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Determination of heavy metal contaminants in unregulated whitening creams using inductively coupled plasma-optical emission spectroscopy (ICP-OES)

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Abstract
Unregulated whitening creams are widely sold through online platforms in Malaysia, raising concerns about the possible presence of toxic heavy metals that may pose health risks to consumers. Therefore, this study aimed to determine the concentration of heavy metals, specifically Arsenic (As), Cadmium (Cd), and Lead (Pb), in selected unregulated whitening cream samples using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). A total of four samples were prepared through acid digestion and analysed. Method validation included linearity, limit of detection (LOD), limit of quantitation (LOQ), recovery, precision, and accuracy. The statistical results were reported as mean ± standard deviation. The concentration of As was detected in one sample (0.0193 ± 0.0005 ppm), while Cd and Pb were found in all four samples, with Cd ranging from 0.0043 ± 0.0003 ppm to 0.0090 ± 0.0003 ppm, and Pb ranging from 0.0796 ± 0.0011 ppm to 0.1059 ± 0.0014 ppm. All values were within the permissible limits established by the World Health Organization (WHO), United States Food and Drug Administration (USFDA), and National Pharmaceutical Regulatory Agency (NPRA) Malaysia. Although the detected concentrations complied with safety standards, the consistent presence of these toxic metals in unregulated cosmetic products may lead to long-term accumulation and health effects. Therefore, strict monitoring of heavy metals in whitening creams is necessary to protect public health, particularly due to the high usage rate of cosmetic products among consumers.
Keywords: heavy metals, ICP-OES, arsenic, cadmium, lead)

Introduction

In recent years, the global cosmetics industry has expanded rapidly, with skin-whitening products gaining significant popularity in various parts of the world, particularly in Southeast Asia. In many societies, fair skin is often associated with beauty, youth, and higher social status, which contributes to the growing demand for whitening creams [1]. As this demand increases, so does the presence of products that are unregulated or unregistered, especially those sold through informal markets or online platforms. These products are not subjected to proper safety evaluations, raising serious concerns regarding consumer health [2].

Unregistered whitening creams often do not comply with the safety standards set by regulatory bodies such as the National Pharmaceutical Regulatory Agency (NPRA) in Malaysia and the United States Food and Drug Administration (FDA). These agencies have established strict guidelines for permissible levels of heavy metals in cosmetics to protect consumers from harmful exposure [3]. However, products that are not approved may contain hazardous substances like lead (Pb), cadmium (Cd), and arsenic (As), which can enter the formulation through contaminated raw materials or inadequate manufacturing practices [4]

Scientific findings have shown that prolonged exposure to these heavy metals can lead to serious health issues. Lead is a well-known neurotoxin that can impair brain development and cause organ damage. Cadmium exposure is linked to kidney dysfunction and is considered a carcinogen,

while arsenic is also recognized for its toxic effects on internal organs and its role in cancer development [5]. Even low concentrations, if used regularly through cosmetic application, may accumulate in the body and result in long-term health complications.

Although some research has been carried out on heavy metal contamination in cosmetic products, most existing studies have focused on registered or imported brands. There is limited data available on unregistered whitening creams that are still widely sold and used in local markets. This lack of current, region-specific information represents a significant research gap, especially given the public's increasing use of these products without proper awareness of their safety [6]. For instance, past studies in Malaysia have detected high levels of heavy metals in cosmetic samples, reinforcing the need for more focused investigations into unregulated items [6].

A reliable and accurate method for detecting trace levels of heavy metals in complex matrices like creams is Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). This technique has been widely used due to its high sensitivity, efficiency, and ability to analyze multiple elements simultaneously [7]. Therefore, the general objective of this study is to determine the concentration of selected heavy metal contaminants, specifically lead, cadmium, and arsenic, in unregistered whitening creams available in local markets using ICP-OES, to assess the potential health risks and support regulatory actions aimed at improving product safety.

Materials and methods

Chemicals and samples

As, Cd, and Pb standard from Perkin Elmer, United States. Analytical grade of nitric acid from Merck, Germany and MilliQ from Millipore, USA. Unregulated whitening cream samples were purchased from Malaysia e-commerce platforms (Shopee and TikTok shop).

Standard solution preparation

Stock solutions of As, Cd, and Pb at 1000 mg/L were obtained from certified reference standards. Working standard solutions were prepared through serial dilutions using deionized water to achieve desired concentrations of 0 to 1 mg/L for As and Cd, and 0 to 10 mg/L for Pb. Nitric acid (HNO_3, 65%) and hydrochloric acid (HCl, 37%) were used for sample digestion. Hydrogen peroxide (H_2O_2, 30%) was included as an oxidizing agent.

Sample preparation

Sample (1g) was accurately weighed and digested using 6 mL of nitric acid (HNO_3, 65%) and 3 mL of hydrochloric acid (HCl, 65%), followed by the addition of 0.25 mL of hydrogen peroxide (H_2O_2, 30%). The digestion process was performed using a microwave digestion system at a controlled temperature of 70°C for approximately 1 to 2 hours or until the formation of white fumes was observed, indicating complete digestion. The samples were cooled to room temperature and filtered. The filtrate was diluted with deionized water in a 25 mL volumetric flask. A blank control sample was prepared using the same procedure without the whitening cream, to ensure quality control during calibration. The final liquid extract was analysed using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) for the determination of arsenic (As), cadmium (Cd), and lead (Pb).

ICP-OES condition

A PE 4300 DV type ICP-OES equipment (Perkin Elmer, United States) was used to determine the concentration of heavy metals in samples. Table 3.1 represented the conditions of ICP-OES.

Table 1: ICP-OES operating condition.

Radio frequency power	0.7 kW to 1.5 kW, 1.2 kW to 1.3 kW for axial
Plasma gas	Argon
Flow rate	Radial: 10.5 L/min to 15.0 L/min
	Axial: 15.0 L/min
Auxiliary gas flow rate	Argon: 1.5 L/min
Viewing height	5 to12 mm

Method validation

To validate the ICP-OES method, several parameters were evaluated. Linearity was confirmed with calibration curves for each metal showing an R^2 value greater than 0.995. Precision was assessed using % Relative Standard Deviation (% RSD) across multiple replicates, while accuracy was determined through standard recovery tests by spiking known concentrations into samples. LOD and LOQ were calculated using standard deviation and calibration slope.

Linearity

The regression line method of the least squares technique was used to obtain the coefficient of determination (R^2) for assessing linearity. To construct a calibration curve of unregulated whitening cream samples from different sources, the mean corrected intensity was used as the y-axis of the calibration curve, and the concentration of heavy metals added was used as the x-axis. The acceptance criteria for the calibration curve were set at $R^2 > 0.995$ [19] and a relative standard deviation (RSD) < 20% [20].

Limit of Detection (LOD) and Limit of Quantification (LOQ)

The ratio signal to noise 3:1 of the lowest analyte concentration was used for the determination of LOD, while the ratio signal to noise 10:1 of the lowest analyte concentration was used for the determination of LOQ in calibration curve[20]. These two parameters were calculated from respective calibration curves using equations proposed by prior studies for all of the heavy metals studied [20].

$$LOD = \frac{3.3 \times standard\ deviation}{slope\ of\ calibaration\ curve} \quad \text{Equation 1}$$

$$LOD = \frac{3.3 \times standard\ deviation}{slope\ of\ calibaration\ curve} \quad \text{Equation 2}$$

Precision and accuracy

Precision measures the consistency of results across repeated measurements and is expressed as the relative standard deviation (% RSD). Precision testing involves multiple analyses of standards or uniform samples across different samplings. The accuracy was presented in the percentage differences of the estimation concentration from the nominal values and the precision was presented in the coefficient of variation (RSD) value.

Recovery

The recovery percentage was calculated by comparing the blank samples in nitric acid with the mean corrected intensity of spiked known heavy metal standard solution in those samples.

Data Analysis

Data obtained from ICP-OES were processed to calculate metal concentrations in parts per million (ppm). Statistical analysis, including one-way ANOVA and t-tests, was conducted to compare the levels of heavy metals across different batches and products. Results were presented as mean values with standard deviations.

Results and discussion

Method validation

Four method validation parameters (linearity, LOD and LOQ, precision and accuracy, recovery) were measured for the determination of As, Cd, and Pb in unregulated whitening creams samples. From previous studies, the acceptance criteria for a calibration curve were defined as having a correlation coefficient (R^2) greater than 0.995 [19] and a relative standard deviation (RSD) less than 20% [20]. These criteria were adopted in this study, as the calibration curves for As, Cd, and Pb met these standards in earlier research focused on toxic metal determination. In this project, calibration curves were constructed using standard solutions within the concentration range of 0 to 2.5 mg/L for As, Cd,

and Pb in alignment with the levels commonly found in cosmetic products. The calibration results demonstrated excellent linearity for all three metals, with R^2 values exceeding 0.999 and RSD values remaining below 20%. Table 2, presents the calibration ranges, correlation coefficients, and regression equations for each heavy metal analyzed in unregulated whitening cream samples.

The data from accepted calibration curves were used to calculate LOD and LOQ. The LOD for As, Cd, and Pb were 0.002 mg/L, 0.026 mg/L, and 0.008 mg/L. Whereas, the LOQ for As, Cd, and Pb were 0.004 mg/L, 0.078 mg/L, and 0.025 mg/L. Table 2 showed the LOD and LOQ for each heavy metal. The precision and accuracy of the ICP-OES measurements for selected heavy metals were evaluated based on the standard deviation (SD) and relative standard deviation (RSD) of the data obtained. The calculated precision values (%RSD) for As, Pb, and Cd in the whitening cream samples ranged from 1.05% to 6.98%, indicating good repeatability of the method. Among all elements, Cd showed the highest RSD value of 6.98% in WC3, but this is still below the commonly accepted threshold of 10%, which meets the criteria reported in previous studies [20]. Therefore, the data presented for precision and accuracy in this research were reliable. Table 2 showed the precision and accuracy for each toxic metal.

Table 2: Limit of Detection (LOD), Limit of Quantification (LOQ), Standard Error (SE), Standard Deviation (SD), and Regression Formula for As, Cd, and Pb Determined by ICP-OES.

Elements	LOD	LOQ	SE	SD	Regression formula
As	0.001456739	0.00441436	4.787814674	9.575629348	y = 21692x + 80.38
Cd	0.025678449	0.077813481	3890.674059	7781.348118	y = 1E+06x - 7415.4
Pb	0.008192916	0.024827017	128.6101546	257.2203092	y = 103605x + 125.64

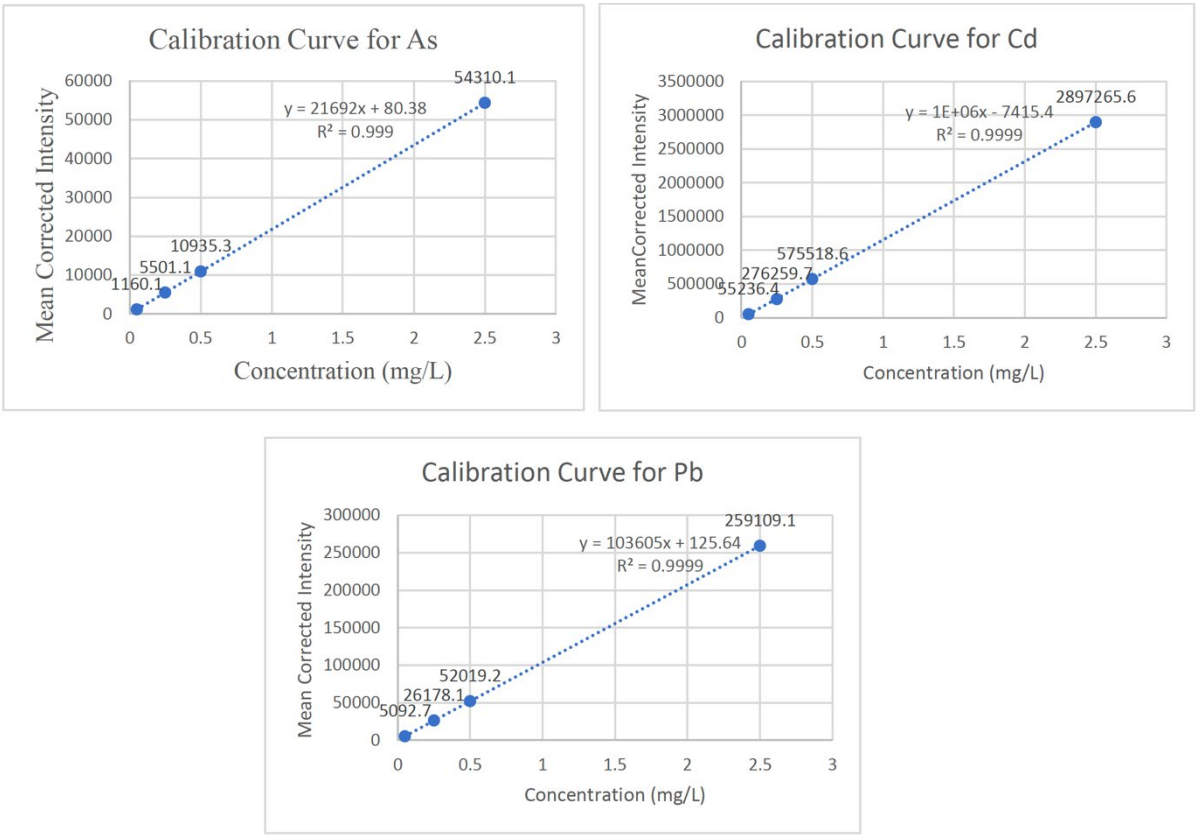

Figure 1 Calibration Curve of As, Cd, Pb Standard.

The recovery results for each toxic metal presented as RSD value and the results for As, Cd, and Pb were 101.8%, 99.0% and 101.0% respectively. As a result, the recovery percentages of each heavy metal are in the acceptance range, which was 85% - 115% [20]. Hence, the data presented for recoveries in this research was reliable suitable for trace heavy metal determination in cosmetic samples. These findings support the validity of the analytical method used for quantifying heavy metals in the tested unregulated whitening cream products.

Table 3: The amount of selected heavy metals in unregulated whitening creams mean levels (ppm) in different product (Mean ± SD, n = 3).

Elements	Batch	Mean ± SD			
		WC1	WC2	WC3	WC4
As	1	-0.025 ± 0.002	-0.015 ± 0.0017	-0.017 ± 0.0021	0.0194 ± 0.0011
	2	-0.041 ± 0.0028	-0.019 ± 0.0015	-0.001 ± 0.0049	0.0183 ± 0.0009
	3	-0.045 ± 0.0007	-0.021±0.0013	-0.018 ± 0.0018	0.0201 ± 0.0010
	Mean	-0.037 ± 0.0183	-0.018 ± 0.0015	-0.015 ± 0.00293	0.0193 ± 0.0005
Pb	1	0.0856 ± 0.0015	0.1072 ± 0.0020	0.0940 ± 0.0018	0.0783 ± 0.0013
	2	0.0881 ± 0.0018	0.1045 ± 0.0016	0.0967 ± 0.0015	0.0811 ± 0.0010
	3	0.0872 ± 0.0013	0.1061 ± 0.0019	0.0955 ± 0.0017	0.0795 ± 0.0012
	Mean	0.0870 ± 0.0012	0.1059 ± 0.0014	0.0954 ± 0.0010	0.0796 ± 0.0011
Cd	1	0.0064 ± 0.0006	0.0092 ± 0.0007	0.0041 ± 0.0003	0.0058 ± 0.0005
	2	0.0071 ± 0.0005	0.0088 ± 0.0006	0.0046 ± 0.0004	0.0061 ± 0.0006
	3	0.0069 ± 0.0004	0.0090 ± 0.0005	0.0043 ± 0.0002	0.0060 ± 0.0004
	Mean	0.0068 ± 0.0004	0.0090 ± 0.0003	0.0043 ± 0.0003	0.0060 ± 0.0003

Table 4: The precision, accuracy for determination of As, Cd, and Pb in unregulated whitening creams samples.

Elements	Samples	Mean (ppm)	SD	Precision (%RSD)	Accuracy (%)
As	WC4	0.0193	0.0005	2.59	96.55
Pb	WC1	0.0870	0.0012	1.38	98.66
	WC2	0.1059	0.0014	1.32	98.86
	WC3	0.0954	0.0010	1.05	99.50
	WC4	0.0796	0.0011	1.38	98.63
Cd	WC1	0.0068	0.0004	5.88	94.12
	WC2	0.0090	0.0003	3.33	96.67
	WC3	0.0043	0.0003	6.98	93.02
	WC4	0.0060	0.0003	5.00	95.00

Concentration of As, Cd, Pb in Whitening Creams in Local

Heavy metals such as arsenic (As), cadmium (Cd), and lead (Pb) are commonly identified as environmental and public health threats due to their toxicity, persistence, and tendency to accumulate in biological systems. Although often present in small quantities, these elements can pose serious health risks when exposure occurs regularly over time, particularly through daily-use cosmetic products such as skin-whitening creams. The concern is greater for unregulated products that are not subjected to the same quality control or toxicological screening as registered brands. These items are widely available through online platforms and informal markets, which often lack transparency regarding their formulation or safety compliance [21 (, 15]

In this study, four different whitening cream samples were analysed to determine the

concentrations of arsenic, cadmium, and lead using inductively coupled plasma optical emission spectroscopy (ICP-OES). The analytical method was validated for precision, accuracy, linearity, and sensitivity. The high coefficient of determination values and consistent recovery rates confirmed the reliability of the instrument and the overall analytical procedure.

Arsenic was detected in only one out of four samples, specifically in sample WC4, where the concentration was found to be 0.0193 ± 0.0005 ppm. This level is well below the maximum allowable limits specified by the World Health Organization and the United States Food and Drug Administration, which both recommend a limit of 3 ppm. It also complies with the guideline set by Malaysia's National Pharmaceutical Regulatory Agency, which allows up to 5 ppm in cosmetic products

Cadmium was detected in all four samples with concentrations ranging from 0.0043 ± 0.0003 ppm to 0.0090 ± 0.0003 ppm. These values are well within the limits established by the NPRA, which allows up to 5 ppm, and the FDA, which permits up to 10 ppm in cosmetics. However, they fall below the more stringent guideline proposed by the World Health Organization, which recommends a limit of 0.3 ppm.

Lead was also present in every sample, with concentrations ranging from 0.0796 ± 0.0011 ppm to 0.1059 ± 0.0014 ppm. These values are far below the limits set by WHO and FDA, both of which consider 10 ppm acceptable in cosmetics, and significantly under Malaysia's NPRA limit of 20 ppm. Despite being within regulatory bounds, the presence of lead remains concerning because no threshold has been universally identified as completely safe, particularly for developing children or pregnant women. Lead is a potent neurotoxin, and chronic exposure has been linked to developmental delays, learning disabilities, kidney impairment, and reproductive problems [22].

Therefore, although the findings of this study indicate that the levels of heavy metals in the tested unregulated whitening cream samples were within the permissible limits, the potential health risks associated with long-term exposure should not be underestimated. Continuous use of cosmetic products containing trace amounts of toxic metals may lead to accumulation in the body and pose serious health effects over time. In view of this, it is recommended that regulatory agencies such as the National Pharmaceutical Regulatory Agency (NPRA) enhance their post-market surveillance and enforcement strategies.

In conclusion, while the measured concentrations of arsenic, cadmium, and lead in these unregistered whitening creams do not violate current safety guidelines, their detection indicates potential exposure risks that deserve further attention. Consumers who frequently use such products are at risk of chronic low-dose exposure, which may lead to adverse health effects over time. Greater regulatory enforcement increased public awareness, and better labelling requirements are essential to ensure that cosmetic products marketed in Malaysia meet acceptable safety standards and do not pose a threat to human health.

Conclusion

The purpose of this study was to determine the concentration of arsenic (As), cadmium (Cd), and lead (Pb) in selected unregulated whitening cream samples sold through online platforms in Malaysia using inductively coupled plasma optical emission spectrometry (ICP-OES). The validated method provided accurate and reliable results. The concentration of As, Cd, and Pb in the whitening cream samples was then compared with the maximum permissible limits set by the World Health Organization (WHO), the United States Food and Drug Administration (FDA), and the National Pharmaceutical Regulatory Agency (NPRA) of Malaysia.The results showed that As was detected in one sample, while Cd and Pb were present in all four samples. All values were within the permissible limits of WHO, FDA, and NPRA. However, the presence of heavy metals in unregulated whitening creams, even in low concentrations, indicates the possibility of long-term exposure and accumulation in the human body. This may cause health effects such as cancer, kidney damage, and neurological disorders due to the toxic nature of these metals. Therefore, continuous use of these unregistered whitening creams may pose risks to consumer health. It is important that regulatory authorities implement stricter monitoring and quality control, especially for cosmetic products that are sold online without proper approval.

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