

**DISCRIMINATION OF DIFFERENT TYPE OF MEATS USING  
LASER INDUCED BREAKDOWN SPECTROSCOPY AND  
CHEMOMETRIC TECHNIQUES**

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LASER INDUCED BREAKDOWN SPECTROSCOPY AND  
CHEMOMETRIC TECHNIQUES

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This thesis is dedicated to my family for their endless love and support.

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## ABSTRACT

Laser-induced breakdown spectroscopy (LIBS) is an analytical technique used for the identification of elements by analysing the emission line spectrum from samples. In this research, the possibility of classification of raw meat species based on emission spectra by using laser induced breakdown spectroscopy (LIBS) and chemometric techniques such as principal component analysis (PCA) and support vector machine (SVM) were implemented. An experimental setup was developed using Q-Switched Nd:YAG laser operating at 1064nm (208mJ per pulse) and a spectrometer connected to a fiber optic in order to collect the atomic emission. Different types of muscle tissues (beef, mutton, pork, fish, and chicken) were prepared as samples for the ablation process and the procedure for pork sample followed a specific guideline. The LIBS experiment was able to detect the elements in the meat samples such as magnesium, iron, calcium, sodium, carbon, nitrogen, and hydrogen. The raw spectra data were preprocessed and grouped into six datasets for PCA and SVM analysis. Standard ratio combination dataset showed the best result of PCA with variance of 99.8% which were later used for SVM classification. In SVM classification, the maximum accuracy of 89.33% was achieved by using a splitting ratio of 70:30 and linear kernel. The results obtained suggest a successful classification on the target tissues with high accuracy. This is valuable for an automatic discrimination in food analysis.

## ABSTRAK

Spektroskopi runtunan aruhan laser (LIBS) adalah teknik analisis yang digunakan untuk mengenalpasti unsur-unsur dengan menganalisis spektrum garis pancaran dari sampel. Dalam kajian ini, keupayaan untuk mengkelaskan pelbagai jenis daging mentah berdasarkan spektrum pancaran dengan menggunakan teknik spektroskopi runtunan aruhan laser (LIBS) dan teknik kemometrik seperti analisis komponen utama (PCA) dan mesin vektor sokongan (SVM) telah dilaksanakan. Peralatan eksperimen telah dibangunkan dengan menggunakan laser Nd:YAG bersuis-Q beroperasi pada 1064 nm (208 mJ per denyut) dan spektrometer yang disambung dengan gentian optik untuk mengumpulkan pancaran dari atom. Pelbagai jenis tisu otot (lembu, kambing, babi, ikan, dan ayam) telah diambil sebagai sampel untuk proses ablasi ini dan prosedur untuk daging babi mengikuti garis panduan yang khusus. Eksperimen ini dapat mengesan unsur-unsur dalam sampel daging seperti magnesium, besi, kalsium, sodium, karbon, nitrogen dan hidrogen. Data spektrum mentah telah diproses dan dibentuk menjadi enam dataset untuk analisis PCA dan SVM. Dataset nisbah kombinasi piawai menunjukkan hasil yang terbaik daripada analisis PCA dengan variasi 99.8% yang kemudiannya digunakan untuk pengkelasan SVM. Dalam pengkelasan SVM, ketepatan maksimum 89.33% telah tercapai dengan menggunakan kadar pecahan 70:30 dan kernel linear. Keputusan yang diperoleh menunjukkan keupayaan mengkelaskan tisu sasaran dengan kejituan yang tinggi. Hasil kajian ini sangat bernilai untuk pengasingan secara automatik dalam menganalisis makanan.

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## LIST OF SYMBOLS

$X$	-	Original data matrix
$C$	-	Matrix consisting of the wavelengths of each element
$S$	-	Matrix consisting of the intensity of each elements
$E$	-	Error matrix
$X_C$	-	Centered data matrix
$\bar{x}$	-	Mean
$P$	-	Loading matrix
$T$	-	Score matrix
$\mu\text{s}$	-	Microsecond
ns	-	Nanosecond
J	-	Joule
Hz	-	Hertz
mm	-	Millimeter
$\text{cm}^2$	-	Centimeter squared
W	-	Watt
nm	-	Nanometer
$x_n$	-	Normalized value for variable x
$x_o$	-	Original value for variable x
$x_{min}$	-	Minimum value in data sample
$x_{max}$	-	Maximum value in data sample
$k$	-	SVM classifier
$n$	-	Number of class
C	-	Cost
g	-	gamma

## LIST OF ABBREVIATIONS

LIBS	-	Laser induced breakdown spectroscopy
SVM	-	Support Vector Machine
PCA	-	Principal Component Analysis
PCR	-	Polymerase Chain Reaction
ELISA	-	Enzyme Linked Immunosorbent Assay
IR	-	Infrared
FTIR	-	Fourier-Transform Infrared Spectroscopy
NIR	-	Near infrared
ICP-AES	-	Inductively coupled plasma-atomic emission spectrometry
ICP-MS	-	Inductively coupled plasma-mass spectrometry
AA	-	Absorption spectrometry
LA-ICP-MS	-	Laser ablation inductively coupled plasma mass spectrometry
PLS	-	Partial least square
Thz	-	Terahertz
RBF	-	Radial Basis Function
ICP-OES	-	Inductively coupled plasma-optical emission spectrometry
NIST	-	National International Standard

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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background of Study**

In early 2013, the horsemeat burger scandal is ongoing in Europe especially in Irish and British supermarkets when frozen beef burgers has been discovered contained horse DNA. Moreover, an analysis done by The Food Safety Authority of Ireland (FSAI) that pig DNA were found in 23 samples of beef burgers which are prohibited for Muslim communities. Thus, testing of food products to assure consumer protection against fraudulent practices in the food industry is of a greater interest.

Food adulteration with non-halal ingredients is becoming a common phenomenon in food industries. Adulteration occurs when high cost raw material is swapped with cheaper materials for reducing their production cost. Such cheap ingredients can jeopardize health of the consumers who may be allergic to specific foods and emotionally disturbed due to religious reasons. For this purpose, different analysis based on certain identified biomarkers such as oil/fat-based, protein-based, DNA-based and metabolite-based were proposed for halal products authentication (Che Man and Mustafa, 2010).

After all, laser induced breakdown spectroscopy (LIBS) is one of several analytical techniques that can be deployed in authentication of halal products. Over the past decade, intense scientific activity has been study of LIBS in identification of elements by analyzing the emission line spectrum from samples. The reason is its potential advantages like simple experimental setup, very little or no sample preparation and universal type of samples.

Combination of LIBS with chemometric methods provides a powerful approach in pattern recognition and classification. Most recently, the use of LIBS spectra in combination of support vector machine (SVM) has applied successfully in discrimination of rocks (Zhu *et al.*, 2014). Moreover, a successful classification using SVM had done on different types of proteins from LIBS spectra has potential in detection ovarian cancer (Vance *et al.*, 2010). This proves the ability of LIBS to distinguish between the biological species with similar compositions on the basis of their spectral signatures.

## **1.2 Research Problem**

Food adulteration especially in meat products is becoming a common phenomenon in food industries. For this purpose, scientists come up with some various approaches. The most commonly approach is to use some analytical methods derived from the measurements of the physical or chemical characteristics of specific components present in the food products. However, the currently available analytical techniques require sample preparation especially in chemical form. This type of chemical preparation is a time-consuming and sometimes labor-intensive process.

Combination of LIBS and chemometrics analysis has a great potential in identification and classification of biological samples for many application in recent years. Kanawade *et al.* (2013) found that application of LIBS with multivariate analysis has successfully differentiated four different structures of tissue types (skin, muscle, fat, and nerve). Instead of using multivariate analysis, machine learning such as Support Vector Machine (SVM) is proposed to increase the accuracy of LIBS in qualitative analysis. Hence, this study will try to discriminate between five different type of meats (beef, chicken, lamb, pork, and fish) which including a non-halal meat by using LIBS with PCA and SVM application.

### **1.3 Objectives of Study**

- To obtain spectral lines from various types of meats using LIBS.
- To identify the elements present in all meat samples.
- To establish performance of PCA in dimensional reduction and classification of different type of datasets
- To differentiate between different types of meats from the best separation dataset using SVM.

### **1.4 Scope of Study**

Nd:YAG laser was used to induced breakdown and generate plasma formation onto the meat species. The plasma emission spectrum will provide information and hence, the factors affecting the plasma such as laser characteristics, pulse duration of laser and time-window of observation has to be controlled. The focus study dealing with the multiple spectra per sample and spectra training via PCA and SVM. The wavelength

range of 200 nm to 700 nm which is exactly the range wavelength detectable by the spectrometer was used.

### **1.5 Significance of Study**

The outcome of this study is important in improving the halal authentication techniques. Generally, there been efforts made to develop new application of existing analytical techniques for detection and quantification halal and non-halal of food systems. However, the methods still have their limitations. Thus, combination between LIBS and SVM will provide an automatic discrimination between halal and non-halal food.



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