COMPARISON AND OPTIMIZAITON OF GATED AND UNGATED LIBS SYSTEMS

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Dedicated to my family for their continuous moral support during whole of my academic career.

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ABSTRACT

Laser-induced breakdown spectroscopy (LIBS) is a direct and versatile analytical technique that performs the elemental composition analysis based on optical emission. The performance of the LIBS technique relies on the choice of experimental conditions. High cost, accuracy and precision insufficiency, understanding of experimental conditions are key challenges in LIBS. In this research work, a comparison between gated and ungated LIBS systems is presented to determine the efficiency and applicability of ungated LIBS system in context of quantities analysis of elements. Two different LIBS systems gated (high cost) and ungated (low cost) are compared for quantitative analysis of Ca and Mg elements which are prepared in laboratory with known composition. Optimization of experimental parameters (Gate delay, integration time, and laser energy) has performed for both gated and ungated LIBS systems to compare performance of two systems based on signal to background ratio. The calibration curves are plotted to determine the accuracy and sensitivity of both gated and ungated systems. The accuracy of system is obtained from regression (R^2) and sensitivity from the slope of plotted calibration curves at optimized conditions for both LIBS systems. The R^2 and slope values obtained from calibration curves show that the gated measurements are better as compared to those values obtained from the ungated measurements. However, the $R^2 > 0.8$ obtained from calibration curves with ungated measurements indicates the potential of ungated LIBS for quantitative investigations which can be further improved by mitigating the influencing factor.

ABSTRACT

LIBS ialah teknik analisis secara terus dan serba guna yang dapat melakukan analisis komposisi unsur berdasarkan pancaran optik. Prestasi teknik LIBS bergantung kepada pemilihan syarat eksperimen. Kos yang tinggi, kejituan dan kepersisan yang lemah, dan pemahaman syarat eksperimen adalah cabaran utama terhadap LIBS. Dalam kerja penyelidikan ini, perbandingan antara sistem LIBS berpintu dan tak berpintu dikemukakan untuk menentukan kecekapan dan kebolehgunaan sistem LIBS tak berpintu dalam konteks analisis kuantitatif keunsuran. Dua sistem LIBS yang berbeza, pintu (kos tinggi) dan tak berpintu (kos rendah) dibandingkan untuk analisis kuantitatif bagi unsur Ca dan Mg dimana unsur-unsur ini telah disediakan didalam makmal dengan komposisi yang diketahui. Pengoptimuman parameter eksperimen (lengah berpintu, masa integrasi, dan tenaga laser) telah dilakukan untuk sistem LIBS berpintu dan tak berpintu untuk membandingkan prestasi dua sistem berdasarkan nisbah isyarat terhadap latar belakang. Keluk tentukuran ditandakan untuk menentukan kejituan dan kepekaan kedua-dua sistem pintu dan tak pintu. Kejituan sistem diperolehi daripada regresi dan kepekaan daripada cerun yang ditandakan oleh keluk tentukuran pada syarat yang teroptimum untuk kedua-dua sistem LIBS. R^2 dan nilai cerun yang diperolehi daripada keluk tentukuran menunjukkan bahawa ukuran berpintu adalah lebih baik dibandingkan nilai yang diperolehi daripada ukuran tak berpintu. Walau bagaimanapun, nilai $R^2 > 0.8$ yang diperolehi daripada keluk tentukuran daripada ukuran tak berpintu menunjukkan keupayaan LIBS tak berpintu terhadap penyiasatan kuantitatif dimana dapat diperbaiki dengan mengurangkan faktor yang mempengaruhi.

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LIST OF ABBREVIATIONS

et. al.	-	and others
Ar	-	Argon
ANN	-	Artificial Neural Networks
Ba	-	Barium
Ca	-	Calcium
Ca II	-	Calcium (Ionic type)
Ca I	-	Calcium (Neutral type)
$CaCl_2$	-	Calcium Chloride
CCD	-	Charge-Coupled Device
Cr	-	Chromium
Cu	-	Copper
EMCCD	-	Electron Multiplying CCD
H	-	Hydrogen
ICP	-	Inductively Coupled Plasma
ICP-OES	-	Inductively Coupled Plasma Optical Emission Spectrometry
Fe	-	Iron
KrF	-	Krypton Fluoride
LIBS	-	Laser-Induced Breakdown Spectroscopy
LIP	-	Laser-Induced Plasma
Pb	-	Lead
Li	-	Lithium
LTE	-	Local thermodynamic equilibrium
Mg	-	Magnesium
Mg II	-	Magnesium (Ionic type)
Mg I	-	Magnesium (Neutral type)
Mn	-	Manganese
MSM	-	Mechanically Separated Meat

Mo	-	Molybdenum
NIST	-	National Institute of Standards and Technology
Nd:YAG	-	Neodymium-doped Yttrium Aluminium Garnet or $Nd:Y_3Al_5O_{12}$
Ni	-	Nickel
N	-	Nitrogen
O	-	Oxygen
PLSDA	-	Partial Least Squares Discriminant Analysis
PLS-R	-	Partial Least Squares Regression
P	-	Phosphorous
K	-	Potassium
KBr	-	Potassium Bromide
RF	-	Random Forest
SBR or S/R	-	Signal-to-Background Ratio
SNR or S/N	-	Signal-to-Noise Ratio
SIMCA	-	Soft Independent Modelling of Class Analogy
W	-	Tungsten
UV	-	Ultra-violet

LIST OF SYMBOLS

cm	-	Centimeter
°	-	Degree
eV	-	Electron Volt
i.e.	-	That is
J/cm^2	-	Joule per centimeter square
kN	-	Kilo-Newton
µg	-	Micro-gram
µJ	-	Micro-Joule
µm	-	Micro-meter
µs	-	Micro-second
mbar	-	Mili-bar (unit of pressure)
mJ	-	Mili-Joule
ms	-	Mili-second
nm	-	Nano-meter
ns	-	Nano-second
ppm	-	Part per million
Pa	-	Pascal (unit of pressure)
%	-	Percentage
psi	-	pound per square inch
R^2	-	Regression coefficients

CHAPTER 1

INTRODUCTION

1.1 Research Background

Laser-induced breakdown spectroscopy (LIBS) is a direct and versatile analytical technique that performs the elemental composition analysis based on optical emission produced by laser induced plasma, with a little pre-treatment procedures or nearly no sample preparation. Moreover, LIBS has outstanding capabilities to provide real time in-situ and simultaneous multi-element detection of materials (i.e. solid, liquid or gas), unique elemental identification as well as the remote and stand-off analysis in various fields of applications of science and technology [1, 2].

The capability of real-time analysis gives LIBS potential to test larger number of samples and replace the existing analytical tools. Currently, LIBS technique has successfully been applied in different technological fields such as manufacturing processes, material science, biology, archeology, forensics, geological and environmental materials, food products, agricultural products, biomedicine, space exploration, radiological and nuclear materials and so on [3].

Typically, the LIBS system consists of number of components which can be variably setup in different configuration for laboratory experiments, made compact or portable for field use. LIBS system can be configured for either close-in analysis, remote or stand-off detection at far distance. The main components of the LIBS system are (i) a solid-state, Q-switched pulsed laser is used to induce a plasma on the target surface, (ii) optical lenses to focus the laser beam onto a sample surface and to deliver the optical emission produced during the plasma event (iii) detection unit includes spectrometer, and (iv) a computer to control whole LIBS system and data acquisition processing [1].

In LIBS, a high energy, ultra-short pulse duration, pulsed-laser is closely aligned and focused on to a target surface causing the breakdown of the material and the formation of extremely high temperature induced plasma which contains excited ionic, atomic, molecular and neutral species. During plasma generation, small amount of the material is ablated and vaporized from the target surface. As laser induced plasma cools down gradually, optical emissions are radiated. Then, the plasma emission is spectrally recorded and measured for elemental composition analysis of the target material [1, 4].

Although, LIBS is rapid and in situ rapid analytical technique, there are several issues associated to LIBS technique for instance efficient quantification, high cost, accuracy and precision insufficiency, understanding of experimental conditions contribution and extensive maintenance [5]. Most of the LIBS experiments are generally conducted using gated system in which a pulse generator is introduced in the LIBS setup to control the gate delay time and shutter opening time during detection of optical signal to enhance signal-to-background (SBR) and signal-to-noise (SNR) ratios [6, 7]. As the cost of system is also an important consideration which can be cut down greatly using spectrometer with an ungated camera. The ungated LIBS system can provide a simple, cost-effective and robust tool [8]. On the other hand, ungated LIBS experiments have been conducted recently by several research groups using low cost spectrometers and simplified experimental configurations [9]. Therefore, it indicates the feasibility and applicability of using ungated system to conduct multi-elemental analysis in combination with optimized parameters (i.e. laser pulse energy, wavelength, pulse duration, focusing and detection geometries), approaches and operating conditions.

1.2 Problem Statements

LIBS is capable of performing rapid, in-situ, multi elemental composition analysis of virtually any materials regardless of their physical conditions, depth profiling, elemental surface mapping, diagnosing and solving the industrial and scientific problems in real time, with minimum or no sample preparation as compared to other common techniques (e.g. thermal ionization mass spectrometry, atomic absorption spectrometry, inductively-coupled mass spectrometry) that often require complex and time-consuming laboratory procedures. Despite of all the advantages and capabilities that LIBS technique possesses, it does have several drawbacks reported in the studies and, however, most of the LIBS system employ the gated control with complex and expensive setup cost that including the gated control features (e.g. gate delay time, gate width, integration time, device-control synchronization), intensified charge-coupled device (ICCD) camera as a detector and high resolution spectrometer. In addition, the early stage of laser-induced plasma is inevitably dominated by the continuum emission whose time of decay can be affected by the experimental parameters such as laser energy, laser wavelength, laser pulse duration, sample material and ambient pressure etc.

In present research work, a comparison between gated and ungated LIBS systems is presented to explore their performance, optimize the experimental parameters such as gate delay and laser energy etc. used in the systems, and the applicability of ungated LIBS system, with respect to the quantification analysis of elements. The Calcium and Magnesium elements are chosen to perform the comparative analysis as these elements are found to be present in most of the food and geological samples. Additionally, the target samples which consist of different concentration of Ca and Mg were prepared in the laboratory. The effect of chosen gate delay and laser energy on specific emission intensities of interest, signal to background ratio (SBR) and continuum emission, has been studied and discussed in detail in the subsequent chapters.

1.3 Objectives

The main objective of research is to explore the performance of ungated LIBS system in comparison with gated LIBS system in terms of quantitative estimations of Calcium (Ca) and Magnesium (Mg) concentrations in laboratory prepared samples.

The specific objectives of the research are:

i. Optimization of experimental parameters (i.e. gate delay and laser energy) in gated and ungated LIBS systems on the basis of SBR.
ii. Quantitative estimation of Ca and Mg in samples using gated LIBS system and ungated LIBS system in separate experiments.
iii. Performance comparison of gated and ungated LIBS systems for quantitative estimation of Ca and Mg.

1.4 Scope of Research

In this study, experiments are performed on two different LIBS setups, one is low cost ungated LIBS system and the other is expensive gated LIBS system. Eight target samples are carefully prepared and compacted into the form of hard pallets, each sample having unique concentration of Ca and Mg. Experimental parameters such as laser energy and gate delay, are optimized using whole range of values allowed on the system. A permissible range of laser energy (5 mJ ~ 650 mJ) and gate delay (0 ~ 23.75 μs) is used on gated LIBS system and three laser energies only (236 mJ, 350 mJ and 400 mJ) are used on ungated LIBS system due to the limitation of the system can support. Using ungated LIBS system, the elemental measurements are conducted without synchronizing the laser source with the spectrometer. In contrast, with gated LIBS system, the measurements are conducted at optimized gate delay time. The experiments for both gated and ungated systems are conducted at atmospheric pressure under the same conditions.

1.5 Significance of the Study

With the empirical optimization of LIBS operating parameters and conditions, a reliable quantitative and qualitative elemental analysis of the target samples can be highly achieved. In addition, the intensities of detected emission lines will be closely proportional to the elemental concentrations of the samples. Typically, the measured emission lines from the atomic species can be unavoidably suppressed by continuum radiation during the initial stage of plasma formation and the continuum radiation is possibly caused by the radiative recombination and the Bremsstrahlung effect etc. Therefore, an optimized delay time for the acquisition window, optimized operating parameters such as laser energy and laser pulse duration (i.e. micro-second to femto-second), sample material and environmental condition are taken into consideration in order to reduce the undesirable effect of the continuum radiation during the measurement. On the other hand, the comparative performance of gated and ungated LIBS systems suggests the possibility and applicability of using the ungated LIBS system for quantitative investigation. Moreover, it can simplify the experimental setup in terms of selected configuration that suited for particular experiment and cost of the components that will enable the current LIBS development to future compact, portable or even handheld devices designed for use in the fields of various applications including food, biomedical, pharmaceutical, agricultural and so on.

1.6 Thesis Outline

Present thesis includes 5 Chapters in total. Chapter 1 contains background history, problem statement, research objectives, significance and scope of study. Chapter 2 includes the literature review related to this research. Chapter 3 describes the research methodology which contains description of experimental materials, equipment, procedures, parameters and flow chart of the experimental methodology. Chapter 4 presents the results obtained from experiment and discussion on the results. The conclusion and future work is described in Chapter 5.

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