# PRINTING INKS ANALYSIS AND DISCRIMINATION ABILITIES USING LASER INDUCED BREAKDOWN SPECTROSCOPY AND ENERGY DISPERSIVE X-RAY FLUORESCENCE

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*To my beloved parents and sister, thank you for the unconditional love and support given. Without whom none of my success would be possible.*

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# ABSTRACT

Establishing the authenticity of a questioned document remains one of the pertinent aspects in criminal as well as civil investigations. Because different manufacturers may use varying compositions of inorganic materials for producing inks, its quantitation may lead to the possibility of identifying the unique chemical fingerprint for each manufacturer. Hence, this present research that reported the use of in-house developed laser-induced breakdown spectroscopy (LIBS) and energy dispersive x-ray fluorescence spectrometry (EDXRF) to determine the inorganic composition of several different black printing inks, coupled with principal component analysis (PCA), acquires forensic significant. The samples analysed were from three types of printers *viz.* inkjet, laser, and photocopier (three different brands for each type) and one control blank white A4 paper. The optimum laser energy for enabling LIBS analysis was found at 900 mJ. While LIBS was found suitable for detecting several elements, especially those with low atomic numbers (Z<30), detection of higher atomic number (Z>30) elements was evidently suitable following the use of EDXRF. PCA-LIBS was found to sufficiently discriminate all the different printing inks based on qualitative elemental differences. Lower discriminative abilities were observed for PCA-EDXRF and PCA-LIBS-EDXRF combinations. Therefore, considering its time and cost effectiveness as well as requiring only minute amount of sample with no sample pre-treatment steps, combination of PCA-LIBS evaluated here, may prove useful for forensic questioned document practical caseworks.

# ABSTRAK

Pembuktian dokumen yang dipertikaikan ketulenannya merupakan salah satu aspek penting dalam siasatan jenayah dan juga siasatan sivil. Oleh sebab pengeluar dakwat yang berbeza menggunakan bahan bukan organik yang berbeza dalam menghasilkan dakwat, penentuan ini dapat membawa kepada kemungkinan bagi mengenal pasti pengenalan unik untuk setiap pengeluar. Oleh itu, kajian ini melaporkan tentang penggunaan teknik spektroskopi plasma laser (LIBS) dan komersial Pendaflur Serakan Tenaga Sinar-X (EDXRF) bersama-sama dengan analisis komponen utama (PCA) dalam menentukan komposisi bahan bukan organik daripada beberapa dakwat percetakan hitam bagi mendapatkan keketaraan forensik. Sampel dakwat percetakan hitam terdiri daripada tiga jenis pencetak iaitu inkjet, laser, dan mesin fotostat (tiga jenama yang berbeza bagi setiap jenis) serta satu sampel kawalan (kertas putih kosong bersaiz A4) yang telah melalui analisi LIBS. Tenaga laser optimum untuk menganalisis LIBS ialah 900 mJ. Didapati bahawa analisis LIBS adalah lebih sesuai bagi mengesan elemen yang bernombor atom rendah ($Z<30$), manakala EDXRF pula adalah lebih sesuai bagi nombor atom yang tinggi ($Z>30$). Penggunaan analisis komponen utama (PCA) untuk data yang diperolehi sama ada daripada LIBS atau komersial EDXRF dan juga gabungan antara dua set data dapat menilai kesahihan sampel. Hasil kajian menunjukkan bahawa penggunaan PCA dengan data LIBS dapat memberikan kebolehan diskriminatif atas perbezaan elemen kualitatif antara semua dakwat percetakan hitam. Diskriminatif yang rendah telah ditunjukkan oleh PCA-EDXRF dan kombinasi PCA-LIBS-EDXRF. Dengan mempertimbangkan masa dan keberkesanan kos serta keperluan jumlah minit sampel dengan tiada langkah pra-rawatan sampel, kombinasi LIBS dan PCA telah terbukti sangat berguna untuk pemeriksaan kesahihan dokumen dalam praktikal kes forensik.

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## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| AAS | - | Atomic Absorption Spectroscopy |
| CIJ | - | Continuous Inkjet System |
| CCD | - | Charge-Coupled Device |
| DA | - | Discriminant Analysis |
| DOD | - | Drop-On Demand |
| EDXRF | - | Energy Dispersive X-ray Fluorescence Spectrometry |
| FTIR | - | Fourier Transform Infared Spectroscopy |
| GC-MS | - | Gas Chromatography-Mass Spectrometry |
| HPLC | - | High Performance Liquid Chromatography |
| ICP-MS | - | Inductively Coupled Plasma Mass Spectrometry |
| IR | - | Infrared |
| LDMS | - | Laser Desorption/Ionization Mass Spectrometry |
| LIBS | - | Laser-Induced Breakdown Spectroscopy |
| LA-ICP-MS | - | Laser Ablation-Inductively Coupled Plasma Mass Spectrometry |
| MCR-ALS | - | Multivariate Curve Resolution Alternating Least Squares |
| PCA | - | Principal Component Analysis |
| PLS-DA | - | Partial Least Square Method Discriminant Analysis |
| Py-GC | - | Pyrolysis Gas Chromatography |
| QDE | - | Question Document Examination |
| R-A-IR | - | Reflection-Absorption Infrared Microscopy |
| SEM-EDX | - | Scanning Electron Microcopy with Energy-Dispersive X-ray Spectroscopy |
| SIMCA | - | Soft Independent Modeling of Class Analogy |
| SQA | - | Soil Quality Assessment |
| TLC | - | Thin Layer Chromatography |
| UV-VIS | - | Ultraviolet-Visible Spectroscopy |
| VSC | - | Video Spectral Comparator |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Writing has been defined as something that is written or printed for communicating information (Cambridge Advanced Learner's Dictionary, 2013). Dated as early as the Neolithic period, ideographic and early mnemonic symbols were used to convey messages (Powell, 2009). For facilitating the writing process, many devices have been crafted, dated to the oldest rigid tool without the use of ink pigment (Bellis, 2011), moving towards the more sophisticated printers (Dittmar, 2011). Printer was first invented by Johannes Gutenberg in 1436 using replaceable wooden or metal letters as printing press (Strother *et al.*, 2012) and since then the printing technology has evolved tremendously with the invention of inkjet printers, laser printers, 3-dimensional printers as well as photocopiers. Considering the importance of documents in daily life and since printers may prove to be one of the suitable means for document preparation, a volume of about 100 million printers sold throughout the world (Statistic Brain, 2014) appears to be within expectation.

The high degree of verisimilitude for forged documents has drastically increased due to the rapid improvement of computer hardware, software and high-quality printers (Warner and Adams, 2005), coupled with malicious intentions driven by multifactorial factors such as job demands, self-esteem, financial constrain, and pressure from other parties (Kennedy, 2012). Putting into perspective, fraudulent activities such as faking police and/or medical reports, wills, certificates, counterfeited bank notes, and letters may carry significant repercussions that may prove detrimental for societal well-beings. Given such importance, the ability for detecting forged documents requires continuous empirical studies, capitalising on the forefront of scientific endeavours for ensuring the admissibility of the evidence in the court of law. It is pertinent to indicate here that because the availability of forensic evidence can be limited, the use of less destructible scientific means for analysing a questioned document may prove necessary. Assigning the authenticity of a questioned document remains one of the related aspects in criminal as well as civil investigations, especially relating to its origin. It has to be indicated here that the different manufacturers for printing ink may use varying formulations in their preparation of inks, an aspect that can be of importance for forensic application.

## 1.2 Problem Statement

The use of inorganic materials in the ink formulations at varying amounts and constituents may lead to the possibility of providing the chemical fingerprint of an individual manufacturer. While destructive chemical methods such as Inductive Coupled Plasma Mass Spectrometer (ICPMS) and High-Performance Liquid Chromatography (HPLC) have been routinely used for ink analysis, this approach appears to be unfavourable for forensic use due to limited amount of forensic evidence. Hence, the use of less destructible methods such as Laser Induced Breakdown Spectroscopy (LIBS) coupled with principal components analysis (PCA) for providing chemical fingerprints of inorganic portions of ink, although limited in the literature, has been suggested (Hoehse *et al.*, 2012; Lennard *et al.*, 2015). While the use of commercial LIBS and energy dispersive x-ray fluorescence spectrometry (EDXRF) for investigating the authenticity of writing ink has been reported, similar approach for assigning authenticity for printed documents from different types of printers remains limited. Previous studies in this aspect pertained to conditions prevailing in temperate countries (Rožić *et al.*, 2005; Kula *et al.*, 2014) which may differ from conditions commonly observed in tropical countries like Malaysia. Therefore, generalising the behaviour of chemical decompositions observed by those researchers for Malaysian context can be erroneous. Hence, this present research that evaluated the application of PCA with data obtained from either an in-house developed LIBS or commercial EDXRF, as well as in-combination of these two methods, on printed materials from inkjet and laser printers, as well as photocopiers, merits forensic considerations.

## 1.3 Research Objectives

The objectives of this research included:

(1) To determine the elemental compositions of printing inks produced by inkjet and laser printers as well as photocopiers using an in-house developed LIBS and commercial EDXRF.

(2) To evaluate the individual discrimination abilities of LIBS and EDXRF when coupled with PCA for determining elemental compositions in printing ink samples.

(3) To evaluate the combined discrimination ability of LIBS and EDXRF when coupled with PCA for determining elemental compositions in printing ink samples.

## 1.4 Scope of Research

Standard white A4 papers (70 gsm) from a manufacturer were used in this research. Using used inkjet, laser, and photocopier printers from three different brands respectively, square boxes (measuring 0.25 cm x 0.13 cm each) printed with each genuine black ink were analysed for elemental compositions using an in-house developed LIBS as well as commercial EDXRF. The spectra produced from blank white A4 paper was used as the baseline data for indicating the presence of inorganic compounds in the inks. Using PCA, the compositions of inorganic compounds in the printing inks obtained from the in-house developed LIBS and commercial EDXRF, both individually as well as in-combination, were used as "chemical fingerprints" for ascertaining the origins.

## 1.5 Significance of Research

Because compositions of chemical compounds in printing ink is susceptible to environmental conditions such as temperature and humidity, and since specific studies focusing on this aspect in tropical countries like Malaysia have never been reported, this research that investigated the inorganic compounds of printing inks using an in-house developed LIBS and commercial EDXRF proves to be relevant. It is expected that the data gathered here would be of applied values in forensic investigations pertaining to the authenticity of questioned documents.

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