pulses and the benefit of Q-switched laser was reported. Biological materials with LIBS were investigated in the year 1972. In 1980 laser spectrochemical analysis of aerosols was carried out and reported.
1981-1990	The use of LIBS for diagnosis of corrosion in nuclear reactors was reported in year 1982. The acoustic properties of the laser induced spark were first used in 1984. In the year 1988liquid samples and hazardous aerosols were analysed. In 1989 lots of attempts were made to increase the intensities of LIBS through magnetic and electric fields.
1991-2000	In 1992 laser plasma method was used for detection of metals in soil. While portable LIBS unit was developed for monitoring the surface contaminants. In 1995 the use of multiple pulse LIBS on the steel samples was reported and fibre optics delivery of laser pulses was also demonstrated. LIBS found applications in painted works of art as well as illuminated manuscripts in 1997. In 1998, on the basis of cone penetrometers, LIBS was used for subsurface soil analysis and also the use of echelle spectrometers coupled with CCD detectors was reported. Calibration free LIBS was introduced in 1999. For enhancement of LIBS performance, pulses from different lasers were used. On a NASA Mars rover demonstration of LIBS was done in 2000. In the same year, the 1st international conference on LIBS was held in Pisa, Italy.
2001-2010	In Orlando, Florida the 2nd international conference on LIBS wasorganised in year 2002. In 2004, 3rd international conference on the
	LIBS was held in Malang, Spain. Also, the use of LIBS for Mars mission was approved.

Principle

The principle of laser induced breakdown spectroscopy is similar to the principle of conventional plasma atomic emission spectroscopy. There is no need of sample transportation to the plasma in LIBS and this is the main difference between laser induced breakdown spectroscopy and conventional atomic emission spectroscopy. Vacuum air and low-pressure inert gases are the medium in which laser induced breakdown spectroscopic experiment can be performed.[13] High energy laser pulses are utilized to create a high temperature micro plasma at the surfaces of the target as the vaporization, excitation and atomization source. LIBS is based on the spectroscopic analysis of plasma emitted radiation obtained from the interaction of focused and strong enough laser beam with a sample. Various species like ions, electrons, atoms, molecules and also small diatomic molecules are present in the laser induced plasmas.[14]

Instrumentation

The basic parameters that are required to be considered to understand and control a LIBS experiment are: first, those related with the direct interaction of laser with the sample then, the interaction of laser with the ablated materials such as atoms, ions, tiny particles, electrons and molecules and also the coupling of its remaining pulse energy. To obtain the condition of sensitive, reproducible excitation and also spectral emission the above phenomenon is of primary importance.[2]

A typical LIBS instrument is made up of following parts (Figure 1):

- A laser source;
- An optical system;
- A wavelength analyzer.
- Detectors.

Laser

Providing stable and sufficient pulse energy for plasma generation is the main purpose of LIBS lasers. Important criteria related to lasers are: pulse time, wavelength, energy per pulse and number of pulses per burst.[15] The wavelength of the laser depends on some

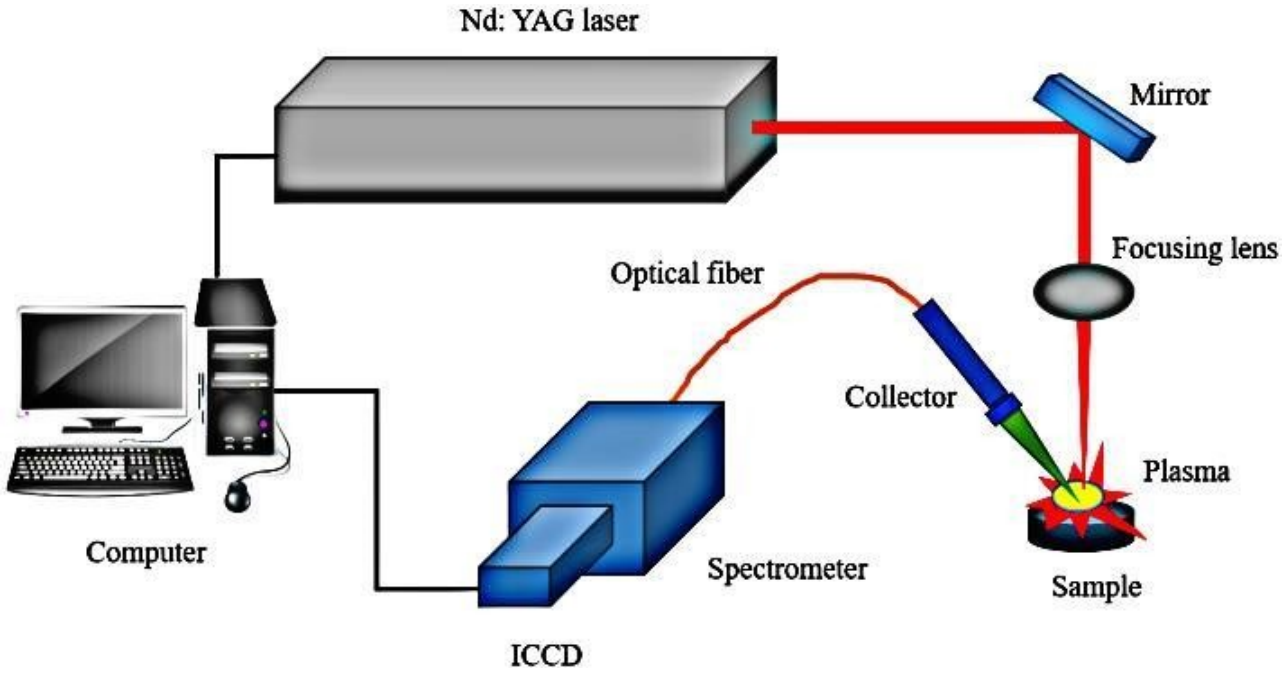

Figure 1: Instrumentation of Laser Induced Breakdown Spectroscopy.

parameters such as: B. Transition from higher to lower energy levels, resulting in the emission of radiation and the composition of the active medium in the laser cavity. Different types of solid-state lasers are used in LIBS, such as Nd:YAG laser, ruby laser, carbon dioxide gas laser, nitrogen laser and excimer laser.[16] Nd:YAG lasers are most commonly used to generate plasmas in LIBS experiments. This laser provides a reliable, compact and simple method to generate plasma in LIBS experiments. Typical output energies from Nd:YAG lasers range from 10 to 100 millijoules per pulse, with peak powers in the megawatt range.[13] The basic wavelength of Nd:YAG laser is 1064 nm.[5] The Nd:YAG laser delivers approximately 300 mJ of energy at 532 nm. Fluctuations in LIBS are the biggest problem because only very small amounts of sample (usually micron-sized spots) are used for analysis.[5]

An Optical System

In LIBS, optical components are crucial to collect the radiation emitted by the plasma in order to direct the laser radiation onto the surface of the sample and wavelength selector. To change the direction of the laser beam, when the laser beam is not directed at the sample surface, a dichroic mirror or prism must be used, such as a rotating or folding prism. Another type of optics is used in laser-induced breakthrough spectroscopy to collect the radiation emitted by the plasma and focus it onto a laser beam. Fiber optics are used to collect the radiation emitted by the plasma or guide the laser light, or both. Laser light and radiation are propagated through various fibers that are emitted from the plasma when using fiber optic cables.[17-19] In most cases, LIBS consist of optical fibers made of quartz glass.[8]

Wavelength Analyzer

Wavelength is an important factor in the initiation of plasma.[20] Capability of covering the wide wavelength ranges with higher solution is an important criterion for the ideal wavelength selector. Wavelength analyser not only decreases the capability of multi-element detection but also covers a wide wavelength range with less resolution. To overcome these limitations, four grating-based spectrographs were employed by the Body and Chadwick which are increasingly used in LIBS.[21]

Detectors

In order to collect, resolve and measure atomic emission lines from the laser-induced plasma detectors are used in LIBS. Photographic plate was used as a detector in the early days, which has the advantage of a relatively low cost with wide wavelength range. But due to disadvantage of being time consuming with low reproducibility it is not used nowadays.[16] Based on their application, different types of detectors are used in the laser induced breakdown spectroscopy (Figure 2). Avalanche Photodiode (APD) or a Photomultiplier Tube (PMT) can be used to measure the light intensity without any spectral decomposition. Combination of a spectrograph and a Photodiode array or an intensified Photodiode array can be used for one dimensional spatial information and time-resolved measurements. Commonly used devices to obtain two-dimensional spatial information are Intensified Charge-Coupled Devices (ICCD) and Charge-Coupled Devices (CCD). Charge-coupled devices provides less background signal while intensified charge-coupled devices is better for time resolved detection and it improves signal-to-noise ratio as well. The problem of ICCD is that its

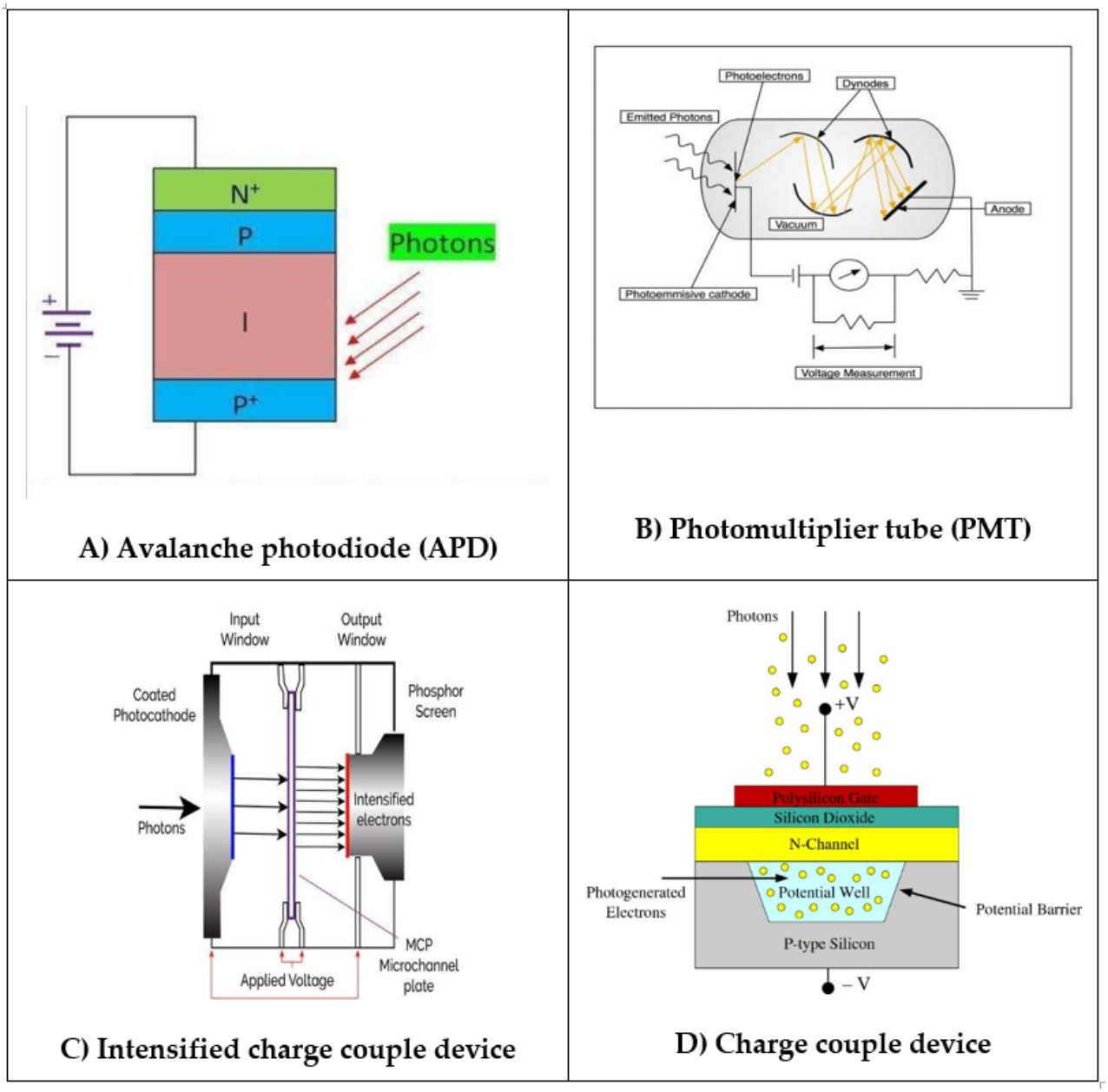

Figure 2: Types of detectors.

price is much higher than the CCDs.[22] To obtain the temporal development of emission lines for LIBS a monochromator is used.

ADVANTAGES

- No need of sample preparation.
- Vaporization of very small amount of material. (10s of ng).
- Atomization and excitation occurs in one step.
- Capable of multi-elemental analysis.
- Easy analysis of refractory materials.
- All states of matter can be analysed.[23]

LIMITATIONS

Based on the type of specimen and the experimental apparatus used, the detection limits for the LIBS may vary from one element to the other.[24]

- Use of high laser power can damage the optics.
- Technological integration in the environment can be difficult.[25]
- Finding useful or useless.

spectra depends on a number of elements, including the sample matrix, ambient circumstances and experimental parameters for spectral measurements. But it has certain drawbacks that stand in the way of LIBS's ability to provide trustworthy analysis.[26]

APPLICATIONS

Nuclear Application

Laser induced breakdown spectroscopy has a most important applications in the nuclear field such as contamination and leakage detection and the analysis of *in situ* materials for inspection. LIBS also have the two potential roles such as bulk analysis and end point detection for preservation of bulk materials. 2-dimensional analysis of multi-layered plasma facing component samples in vacuum environment is also done with the help of LIBS. Laser induced breakdown spectroscopic technique is also used for the monitoring of retention of fuel and deposition of impurities on the first wall of EAST. LIBS is also used as an online control technique for cleaning process of the laser of polluted first mirror by Hai *et al.*[27]

Explosives

LIBS also have the very great potential for rapid and remote detection of the explosives. By using chemometric approaches Wang *et al.* analyses the LIBS spectra of certain organic and inorganic explosives. Wang *et al.* also investigated the time resolved LIBS spectra of sodium nitrate, potassium nitrate and black powder.[28]

Industrial Application

Laser induced breakdown spectroscopy has wide range of industrial applications such assorting, recycling, monitoring of process as well as control of quality of product during manufacturing.

Pharmaceutical Industry

LIBS has been used to get the composition of alloy. Also, the characterization and quality control of pharmaceutical products is done with the help of LIBS.[28] It is also used in analysis of saline solution and drug mapping. In recent years, LIBS has the great potential for quality control & analysis of the traditional medicinal pharmaceutical industry.

Waste Management Industry

LIBS are used for the sorting and identification of plastic substances. LIBS are also used for achieving the control on process of recovering of precious metals from electronic wastes. Efficient sorting of nonferrous metallic scrap from the automotive industry is done with the help of LIBS.[29]

Biomedical Applications

Analysis of chemical compositions of biological samples such as tissues, fluids and human bones is done by using laser induced breakdown spectroscopy. This technique is also helps to detect the deficiency or excess of minerals as well toxic elements in teeth, bones, nails and tissues. This technique is also used for the analysis as well as characterization of biological aerosols. LIBS are also used as a bio aerosol classifier. LIBS also provide a rapid and non- destructive tissue analysis and it has ability to differentiate between normal and malignant tissues. Calibration free laser induced breakdown spectroscopy is used for the measurement of main minerals concentration present in the human hairs. LIBS is also used for the of traces element detection present in the skin of human being.[30] Detection of cancer is also possible with the help of LIBS. Surgical device can be provided by the LIBS for detection and eradication of tumour cells.

Microbiological Assessment

LIBS have the great potential for bacterial detection on the basis of their unique atomic composition. It is also used for the bacterial detection in the food products. This technique is used for the determination of moisture content present in the dairy products such as cheese. LIBS are useful analytical tool for the determination of pesticides.[31] Quantification of various elements present in microorganisms is done by using LIBS technique. LIBS technique is also used for the differentiation of bacterial strain according to their ever-changing accumulation of certain elements. Double pulse stand-off laser induced breakdown spectroscopic technique is used for the detection of various types of hazardous materials. LIBS technique is also used for the detection of bacteria present in the soil.[32]

Agricultural Product Analysis

Metallic elements present in the tobacco ash and tobacco can be analyzed by the LIBS.[33] LIBS is also used for the quantitative analysis of navel orange. Time resolved characterization of laser induced plasma was studied by the Lei *et al.* from the fresh potatoes. Kim *et al.* quantifies the nutrients present in the rice and spinach.[34] LIBS technology used for organic carbon content, total nitrogen and total phosphorus determination in agricultural soil. This technique is also used for the measurement of carbon density of the soil. LIBS technology is also used for early diagnosis of plant diseases such as citrus cancer, nutrient deficiencies and Huanglongbing (HLB) bacterial destructive diseases.[35]

Application to Nanomaterials

LIBS are used for the analysis and online monitoring of the nanomaterials. It is also used for counting of nanoparticles in colloidal suspension. Also used for the analysis of nanoparticles powder. LIBS technique is also used during the process of ultrafiltration. Enhanced spectra of the transparent media such as glasses are obtained with the help of LIBS without causing any damages or cracking of the sample. LIBS are also used for the mapping of distribution of inorganic materials without help of labelling. LIBS is also used for the differentiation of carbon nanomaterials.[9]

Multivariate Analysis

Multivariate analysis is a chemometric method which is done with the help of LIBS; multivariate analysis involves more signal information unlike univariate analysis. Multivariate analysis is also used for the reduction of data dimensionality and extraction of relevant information.[36] Multivariate analysis has been applied as a calibration strategy for prediction of the elemental concentration. It is also used for the online monitoring of the restoration process.[10]

LIBS for Restoration

LIBS are used as diagnostic tools for artwork conservation by monitoring the effectiveness of laser cleaning process. Double pulse LIBS configuration is also applied for restoration purpose. Double pulse LIBS is used for cleaning of ancient alloy and bronze. LIBS also have the ability to perform depth profile analysis. Restoration of archaeological samples is done with the help of LIBS technique. This technique is the diagnostic tool for this purpose. LIBS is also used for the identification of the eroded materials present in the cleaning stage also it avoids the sample damage.[37]

Environmental Monitoring

LIBS has been used for various purposes of monitoring of environmental conditions such as fast, remote and *in situ* detection as compared to other element analysis techniques. Calibration free LIBS are used for the quantification of the various harmful elements in industrial sludge. Detection of heavy metal pollution contain by water sources is done with the help of this technique. Quantification of soil collected from the vicinity of the leather industries is done with the help of magnetic field assisted calibration free LIBS.[38]

Water monitoring

Long-term consumption of water contaminated with high levels of heavy metals has been linked to a number of illnesses. For instance, illnesses of the nervous and digestive systems might result from consuming water contaminated with high levels of lead. Water contamination monitoring is so vital and urgent. Water detection has made extensive use of LIBS, an essential detection technology, in recent years. Indirect liquid detection using LIBS resulted in low detection sensitivity and stability because of issues with liquid splash and plasma quenching. Therefore, a number of pretreatment techniques, such as liquid flow, liquid-solid conversion, liquid enrichment and so forth, have been developed to improve the detection sensitivity and spectral stability.

Deep sea-exploration

With the ocean making up around 71% of the planet's surface and having an average depth of 3792 meters, it is one of the planet's most difficult and inaccessible environments. Deep-sea detection is not possible for many detection techniques due to extreme conditions including hypoxia and high pressure. Because of LIBS's benefits, maritime exploration has seen a rise in the use of LIBS. LIBS employed an on-deck equipment known as "AQUALAS " for underwater detection as early as 2012. The main body of AQUALAS was installed on board and an umbilical cable was used to supply laser pulses and the LIBS signal. The LIBS detection was carried out by a diver who held the LIBS probe to touch alloy sample.[39]

Other Applications

Analysis of painted artwork, ceramics, wood and stone can be done with the help of LIBS. With the help of LIBS technique, the fast analysis of atmospheric pressure can be done.[39] LIBS is used for the detection of unburned carbon present in the fly ash. LIBS are also used for the measurement of fuel-air ratio in combustion.[40] LIBS is also used to determine the homogeneity of the sample and detection of element in another planet. Used for the conservation of pigments and ancient metals. It is also used for the fuel reprocessing in the fuel industry. It is also used for the paper and polymer analysis. LIBS is also applied for compositional mapping of commercial printed circuit board.[41,42]

CONCLUSION

Laser induced breakdown spectroscopy is a potential analytical technique used for sample analysis. It has wide variety of applications and will be used widely in the upcoming years for determination of elemental composition of materials. It is a very versatile technique as it can be used on any type of material viz. solid, liquid and gas. This review article provides all basic information related to the laser induced breakdown spectroscopy which is helpful for researchers as well as students who work on LIBS.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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Cite this article: Mehta P, Populwar V, Shinde N, Dhebe D, More P. Laser Induced Breakdown Spectroscopy: A Promising Analytical Technique. Indian J of Pharmaceutical Education and Research. 2025;59(1):30-6.