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DETERMINATION OF SELECTED HEAVY METAL CONCENTRATIONS IN A BLUSHER COSMETIC OF DIFFERENT COLOUR SHADES

by

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LIST OF ABBREVIATIONS AND SYMBOLS

^{o}C	Degree Celcius
µg/g	Microgram per gram
AAS	Atomic Absorption Spectroscopy
ACD	ASEAN Cosmetic Directive
As	Arsenic
ASEAN	Association of Southeast Asian Nations
BPOM	Badan Pengawas Obat dan Makanan
C	Concentration
Cd	Cadmium
CF-LIBS	Calibration Free-Laser Induced Breakdown Spectroscopy
Cr^{3+}	Chromium (III), Trivalent Chromium
Cr^{6+}	Chromium (VI), Hexavalent Chromium
Cr	Chromium
EC	European Community
EU	European Union
FAAS	Flame Atomic Absorption Spectroscopy
FDA	Food and Drug Administration
g	Gram
GFAAS	Graphite Flame Atomic Absorption Spectroscopy
H_2O	Water
H_2SO_4	Sulfuric Acid
HCl	Hydrochloric Acid
$HClO_4$	Perchloric Acid
Hg	Mercury
HMs	Heavy Metals
HNO_3	Nitric Acid
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
kg	Kilogram

LOD	Limits of Detection
LOQ	Limits of Quantification
M	Molar
m	Mass
mL	Millilitre
MLs	Maximum Limits
mg/L	Milligram per litre
mg/kg	Milligram per kilogram
Mn	Manganese
MOH	Ministry of Health
Ni	Nickel
No.	Number
NO_2	Nitrogen Dioxide
NPRA	National Pharmaceutical Regulatory Agency
Pb	Lead
$Pb(NO_3)_2$	Lead (II) Nitrate
ppb	Part per billion
ppm	Part per million
Sb	Antimony
SPSS	Statistical Package for the Social Sciences
USA	United State of America
US FDA	United State Food and Drug Administration
UV-Vis	Ultraviolet-visible
V	Volume
WHO	World Health Organization
XRF	X-ray Fluorescence Spectrometry
Zn	Zinc

PENENTUAN KEPEKATAN LOGAM BERAT TERPILIH DALAM KOSMETIK PEMERAH PIPI DENGAN PELBAGAI TON WARNA

ABSTRAK

Pemerah pipi, sebagai salah satu produk losmetik yang digunakan secara meluas oleh pengguna, memainkan peranan penting dalam potensi risiko kesihatan. Kajian ini bertujuan untuk meneliti kepekatan logam berat dalam kosmetik pemerah pipi dengan pelbagai kematan warna serta menilai sama ada keamatan warna mempengaruhi kandungan logam berat. Kajian ini dijalankan ke atas satu jenama pemerah pipi berbentuk pelet yang terdiri daripada enam tona keamatan warna berbeza, yang dibeli melalui platform dalam talian dengan jumlah jualan tertinggi. Sampel pemerah pipi dikategorikan mengikut keamatan warnanya bermula daripada sangat rendah (SA), rendah (SB), sederhana rendah (SC), sederhana tinggi (SD), tinggi (SE), dan sangat tinggi (SF). Kepekatan logam berat terpilih iaitu plumbum (Pb), kadmium (Cd), dan kromium (Cr) ditentukan menggunakan Spektroskopi Serapan Atom (AAS) selepas proses penghadaman asid. Kepekatan logam berat terpilih menunjukkan variasi nilai mengikut corak berikut: Cr (17.321 hingga 21.998 mg/kg) > Pb (12.329 hingga 17.820 mg/kg) > Cd (4.838 hingga 6.011 mg/kg), kecuali sampel A (SA) yang menunjukkan corak Pb (27.250 mg/kg) > Cr (23.430 mg/kg) > Cd (5.795 mg/kg). Keputusan kajian menunjukkan bahawa kepekatan Pb dan Cd dalam beberapa sampel melebihi had yang dibenarkan oleh Arahan Kosmetik ASEAN (ACD) dan Agensi Regulatori Farmaseutikal Kebangsaan (NPRA) Malaysia, manakala Cr menunjukkan kepekatan yang relatif tinggi walaupun tiada had peraturan khusus ditetapkan. Selain itu, analisis statistik seperti korelasi pangkat Spearman, ujian Kruskal-Wallis, dan ujian Mann-Whitney digunakan untuk menilai hubungan dan perbezaan kepekatan logam antara sampel. Keputusan statistik menunjukkan variasi kepekatan logam berat dalam sampel pemerah pipi, namun tiada hubungan yang konsisten diperhatikan antara keamatan warna dan

kepekatan logam berat. Oleh itu, pencemaran logam berat dalam kosmetik pemerah pipi adalah berbeza antara produk dan tidak dipengaruhi semata-mata oleh keamatan warna. Hasil kajian ini boleh dijadikan asas untuk kajian lanjutan berkaitan kawalan kualiti pembuatan kosmetik dan proses pemantauan peraturan.

DETERMINATION OF SELECTED HEAVY METAL CONCENTRATIONS IN A BLUSHER COSMETIC OF DIFFERENT COLOUR SHADES

ABSTRACT

Blusher, as one of the cosmetic products widely used by consumers, playing a pivotal role in potential health risks. This study endeavours to delve into the concentration of heavy metals in blusher cosmetics of various colour intensity, and to evaluate whether colour intensity influences heavy metal content. A study was carried out on one brand of blush pellet consisted of 6 different tones of colour intensities which purchased from online platform with the highest sold numbers. The blush samples were categorised by its colour intensities starting from very low (SA), low (SB), medium low (SC), medium high (SD), high (SE), and very high (SF) colour intensity. The concentrations of selected heavy metals namely lead (Pb), cadmium (Cd), and chromium (Cr) were determined by using Atomic Absorption Spectroscopy (AAS) following acid digestion. The concentration of selected heavy metals varied according to the following trend: Cr (17.321 to 21.998 mg/kg) > Pb (12.329 to 17.820 mg/kg) > Cd (4.838 to 6.011 mg/kg), except for sample A (SA) as it follows trend: Pb (27.250 mg/kg) > Cr (23.430 mg/kg) > Cd (5.795 mg/kg). The results showed that Pb and Cd exceeded permissible limits set by ASEAN Cosmetic Directive (ACD) and National Pharmaceutical Regulatory Agency (NPRA) of Malaysia in selected samples, while Cr exhibited comparatively higher concentrations despite the absence of a specified regulatory limit. Furthermore, statistical analyses like Spearman's rank correlation, Kruskal-Wallis, and Mann-Whitney tests were employed to evaluate relationship and differences in metal concentrations among samples. The statistical results indicated variability in heavy metal concentrations across blush samples, with no consistent association was observed between colour intensity and heavy metal concentration, thus, the heavy metals contamination in blush cosmetics varies among products

and is not solely influenced by colour intensity. The results of this study can be a basis for future studies on cosmetic manufacturing quality control and regulatory monitoring process.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Cosmetics are personal hygiene products in common use for people across all demographics, e.g., lipsticks, foundations, eyeshadows, and blushers (Abdul Latif et al., 2024). These personal care products are used by both genders and a wide range of age groups to improve appearance and instil confidence. As these products are applied directly to the skin on a long-term basis frequently, their chemical safety is seen to be important in public health especially for facial cosmetics due to their direct and prolonged contact with face, which is sensitive skin areas.

Amongst these, products of blusher are used on facial skin to provide colour and brilliance to the cheeks. It is to provide colour and a healthy glow. Blush preparations are specifically noted as a category of colour cosmetics regularly analysed for contaminants (Kicińska & Kowalczyk, 2025). Blusher cosmetics usually contain pigments, binders, fillers, and preservatives. The colour shades in blushers, usually range from light to darker shades, are determined by the type and concentration of pigments present in formulation. The differences in pigment type, formulation composition, and pigment concentration helped in achieving variation in colour shade. Both organic and inorganic pigments used in blush products, with mineral-based pigments being widely favoured because of their cost-effectiveness and colour stability.

However, there are association of trace heavy metal impurities from these mineral-derived pigments, whereas concerns regarding the chemical safety of blush cosmetics are raised. The pigments and colorants used in cosmetics may contain trace levels of heavy metals

such as lead (Pb), cadmium (Cd), nickel (Ni), and chromium (Cr) from contaminated raw materials, natural mineral sources or manufacturing processes. These metals can enter cosmetics unknowingly during extraction, formulation, or even packaging of the pigments.

Generally, the manufacturing process of blush cosmetics involves several stages. Heavy metal contamination tends to be introduced at multiple points throughout the process of raw material selection, pigment blending, milling, binding, and packaging. Naturally occurring minerals used as fillers or pigments are where the impurities might be originated from, along with inadequate purification during pigment processing, cross-contamination from shared manufacturing equipment, and insufficient measures of quality control. The result of inconsistency of heavy metal content across cosmetic products with similar intended use may due to variability in manufacturing standards, particularly among different brands and production scales.

In recent years, consumer access to cosmetic products has significantly increased as the rapid expansion of online retail platforms. In addition, these products especially blushers are sold at relatively low prices. The reason they are highly accessible and widely purchased are because of aggressive marketing through social media and e-commerce platforms. Apart from that, lower-priced cosmetics may be produced with reduced regulatory oversight or cost-driven compromises in manufacturing control and raw material quality, as well as to make them affordable and convenience for consumers benefit. Thus, concerns regarding the potential presence of hazardous contaminants have raised, including heavy metals, in such products.

Crucially, the concern is particularly pronounced for cosmetic products, such as blush, that are sold online or originate from foreign/nonlocal markets, as manufacturers in countries with less restrictive regulations may introduce products that exceed safety limits (Rahil S. Y. et al., 2022). Foreign or nonlocal blush, particularly those sold online and nearby markets, are

now widely sought after by Malaysians due to their affordability and availability of many options. These cosmetic products have become increasingly available through online shopping platforms in recent years and often low-priced and look attractive to buyers who may be uninformed about possible health complications.

Such foreign blush may not necessarily comply with local safety regulations set by the National Pharmaceutical Regulatory Agency (NPRA) or the Ministry of Health (MOH) at all times. It is due to most of the unbranded blushers are manufactured without strict quality control and disclosure of the source of their raw materials, hence there is a chance of heavy metal contamination. Previous studies have confirmed the presence of heavy metals in blush, sometimes exceeding regulatory limits (Sylvia et al., 2021). Besides, different colour shades of the same line of unbranded products can be made from pigments originating from different mineral sources, thus giving way to differences in metal concentrations. Still, scientific studies addressing the content of heavy metals across different colour shaded of unbranded blusher cosmetics remain scarce. Hence, it is necessary to assess the safety of such foreign blusher items in terms of heavy metal contamination.

Heavy metals are not biodegradable and have the potential to get accumulated in the human body through dermal penetration or incidental ingestion. They are toxic in trace amounts as well. Heavy metals such as Lead (Pb), cadmium (Cd), and chromium (Cr) are particular concern in cosmetic products as they tend to have high toxicity and cumulative health effects. Lead exposure can cause neurotoxicity or neurological dysfunction, anaemia, and kidney damage, as well as impaired cognitive development and reproductive health effects. Meanwhile, Cadmium exposure can lead to renal disease and bone disease, as it is known for its bioaccumulative nature that has potential in causing skeletal and kidney damage following chronic exposure.

For chromium, this heavy metal depends on its chemical form in addressing adverse health effects. For example, particularly hexavalent chromium (Cr^{6+}), is a confirmed carcinogen. These can pose serious health threats due to their potential to accumulate in the human body over long periods (Abdul Latif et al., 2024). Additionally, chromium may cause allergic contact dermatitis and skin irritation after prolonged dermal contact. Although they are not intentionally used or added through cosmetics formulation and manufacturing process; even small amounts of impurities or contaminants present might build up in the human body to pose a potential risk to consumers, particularly through repeatedly used products over long periods or incidental ingestion (Sh. Mahmood et al., 2022). Hence, monitoring of heavy metal contamination in cosmetics is necessary to provide protection to consumers.

ASEAN Cosmetic Directive (ACD) and National Pharmaceutical Regulatory Agency (NPRA), Malaysia are the responsible regulatory bosies to establish permissible limits for heavy metals in cosmetic products. By complying these guidelines, unavoidable trace contamination can be controlled and that heavy metal levels are ensured to remain within acceptable safety margins. Besides, consumer awareness plays a crucial role in mitigating potential health risks associated with cosmetic use apart from regulatory enforcement. Most of the consumers, especially those purchasing cosmetic products online and at lower cost, are basically unaware of the possibility of heavy metal contamination.

This study aims to ascertain and quantify the level of selected heavy metals (Pb, Cd, Cr) in the blusher product with different colour shades that are highly sold in online platform in the region of Malaysia by using Atomic Absorption Spectroscopy (AAS). The study focuses on one brand of blush palette with various colour shades, which are more likely to contain contaminants based on pigment intensity. Besides instrumental analysis, statistical analyses is conducted to support the findings. This will then be compared with established safety

guidelines by ACD and NPRA for their safety assessment. Thus, these findings of this study are expected to contribute valuable data to cosmetic safety assessment, regulatory monitoring, and consumer awareness, as well as provide forensic insight into product quality and composition.

1.2 Problem Statement

The global cosmetic market has increased with numerous foreign or nonlocal brands which are mostly unbranded or no-named brands penetrating the Malaysian market, especially in online shopping market without proper quality evaluation. Unbranded blushers, which are commonly sold online and often attract consumers due to their lower prices, are typically not safety-tested and may contain hazardous metal content. The potential presence of heavy metals in these products is of concern from a health point of view because long-term exposure to lead, cadmium, and chromium can lead to serious health issues. Although there are regulations that control cosmetic safety, imported cosmetics may not always be compliant with these standards.

Heavy metals have been previously identified in lipsticks, eyeshadows, and foundations, but few data are available for blusher products, especially in a single brand of different colour shades that is highly sold online in Malaysia. The colour pigments used may influence the level of contamination (Hameed Mahdi & Ali Salman, 2024). Comparative analyses of metal content between different colour shades of the same unbranded blusher are limited. Such analyses raise concerns regarding product safety and whether the metal content complies with international safety standards. The lack of these data points to the need for systematic studies on heavy metal presence in these cosmetics. Detection and quantification of heavy metals in various colour shades of the same unbranded blusher are necessary to determine whether the concentrations of these metals vary across shades and remain within permissible safety limits. Therefore, the objective of this study is to detect and compare the

concentrations of selected heavy metals in different colour shades of blusher cosmetic that is highly sold online to see the effect of colour shades intensity onto heavy metal concentrations and their compliances to safety regulations.

1.3 Research Questions

- What are the concentrations of selected heavy metals (Pb, Cd, Cr) in different colour shades of unbranded blusher cosmetic?
- Do different colour shades of the same unbranded blusher product exhibit variation in Pb, Cd, and Cr concentrations and do concentration levels affected by pigment intensity?
- Are the detected concentrations within permissible limits specified by international cosmetic safety guidelines (ACD & NPRA)?

1.4 Significance of The Study

This research delves into insights on the safety of unbranded or unrecognised brand cosmetic products, which are a growing segment of online retail markets. The study would identify the presence and variation of heavy metals among different shades; thus, it will add to consumer awareness and potential risks in purchasing unregulated products. The outcome would also assist regulatory agencies in taking up the task of strengthening product monitoring and enforcing safety standards. The findings will help closer the knowledge gap for heavy-metal pollution in different shades of colour of unbranded blusher and provides information on concentration levels of Pb, Cd, and Cr and its correlation with pigment intensity. Moreover, this study enables compliance assessment with international safety limits and helps bring about consumer awareness of health risks due to improper use of unsafe cosmetics.

The current study represents an academic contribution to forensic toxicology and analytical chemistry by showing AAS as a reliable technique in the detection of trace metals present in

complex cosmetic matrices. It applies in forensic where chemical trace analysis of cosmetics may serve as supporting evidence in cases involving poisoning, counterfeit products, or consumer fraud. The comparison of shades will provide information on how the possibly different composition of pigments may result in differences in contamination levels.

1.5 Objectives

1.5.1 General Objective

To detect and compare the concentrations of selected heavy metals in different colour shades of blusher cosmetic that is highly sold online using Atomic Absorption Spectroscopy (AAS).

1.5.2 Specific Objectives

1. To determine the concentration of lead (Pb), cadmium (Cd), and chromium (Cr) in each colour shade using AAS.
2. To compare the concentration of these metals between different colour shades and evaluate whether pigment intensity affects concentration levels.
3. To assess whether the detected concentrations comply with established permissible limits for cosmetic safety (ACD & NPRA).

1.6 Study Hypothesis

1.6.1 Null Hypothesis (H_o)

There is no significant difference in blusher cosmetics between the colour shades intensity and the level of heavy metal concentrations.

1.6.2 Alternative Hypothesis (H_1)

There is a significant difference in blusher cosmetics between the colour shades intensity and the level of heavy metal concentrations.

CHAPTER 2

LITERATURE REVIEW

2.1 Heavy Metal Contamination in Cosmetic Products

Cosmetics are defined as preparations or materials used externally on the human body for purposes such as cleansing, beautifying, promoting attractiveness, or changing appearance (Permatasari et al., 2024). Cosmetics products intended to be rubbed, poured, sprinkled, or applied externally to cleanse, beautify, or change appearance, are widely used across global markets (Attard Tamara et al., 2022). According to Federal Food, Drug, and Cosmetic Act, cosmetics are known as preparations or materials intended to be rubbed, poured, sprinkled, sprayed, or applied externally to the human body for cleansing, beautifying, promoting attractiveness, or changing appearance, excluding claims as a medicine (Permatasari et al., 2024).

These products are considered a daily human necessity and are widely used by people across all age groups and demographics (Permatasari et al., 2024). They have become staples in daily beauty routines globally, used widely by people of all ages (Abed Mayyadah S. et al., 2024). This widespread and prolonged use creates a significant pathway for consumer exposure to toxic substances, particularly heavy metals (HMs) (Amer Sarah K. et al., 2024).

Many of these beauty products, such as blush, lipstick, eye shadow, and powder, may contain hazardous materials like heavy metals (Ari Wardani et al., 2020). The widespread use of these products exposes consumers to potential health hazards due to the inclusion of heavy metals (HMs) (Abed Mayyadah S. et al., 2024). Heavy metals can be introduced into cosmetics inadvertently from raw materials, pigments, or metal-coated equipment used during manufacturing (Permatasari et al., 2024). The long-term use of cosmetics containing toxic

elements can lead to systemic absorption (Amer Sarah K. et al., 2024). Continuous exposure to heavy metals can result in chronic toxic diseases and pathological changes in organs such as the cardiovascular system, kidneys, bones, and liver (El Sharaa I. A. F. et al., 2025).

Heavy metals are elements generally defined by a density greater than 4.5 g/cm³ and an atomic weight higher than that of calcium (40.04) (Abdelmonem et al., 2025). Heavy metals are regarded as substances that present significant health risks (Amer Sarah K. et al., 2024). The presence of these metals often results from using raw materials containing natural impurities, metallic devices in the manufacturing process, or sometimes intentionally as coloring agents or preservatives (Arshad Hamna et al., 2020). Among the most common and toxic heavy metals found in cosmetics are lead (Pb), mercury (Hg), cadmium (Cd), and arsenic (As) (Abdelmonem et al., 2025).

Heavy metals pose significant risks because they are non-biodegradable and accumulate in the body over time, potentially leading to chronic toxic diseases, neurological, reproductive, and cardiovascular disorders, cancer, and, in fatal cases, death (Irfan Muhammad et al., 2022). Chronic exposure to HMs can lead to severe health issues, including neurological, reproductive, and cardiovascular disorders, as well as cancer (Massadeh A. M. et al., 2017).

The presence of these toxic elements in cosmetics, including powder-based products like blusher, often stems from impurities in raw materials, intentional addition for coloring, or contamination during the manufacturing process (Abed Mayyadah S. et al., 2024). The concern is amplified for non-local or imported products, as they may bypass stringent quality control, leading to inconsistencies in heavy metal concentrations compared to locally regulated goods (Arshad Hamna et al., 2020).

Lead (Pb) is recognized as one of the most studied and toxic heavy metals found in cosmetic formulations (Sh. Mahmood et al., 2022). For example, coloring materials such as iron dioxide may contain trace amounts of Pb (Permatasari et al., 2024), and lead compounds like lead carbonate or lead sulfate may be intentionally used as dyes or pigments (Sylvia et al., 2021). Lead, in particular, can enter the body by penetrating skin membranes, as it is soluble in oil and fat (Ari Wardani et al., 2020). Accumulation of lead in the body is highly dangerous, leading to potential effects like anemia, brain damage, disruption of reproductive and endocrine systems, and even carcinogenic effects in high doses (Attard Tamara et al., 2022).

2.2 Regulatory Thresholds for Target Heavy Metals

The safety of cosmetic products, which are widely used daily, is governed by stringent international regulations to limit the presence of heavy metals (El Sharaa I. A. F. et al., 2025). Entities introducing cosmetic products to the market are responsible for ensuring the products' chemical composition complies with relevant standards (Kicińska & Kowalczyk, 2025). The pervasive nature of heavy metal contamination necessitates that cosmetics meet maximum contamination requirements (Permatasari et al., 2024).

To ensure consumer safety, cosmetic preparations must meet maximum heavy metal contamination requirements set by regulatory agencies (Permatasari et al., 2024). The most commonly referenced heavy metal contamination limit in the sources is for Lead (Pb). According to Indonesia's Regulation of the Head of the Drug and Food Supervisory Agency (BPOM) Number 17 of 2014, amending a 2011 regulation, the maximum limit for lead contamination in cosmetics is no more than 20 mg/kg or 20 ppm (Ari Wardani et al., 2020).

Globally, other jurisdictions have set similar or stricter limits whereas the World Health Organization (WHO) sets a maximum limit of 10.0 mg/kg (10 ppm) for Pb (El Sharaa I. A. F.

et al., 2025), while the U.S. Food and Drug Administration (FDA) has established a maximum permissible limit of 10 ppm for lead in color additives for the manufacture of cosmetics (Attard Tamara et al., 2022). Health Canada's guideline for heavy metal impurities recommends a limit of 10 mg/kg for Pb (Barghash Samia S. et al., 2017). These regulatory limits are crucial because numerous studies, many focusing on products sold in international or "nonlocal" markets, have found heavy metals either within the requirements or exceeding them (Permatasari et al., 2024). For instance, a study of eye shadow preparations sold in an Indonesian market found one product contained lead at 127.356 ppm, which surpassed the BPOM requirements (Ari Wardani et al., 2020).

Due to the toxicity risks posed by heavy metals, international regulatory bodies and organizations have established strict guidelines setting maximum limits (MLs) for these contaminants in cosmetic products (ASEAN Cosmetic Directive, 2022). These limits are crucial when evaluating non-local products, as standards can vary geographically, leading to discrepancies in acceptable contamination levels (Attard Tamara et, 2022).

The European Union (EU) has strict regulations, setting zero tolerance limits for all heavy metals (Irfan Muhammad et al., 2022) and prohibiting substances like Chromium (Cr), Nickel (Ni), Antimony (Sb), and Mercury (Hg) and their compounds (Kicińska & Kowalczyk, 2025). However, the EU also lists heavy metals that can be used at a maximum concentration in cosmetic products (Papadopoulos et al., 2022). The EU regulation (EC) No. 1223/2009, which governs the manufacture of cosmetics, has been in force since 2013 (Kicińska & Kowalczyk, 2025).

Regulatory limits set by various global bodies for lead (Pb) and cadmium (Cd) are critical benchmarks for cosmetic safety such as World Health Organization (WHO), United State Food and Drug Administration (US FDA), Health Canada, and Indonesia's Food and Drug

Supervisory Agency (BPOM). According to WHO, the maximum permissible limits for toxic metals in cosmetics are recommended to not exceed 10.0 mg/kg (10 ppm) for Pb, and Cd should not exceed 0.3 mg/kg (0.3 ppm) (Abed Mayyadah S. et al., 2024). Next, US FDA sets the maximum allowable level for lead contamination in cosmetics at 20 mg/kg (20 ppm) (Attard Tamara et a, 2022). For Pb used in color additives for cosmetics, the limit is stricter at 10 ppm (Abed Mayyadah S. et al., 2024). The FDA allows mercury content up to 1 ppm (Abed Mayyadah S. et al., 2024).

Furthermore, a maximum limit of 10 mg/kg (10ppm) for Pb impurities and 3.0 mg/kg (3.0 ppm) for Cd impurities in cosmetics are recommended by Health Canada (Barghash Samia S. et al., 2017). Meanwhile, under regional context of Indonesia (BPOM), Regulation Number 17 of 2014 has set the maximum limit for Pb contamination in cosmetics at no more than 20 mg/kg (20ppm), and the safe limit for Cd contamination is set at 5 mg/kg (5 ppm) (Ari WardaSylvia et al., 2021). The wide variation in standards, particularly between the strict zero tolerance approach of the EU and the impurity limits set by other bodies like the FDA and BPOM, can lead to confusion and highlights the need for consistent, continuous monitoring, especially for products sold in international markets (Attard Tamara et al., 2022).

Global regulatory bodies establish maximum allowable limits (MLs) for toxic metals in cosmetics to mitigate health risks, especially since dermal exposure to heavy metals can cause long-term adverse health effects (Irfan Muhammad et al., 2022). These limits are critical for assessing the safety of non-local blusher samples that are sold online or imported to local consumers. The USFDA and Health Canada are stricter in stipulating Pb limits as an impurity than BPOM's Head Regulation No. 17 of 2014, as the exposure of Pb is highly concerning due to its systemic toxicity, in which may affect the repsroductive and nervous systems (Abed Mayyadah S. et al., 2024). Cd impurity was set in low values by regulatory bodies because of

its prolonged exposure can lead to accumulation in the kidneys and damage and potentially resulting in brittle bones even in low concentration levels (Sylvia et al., 2021). Additionally, arsenic (AS) and mercury (Hg) are limited and restricted to 3 mg/kg (3 ppm) and 1 mg/kg (1 ppm), respectively, as an impurity in cosmetics (Abed Mayyadah S. et al., 2024).

Maximum allowable limits (MLs) for chromium (Cr) in cosmetic products, including blush, are typically set to control unavoidable metal impurities, as Cr compounds are sometimes prohibited entirely by regulatory bodies like the European Union (EU) (Abed Mayyadah S. et al., 2024). Regulatory standards vary significantly in which the World Health Organization (WHO) sets a strict permissible limit of 0.5 mg/kg (Attard Tamara & Attard Everaldo, 2022), while Health Canada recommends a limit of 10 mg/kg (Abed Mayyadah S. et al., 2024), and the US FDA, which has no specific limit for Cr in cosmetics, restricts it as an impurity in approved colour additives to 50 mg/kg (Ahmed et al., 2024).

Measured concentrations of Cr in blush samples show a wide range whereas one registered blush sample was found to contain 1.8 mg/kg of Cr, a level below thresholds referenced for Nigeria (9.81 µg/g) and the USA (15.09 µg/g) (Sylvia et al., 2021), but another study indicated that blushes generally had the highest mean Cr concentration (110 mg/kg) among various cosmetic products tested, with the highest single blush sample concentration reaching 416 mg/kg (Kicińska & Kowalczyk, 2025). When analysing a blush sample with AAS, a detected Pb content of 0.0599 ppm was considered safe, as it was significantly below the 20 ppm contamination threshold set by the Head of BPOM Regulation No. 17 of 2014 (Ari Wardani et al., 2020). However, research consistently shows that heavy metals like Pb, Cd, Cr, Ni, Cu, and Zn are routinely detected in cosmetic products obtained from international markets (Massadeh A. M. et al., 2017).

2.3 Documented Heavy Metal Levels and Contamination in Blusher Samples

Studies focusing on face makeup, such as foundation, face powder, and blusher, confirm that these products frequently contain trace amounts of toxic metals (El Sharaa I. A. F. et al., 2025). For powder cosmetics like blushes, there is a chance that particles, particularly iron oxide, may become airborne during application, potentially causing respiratory irritation, though the risk of direct iron oxide toxicity is typically considered low (Ahmed et al., 2024).

Blusher, or "blush on," is a form of facial cosmetic categorized under face make-up, similar to face powder and foundation (Zakaria et al., 2015). Studies examining face cosmetics, particularly those sold in foreign or non-local markets (e.g., online or imported brands), have revealed varying levels of heavy metal contamination (Massadeh A. M. et al., 2017). Research specifically examining blusher ("blush on") and comparable face powder products has demonstrated variability in HMs contamination, often highlighting the risks associated with non-locally sourced products.

One study analyzing various cosmetic samples, including blusher, showed that the mean concentration of Lead (Pb) in blusher was 0.49 ppm (0.13 mg/kg) and Cadmium (Cd) was 2.18 ppm (0.05 mg/kg) (Rahil S. Y. et al., 2022). This particular study, which investigated face cosmetics in Benghazi markets, Libya, also found mean concentrations of Chromium (Cr) at 0.35 ppm (0.13 mg/kg), Manganese (Mn) at 0.27 ppm (0.01 mg/kg), and Zinc (Zn) at 0.71 ppm (0.01 mg/kg) in blusher samples (El Sharaa I. A. F. et al., 2025). However, findings can fluctuate greatly depending on the product brand and origin. In an investigation of five blush-on samples sold online, subsequent quantitative analysis using AAS found that the majority of samples did not contain detectable levels of Pb or Cd (Sylvia et al., 2021). Only one sample (VV brand) in that study contained Cr at a level of 1.80 mg/kg (Sylvia et al., 2021).

This observed Cr concentration was below established thresholds, such as 9.81 µg/g (Nigeria) and 15.09 µg/g (USA) (Sylvia et al., 2021). Conversely, earlier related research concentrating solely on chromium in blush-on products found Cr detectable in all tested samples, with reported concentrations ranging from 6.74 ppm (0.02 µg/g) to 28.95 ppm (0.19 µg/g) (Sylvia et al., 2021). In a broad survey of cosmetics, face blushes exhibited a large range of contamination, with Chromium concentrations ranging from 7.18 mg/kg to 416.29 mg/kg and Nickel ranging from 3.79 mg/kg to 95.64 mg/kg (Kicińska & Kowalczyk, 2025). These variations underscore the risk associated with non-local cosmetic sourcing, as manufacturers' adherence to safety limits is often inconsistent, necessitating robust detection methods like AAS (Arshad Hamna et al., 2020).

For instance, a study specifically analyzed the heavy metal content of various cosmetic products, including foundation, blusher, and face powder, sold in Benghazi markets, Libya, using Atomic Absorption Spectrophotometer (AAS) (El Sharaa I. A. F. et al., 2025). Research assessing facial blushes in non-local contexts reported concentration ranges for several metals, including Lead (Pb) (0.29–2.44 mg/kg), Cadmium (Cd) (0.16–1.50 mg/kg), and Chromium (Cr) (7.18–416.29 mg/kg) (Kicińska & Kowalczyk, 2025).

Additionally, heavy metals such as Pb, Cd, Cr, Copper (Cu), Nickel (Ni), and Zinc (Zn) were detected in cosmetic products like blusher, mascara, lipstick, eyeshadow, and face powder sampled from Jordanian, Sudanese, and Syrian markets using Flame Atomic Absorption Spectrophotometry (FAAS) (Ahmed et al., 2024). Although some studies focusing on cosmetic powders may show lower concentrations of HMs compared to other categories like lipsticks (Abed Mayyadah S. et al., 2024), monitoring these products is essential due to their daily application and potential for long-term dermal exposure (Ahmed et al., 2024).

2.4 Analytical Techniques Comparison

The detection of heavy metals in cosmetics, particularly trace amounts, requires highly sensitive and validated analytical methods (Permatasari et al., 2024). The analytical methods used to detect heavy metals are Atomic Absorption Spectrophotometry (AAS), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES), X-ray Fluorescence Spectrometry, and UV-Vis Spectrophotometry. Atomic Absorption Spectrophotometry (AAS), including Flame AAS (FAAS) and Graphite Furnace AAS (GFAAS), is widely applied for determining metal elements, even those contained in solutions at very small concentrations, and is recognized as one of the most sensitive techniques (Papadopoulos et al., 2022).

This is a primary technique used for determining metal elements, even at very small concentrations in solution (Sh. Mahmood et al., 2022). Flame AAS (FAAS) and Graphite Furnace AAS (GFAAS) are common variants (Papadopoulos et al., 2022). Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is the highly sensitive method and is used for simultaneous multi-element analysis (Rahil S. Y. et al., 2022).

Meanwhile, Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) is also widely applied for determining multiple elements (e.g., Cr, Fe, Mn, Ni, and Zn) after sample digestion (Kicińska & Kowalczyk, 2025). Moreover, X-ray Fluorescence Spectrometry (XRF) is mentioned in a study as a technique used for the analytical detection of heavy metals in cosmetics too (Abed Mayyadah S. et al., 2024). Furthermore, UV-Vis Spectrophotometry can be utilized as an alternative method when AAS instruments are unavailable (Ari Wardani et al., 2020). Regardless of the spectroscopic method used, it must be validated for parameters such as linearity, accuracy, precision, limit of detection, and limit of quantitation (Ari Wardani et al., 2020).

AAS is widely applied for metal analysis (Permatasari et al., 2024). It has evolved as a standard procedure due to the weaknesses in accuracy and sensitivity of classical test methods like quantitative tests with precipitation techniques (Permatasari et al., 2024). UV-Vis Spectrophotometry method is sometimes used as an alternative when specialized AAS instruments are unavailable (Ari Wardani et al., 2020). However, it requires a complexing agent, such as dithizone, to form a colored complex (e.g., Pb-ditizonate) that absorbs radiation in the UV-Vis range (Ari Wardani et al., 2020). The UV-Vis method must be thoroughly validated, including parameters such as linearity and precision, to ensure reliability (Ari Wardani et al., 2020).

For ICP-MS/ICP-OES, these methods are generally considered highly sensitive techniques that have evolved alongside AAS (Permatasari et al., 2024). ICP-MS offers multi-element analysis and is often used in modern practice, especially when combined with microwave-assisted digestion, which provides very low detection limits (Sh. Mahmood et al., 2022). AAS is still broadly utilized, often serving as the practical method when advanced instruments like ICP-MS are limited in availability (Ari Wardani et al., 2020).

Atomic Absorption Spectrophotometry (AAS) is a reliable and frequently used analytical technique for determining metal elements in solutions, especially at very small concentrations, and is considered a "gold standard technique" (Rahil S. Y. et al., 2022). The method operates on the principle that atoms, usually in the ground state, absorb radiant energy at specific wavelengths (Jamali Saifullah et al., 2022). The extent of absorbed energy is directly proportional to the concentration of the element present in the sample solution (Permatasari et al., 2024).

2.5 Atomic Absorption Spectroscopy (AAS) Methodology and Instrumentation

AAS is one of the main techniques used for the analytical detection of heavy metals (Sh. Mahmood et al., 2022). It is employed for the determination of metal elements contained in solutions, often at very small concentrations (Ari Wardani et al., 2020). The principle of AAS is based on measuring the absorption of radiation by neutral ground state atoms of a specific element (Jamali Saifullah et al., 2022).

These methods, specifically Flame AAS (FAAS) and Graphite Furnace AAS (GFAAS), are advantageous due to their speed, good repeatability, and high sensitivity (Papadopoulos et al., 2022). AAS is generally tolerant of complex matrices (Permatasari et al., 2024). In specific studies, AAS technique confirmed results from other advanced methods like CF-LIBS with an inaccuracy of about 20% (Jamali Saifullah et al., 2021).

AAS is considered a "gold standard technique" based on the principle that atoms absorb light at unique wavelengths, and the amount of absorbed light is directly proportional to the concentration of the absorbing atoms (Abdelmonem et al., 2025). AAS offers the advantages of being low-cost, simple, portable, and having high accuracy (Abdelmonem et al., 2025). AAS techniques, including FAAS and GFAAS, are capable of measuring metal concentrations typically ranging from parts per million (ppm) down to parts per billion (ppb) (Ahmed et al., 2024). For example, specific parameters for Lead (Pb) analysis using flame AAS often utilize a lead cathode lamp at a wavelength of 283.3 nm (Permatasari et al., 2024). A linearity range for Pb determination using FAAS has been reported at 0.021–20 g/cm³ at a wavelength of 217.0 nm (Świerczek et al., 2019). The preparation of calibration standard curves is essential for quantifying heavy metal levels in samples using AAS (Selvaraju Anusia et al., 2020)

AAS utilizes different atomizer types which are Flame AAS (FAAS) and Graphite Furnace AAS (GFAAS). The variant of Flame AAS (FAAS) is often applied for determining metals such as Pb, Cd, Cr, Mn, and Zn in cosmetic products (Rahil S. Y. et al., 2022). The operating conditions are element-specific; for instance, Pb analysis via FAAS commonly uses a wavelength of 217.00 nm (Świerczek et al., 2019), Cd uses 228.00 nm and Cr uses 357.90 nm (El Sharaa I. A. F. et al., 2025). FAAS is capable of quantifying metal concentrations ranging from ppm to ppb levels (Ari Wardani et al., 2020). In other hands, Graphite Furnace AAS (GFAAS) is known as highly sensitive technique which is appropriate for elements like As and Pb (ASEAN Cosmetic Directive, 2022). GFAAS methods have demonstrated the ability to meet stringent maximum limit requirements set by international regulations (e.g., 5 ppm for As and 20 ppm for Pb) (ASEAN Cosmetic Directive, 2022).

For accurate quantification using AAS, method validation is paramount. This involves establishing linearity using a calibration curve (e.g., standard solutions of $Pb(NO_3)_2$ at 0.02, 0.04, 0.06, 0.08, and 0.1 ppm (Ari Wardani et al., 2020). Parameters such as the limit of detection (LOD) and limit of quantitation (LOQ) must be determined, often using the standard deviation of replicate blank measurements (Zakaria & Ho, 2015). Accuracy tests, typically involving spiking samples with known concentrations to calculate percent recovery, are crucial, with acceptable recovery criteria often being 80% - 110% (Ari Wardani et al., 2020).

2.6 Sample Preparation Strategies for Blush-On Analysis

The most critical step preceding AAS analysis of cosmetic powders like blusher is sample preparation, which must effectively destroy the organic matrix and solubilize the heavy metals into an ionic form suitable for atomic analysis (Papadopoulos et al., 2022). The primary techniques used are dry ashing (dry destruction) and wet digestion (wet destruction). Prior to analysis, solid cosmetic samples like blush require sample preparation via a digestion process

to convert the matrix components into basic chemical forms and separate the analytes from organic compounds (El Sharaa I. A. F. et al., 2025). Common methods include dry destruction (dry ashing), which involves subjecting the sample to drying and graying stages in a furnace, causing the organic material to burn off while heavy metals are converted to metal oxides (Ari Wardani et al., 2020).

Before spectroscopic examination, cosmetic samples must undergo pre-treatment (sample preparation) to release the heavy metals from the complex excipients and convert them into a highly oxidized state (soluble salts) suitable for atomic analysis (Abdul Latif et al., 2024). The primary sample preparation methods are dry ashing and wet digestion (Ahmed et al., 2024). Wet digestion typically involves combining the sample with strong acids, such as nitric acid (HNO_3), perchloric acid ($HClO_4$), or sulfuric acid (H_2SO_4) or mixtures like aqua regia (HCl: HNO_3 = 3:1) (Alqahtani et al., 2024). Microwave digestion is an advanced form of wet digestion often used in this field, offering control over temperature and pressure, which helps to minimize the loss of volatile metals and ensures reliable results (Abdelmonem et al., 2025).

The dry destruction method for analyzing Pb in blush preparations involves initial drying and subsequent graying (ashing) to convert inorganic components into ash (Papadopoulos et al., 2022). This is followed by adding strong acid (e.g., 6M hydrochloric acid) to the destroyed ash, drying again, and finally dissolving the residue in an acid solution (e.g., 1M nitric acid) for testing (Ari Wardani et al., 2020). Dry ashing is often preferred for cosmetic products rich in oils and wax, as the heating process (in the presence of oxidizing agents) decomposes organic matter and transforms the metals into metal oxides (Papadopoulos et al., 2022).

Most of the previous studies used wet digestion method. Microwave-assisted digestion is frequently used in modern practice for wet digestion, as it allows control over temperature and pressure, minimizing the loss of volatile heavy metal compounds (Papadopoulos et al., 2022).

It involves treating the sample with concentrated acids, sometimes combined with heat or microwave assistance, to decompose the organic matrix (Papadopoulos et al., 2022). Importantly, Strong oxidizing acids are utilized in acid mixtures to convert the metal compounds into soluble salts. Nitric acid (HNO_3) is commonly used as it is highly effective by functioning simultaneously as a strong acid and an oxidant as well as its metal salts known as nitrates are generally water-soluble (Papadopoulos et al., 2022). HNO_3 is effective for dissolving many inorganic metal compounds but may not fully decompose complex organic matter (Papadopoulos et al., 2022).

Moreover, a rigorous wet digestion protocol often involves weighing the sample and adding concentrated HNO_3 for blush-on analysis, before allowing it to sit overnight and boiling steps. Addition of perchloric acid ($HClO_4$) in this method along with HNO_3 will be successful in ensuring complete oxidation of the organic matrix, resulting in a colourless solution (Sylvia et al., 2021). For microwave digestion, also known as advanced wet digestion technique that uses Teflon vessels in a microwave digester, will apply controlled heat and pressure to ensure quantitative decomposition which is often employing the mixtures of HNO_3 and H_2O (Świerczek et al., 2019). Microwave digestion is highly beneficial as it minimizes the loss of volatile metals and improves accuracy (Papadopoulos et al., 2022).

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Chemicals and Instruments

Table 3.1 shows the list of chemicals used in this study while Table 3.2 shows the list of instruments.

Table 3.1 List of chemicals used in this study

No.	Name	Brand	Purity
1.	Nitric Acid	Merck, Germany	65%
2.	Perchloric Acid	Merck, Germany	70-72%
3.	Standard Solution of Cd (1000 ppm)	Perkin Elmer Pure	-
4.	Standard Solution of Pb (1000 ppm)	Perkin Elmer Pure	-
5.	Standard Solution of Cr (1000 ppm)	Merck, Germany	-

Table 3.2 List of instruments used in this study

No.	Name	Brand
1.	Analytical Balance	Simadzu
2.	Hot Plate & Magentic Stirrer (EMS-HP-7000)	Perkin Elmer
3.	Lumina Hollow Cathode Lamp (Cd)	Perkin Elmer
4.	Lumina Hollow Cathode Lamp (Pb)	Perkin Elmer
5.	Lumina Hollow Cathode Lamp (Cr)	Perkin Elmer
6.	PinAAcle 900F AA Spectrophotometer	Perkin Elmer
7.	Fume Hood	Fisher Hamilton
8.	Pure-lab Chorus Deionised Water	Elga
9.	Arium Pro Deionised Water	Sartorius
10.	Micropipette 100-1000 µL	Chemopharm
11.	Micropipette 5-50 µL	Chemopharm

3.2 Study Design

This study was a laboratory experimental, which is to determine the presence of heavy metals in blush of different colour shades in a same brand that was highly sold in online platform by using the Atomic Absorption Spectrophotometric (AAS).

3.3 Sampling Method

One brand of blush was chosen which was consisting six different colour shades which had the most purchased numbers or highly sold from an online platform available in Malaysia on October and November 2025. As this implies, these samples only represent the chemical composition of the source in the blush palate of the brand that was purchased online at the time.

3.4 Heavy Metal Analysis

Flame Atomic Absorption Spectroscopy (FAAS) will be used for a few heavy metal analysis. The heavy metals that need to be examined include Cd, Pb, and Cr. To determine the concentration of heavy metal to be examined in this investigation, there are two main procedures namely standard solution preparation and sample preparation before running the sample in FAAS.

3.4.1 Standard Solution Preparation

The stock solution of 1000 ppm of Cd, Pb, and Cr were prepared in 50 mL volumetric flask followed by dilution with deionized water to the mark. Next, working solutions were prepared by serial dilution for preparation of five concentration range for all the selected heavy metals. The concentration range for each stock solution prepared is stated in Table 3.3

Table 3.3 Types of heavy metal and respective concentration value

Types of Heavy Metal	Concentration Value (mg/L)
Cadmium (Cd)	0.0625, 0.1250, 0.2500, 0.5000, 1.0000
Lead (Pb)	0.6250, 1.2500, 2.5000, 5.0000, 10.0000
Chromium (Cr)	0.3125, 0.6250, 1.2500, 2.5000, 5.0000

3.4.2 Sample Preparation

All the glassware first was washed with soap, tap water and distilled water before soaked in 10% nitric acid, HNO_3 solution overnight. The glassware was ready to be used in sample preparation method after they were rinsed thoroughly with distilled water.

The main steps in sample preparation involved acid digestion, filtration, and dilution process. The sample first was undergoing acid digestion. In this process, the conical flasks used were labelled with sample identity (eg: Sample A (1), Sample A (2), Sample B (1), Sample B (2)). Then, the blush was homogenized by using mortar and pestel until powder textures were obtained. The blush sample then was weighed within 0.5 g on analytical balance for each sample directly in the labelled conical flask.

Next, the sample was acidified with 25 mL of concentrated nitric acid (HNO_3) and was stood for 24 hours or overnight. After that, the ample was heated and boiled for 35 minutes using a hot plate (100-120ºC) within a fume extraction hood. The sample then was added with 10 mL of perchloric acid ($HCLO_4$) after cool down. Again, the sample was heated and boiled. This should continue until the solution becomes clear and colourless. After the sample was undergoing cool down process, 25 mL of deionised water was added into the sample flask. The sample was boiled again until all NO_2 and brownish gas were come out. The timing of this boiling process was noted between one hour to one and half hours (Sylvia et al., 2021).