

DETERMINATION OF LEAD CONTENT IN WHITENING CREAM USING LASER
INDUCED BREAKDOWN SPECTROSCOPY ANALYSIS

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DEDICATION

“I humbly thank Allah Almighty, the Merciful and the Beneficent, who gave me health, thoughts and co-operative people to enable me achieve this goal.”

To My Dearest Husband

Mohd Azahar b Che Abdullah

Thank you so much for your love and motivation

To My Beloved Parents,

W. Mohd Shukri b W. Ibrahim & Huzian bt Husin

Thank you very much for your love and supports

To My Lovely Siblings

W. Muhammad Syafiq b W. Mohd Shukri

W. Syazwan b W. Mohd Shukri

W. Nursyamimi bt W. Mohd Shukri

To My Respected and Beloved Supervisor

Prof. Dr. Noriah Ibrahim

Thank you very much for your attention and guidance

And all my friends...

Especially Nurul Hida and Saleha....

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ABSTRACT

Laser Induced Breakdown Spectroscopy (LIBS) technique was applied to determine the concentration of heavy metal such as Pb in five different whitening creams available from retail shop and open market in Johor, Malaysia. The whitening cream in the form of an emulsion was painted on the glass slide and then cryogenically frozen in refrigerator. The harden sample of whitening creams were irradiated with light Q-switch Nd:YAG laser at five different positions on the same sample. The spectral lines of six whitening creams were identified using National Institute of Standards and Technology (NIST) Database. Finally, Pb was detected in all the studied samples and their concentration in the range of 1.78 ppm – 4.64 ppm with limit of detection which is ~ 1 ppm. The LIBS results were validated by Inductive Couple Plasma Mass Spectroscopy (ICP-MS) technique with percentage difference in the range of 2.73% – 7.60%. The detection of Pb in the whitening creams is under permissible limit as set by Malaysian standard. However, the limitation is over as set by WHO which is 0.05 ppm for daily used. Thus, it is better to take precaution because continuously using creams may lead to accumulative toxicity in the body beyond the acceptable limit. Thus, the high sensitivity, portable and *in-situ* system allow LIBS to be an important technique in cosmetic industries.

ABSTRAK

Teknik spektroskopi leraian aruhan laser (LIBS) telah digunakan untuk menentukan kepekatan logam berat Pb di dalam enam jenis krim pemutih yang berbeza yang boleh didapati dari kedai runcit dan pasaraya di Malaysia. Krim pemutih adalah dalam bentuk emulsi telah dicat pada kepingan kaca dan kemudian dibekukan dalam peti sejuk. Sampel krim yang telah dikeraskan kemudian diradiasikan dengan cahaya laser suis-Q Nd : YAG di lima kedudukan yang berbeza pada tempat sasaran yang sama. Spektrum bagi enam jenis krim pemutih telah dikenal pasti dengan menggunakan data dari Institut Piawaian dan Teknologi Kebangsaan (NIST). Akhir sekali, Pb dikesan dalam semua sampel yang telah dikaji dengan kepekatan dalam lingkungan 1.78 pm – 4.64 ppm dengan had pengesanan ialah ~ 1 ppm. Keputusan LIBS telah disahkan oleh teknik plasma berganding aruhan spektrometri jisim (ICP-MS) dengan perbezaan peratus dalam lingkungan 2.73% – 7.60%. Pb yang dikesan dalam semua krim pemutih adalah di bawah had yang dibenarkan seperti yang telah ditetapkan oleh Piawaian Malaysia. Walau bagaimanapun, had ini adalah berlebihan seperti yang telah ditetapkan oleh WHO iaitu 0.05 ppm untuk kegunaan harian. Oleh itu, adalah lebih baik untuk mengambil langkah berjaga-jaga kerana penggunaan krim ini yang berterusan boleh menyebabkan pengumpulan toksik di dalam badan melebihi had yang boleh diterima. Oleh itu, kepekaan yang tinggi, mudah alih dan sistem *in-situ* membolehkan LIBS sebagai teknik penting dalam industri kosmetik.

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LIST OF ABBREVIATIONS/SYMBOLS

Hg	-	Mercury
Pb	-	Lead
Fe	-	Iron
Al	-	Aluminum
Cr	-	Chromium
Cd	-	Cadmium
Zn	-	Zinc
Co	-	Cobalt
Sn	-	Tin
Ag	-	Silver
g	-	Gram
cm	-	Centimeter
nm	-	Nanometer
mm	-	Millimeter
ml	-	Milliliter
ppm	-	Parts per million
mJ	-	MilliJoules
µg	-	Microgram
mg	-	Milligram
Hz	-	Hertz
UV	-	Ultraviolet
V	-	Volt

K	-	Kelvin
°C	-	Celsius
σ_b	-	Standard deviation of the background
S	-	Sensitivity
W/cm ²	-	Watt per square centimeter
Nd : YAG	-	Neodymium-doped yttrium aluminum garnet
LIBS	-	Laser Induced Breakdown Spectroscopy
LIPS	-	Laser Induced Plasma Spectroscopy
LSS	-	Laser Spark Spectroscopy
ICP-MS	-	Inductive Couple Plasma Mass Spectroscopy
ICP-AES	-	Inductive Coupled Plasma Atomic Emission Spectroscopy
AES	-	Atomic Emission Spectroscopy
FDA	-	US Food and Drug Administration
WHO	-	World Health Organization
NIST	-	National Institute of Standards and Technology

CHAPTER I

INTRODUCTION

1.1 Background of the Study

In recent years, the depletion of ozone layer makes the ultraviolet rays become stronger than ever. Excessive exposure to ultraviolet rays leads to human health, especially skin problem including sunburns, skin cancer, skin ageing and pigmentation (Martens et al., 1996). Whitening cream is widely used for skin preventive measure. Various kinds of whitening creams are commercially available either in the supermarket to the retail store with different range of prices. Such growing of whitening cream in the market without concerning the quality of the product may result the reverse effect. Generally preferable creams in the society, those offer faster and cheaper product, especially among the young ones to seeks of beautiful and glamorous, without concerning the side effect. High quality whitening cream products, normally expensive and under quality control and available in supermarket and hypermarket. However, may not be affordable by publics, especially the young ones. Thus they prefer to buy in retail shops and open market which are not provable and controlled by any health agency.

There are numerous researches conducted in analyzing the cosmetic products like whitening cream using different techniques (Meyer et al., 2010; WHO, 1995; AL-Saleh et al., 2009; Papanikolaou et al., 2005; Vahter et al., 2004; Amit et al., 2010; Gondal et al., 2010). The conventional techniques are normally expensive and require long procedure for sample preparation. Thus, alternative approaches are needed to overcome the drawback. In this attempt, a new technique is introduced to analyze heavy metal such as lead in whitening creams. So, Laser induced breakdown spectroscopy (LIBS) is conducted to detect lead in whitening creams. Several whitening creams are collected from retail shops and open market in this study. In order to validate the technique, LIBS data were compared with data obtained on the same whitening cream samples by the application of conventional Inductively Couple Plasma Mass Spectroscopy (ICP-MS).

1.2 Problem Statement

Cosmetic products such as whitening creams are widely available in Malaysia and consumers can easily buy them at any retail shops. Consumers are eager to look beautiful, even there are certain products that did not give any compositional information. So, many of them just use the cosmetic products without think the side effects. To provide consumers with safe cosmetics, it is necessary to control the amount of lead elements in such product. Some techniques are very expensive to get the standard to analyze the sample. Other technique may need a long procedure because various steps need to be followed. The aim of the work presented here is to introduce a technique that is suited for rapid in-situ analysis of lead in whitening cream. Thus, we proposed analysis lead in whitening cream by using laser induced breakdown spectroscopy technique.

1.3 Objectives of the Study

The objectives of this study are:

1. To analyse the lead elements present in whitening cream.
2. To calibrate the concentration of lead contamination in whitening cream.
3. To compare the results of LIBS analysis with conventional method.

1.4 Scope of the Study

Several whitening creams are collected from retail shops and open market in this study. The cream will be hardened using freezing method. A Q-switched Nd:YAG laser will be employed as a source of energy to form plasma and MAYA spectrometer was used to analyze the fluorescence of plasma intensity using the LIBS technique. In order to test the validity of our LIBS results, the standard technique like Inductive Couple Plasma Mass Spectroscopy (ICP-MS) was also applied.

1.5 Significance of the Study

It is important to determine lead content in whitening cream by using Laser Induced Breakdown Spectroscopy to help consumers aware of the danger and harmful lead elements it may contain. The knowledge of the lead elements in the whitening cream is also important to determine safety level, whether the concentration of lead is under the permissible limit.

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